

Research Article

Friedel-Crafts Polyketones: Synthesis, Characterization and Antimicrobial Properties of Unsaturated Polyketones and Copolyketones Based on Difurfurylidene Cycloheptanone

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A new type of unsaturated polyketones and copolyketones having cycloheptanone moiety in a p-conjugated main chain were synthesized via Friedel-Crafts reaction through the polymerization of the monomer: 2,7-bis furfurylidene cycloheptanone I with different diacid chlorides. The model compound was synthesized by reacting I with benzoyl chloride and characterized by $^1\text{H-NMR}$, IR, and elemental analyses. The polyketones and copolyketones were soluble easily in protic solvents like H_2SO_4 and trifluoroacetic acid. The thermal properties of these polyketones and copolyketones were evaluated and correlated to their structural units by TGA and DSC measurements. The crystallinity of some polymers was tested by X-ray analyses; also the morphological properties of selected examples of poly and copolyketones were detected by SEM. All the polyketones were tested for their biological activity against bacteria, fungi, and yeast. It was observed that the majority of the polyketones and its copolymers synthesized can be used as antibacterial and antifungal agents.

1. Introduction

Polyketones represent a new class of potential engineering thermoplastics. Their property set is positioned in the area of polyamides and polyacetals. They are characterized by their stiffness, high melting temperature, and resistance to water and moisture. In addition, one should also mention chemical resistance, stability at hydrolysis conditions, impact toughness, flame resistance without additives containing halides or red phosphor, and short cycle times in injection molding [1–3]. Friedel-Crafts polymers formed on polycondensation of 4,4'-dichloromethyldiphenyl ether (DDE) with benzene, toluene, isomeric chlorotoluenes and xylenes, phenol, and isomeric cresols have been reported [4–6]. Recently, a considerable amount of research effort has concentrated on the synthesis of polyketones via Friedel-Crafts reaction [7–11].

During the last decade an extensive work was carried out towards the prevention of biological degradation of useful materials such as textile fibers, food packaging materials, paints, marine coatings, electrical insulation and pharmaceutical materials using certain polymeric systems having

biocidal properties [12–14]. It was, therefore, thought appropriate to synthesize the polyketones and copolyketones containing cycloheptanone moiety and examine their biocidal properties against *Aspergillus flavus*, *Aspergillus niger*, *Candida albicans*, *Geotrichum candidum*, *Trichophyton rubrum*, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Staphylococcus aureus*.

2. Experimental

2.1. Materials

2.1.1. Reagents. Furfural (Fluka, Germany) was freshly distilled at b.p. 162°C . Cycloheptanone (Merck) was used without purification. Benzoyl chloride (Aldrich, Seelze, Germany) was used as purchased. Terephthaloyl chloride and isophthaloyl chloride (Aldrich, Seelze, Germany) were recrystallized twice from n-hexane (m.p $83\text{--}84^\circ\text{C}$ and 40°C resp.). Adipoyl and sebacoyl dichlorides [15] were freshly distilled at $125^\circ\text{C}/11$ Torr and at $182^\circ\text{C}/16$ Torr, respectively were used. Anhydrous aluminum chloride and sodium

TABLE 1: Elemental analyses, inherent viscosity, yield and color of polyketones **IVa–d** and copolyketones **V** and **VI**.

Polymer Code	Repeating unit	C%		H%		η_{inh} (dl/g)	Yield%	Appearance of polymer
		Calcd.	Found	Calcd.	Found			
IVa	C ₂₅ H ₁₈ O ₅	75.38	74.43	4.52	4.26	0.37	68	Powder-yellow
IVb	C ₂₅ H ₁₈ O ₅	75.38	74.92	4.52	4.13	0.42	73	Powder-yellow
IVc	C ₂₃ H ₂₂ O ₅	73.02	72.58	5.82	5.58	0.56	76	Powder-yellow
IVd	C ₂₇ H ₃₀ O ₅	74.65	73.14	6.91	6.72	0.63	69	Powder-yellow
V	C ₅₂ H ₃₆ O ₈	79.19	77.87	4.56	4.41	0.52	85	Powder-yellow
VI	C ₄₈ H ₄₄ O ₈	77.01	76.72	5.88	5.32	0.46	81	Powder-yellow

* η Inherent viscosity measured in H₂SO₄ at 25 °C.

hydroxide (BDH, Leicestershire, England) were of analytical grade and were used as received.

2.1.2. Solvents. Methanol, 99.8%, Ethanol, 99.9% (Hayman, Eastways Withan, England), acetone and N,N-dimethyl formamide (DMF), (BDH, Leicestershire, England), N-Methyl-2-pyrrolidone (NMP) and benzene (Aldrich, Seelze, Germany) were extra pure solvents and were used as received. Carbon disulphide (Aldrich, Seelze, Germany) was a guaranteed reagent, dried over calcium hydride for 24 h, and followed by distillation under reduced pressure.

2.2. Methods

2.2.1. Monomer Synthesis

Synthesis of 2,7-bis furfurylidene Cycloheptanone I. A mixture of 0.1 mole cycloheptanone and 0.2 mole of furfural in warm ethanol was stirred, and few drops of NaOH (20% conc.) were added. The reaction mixture was stirred at room temperature for 1 hr, and the resulting solid was collected by filtration, washed with cold water, dried and recrystallized from ethanol, as yellow needles; yield 95%, m.p 166 °C. Calculated for C₁₇H₁₆O₃: C, 76.69; H, 6.02%. Found: C, 76.53; H, 5.85%. IR (KBr, cm⁻¹): at 1600 (s, C=C), at 1660 (s, C=O). ¹H-NMR (CDCl₃, ppm), at 7.0–8.0 (m, 6H of furfuryl moieties and 2H of 2CH=C); at 2.2–2.5 (d, 4H of 2CH₂); at 1.8–2.1 (d, 4H of middle 2CH₂ of cycloheptanone).

Synthesis of 2,7-bis(benzylidene)cycloheptanone II. This monomer was prepared as described previously [16].

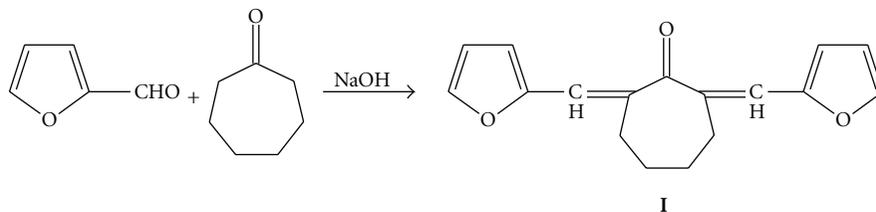
2.2.2. Synthesis of Model Compound III. In a three necked flask equipped with a condenser, dry nitrogen inlet and outlet, and a dropper, a mixture of 1 mmole of monomer **I** suspended in 40 mL of carbon disulphide and 2 mmole of benzoyl chloride were introduced. A 20 mmole of anhydrous aluminum chloride was added portion wise to the solution. The solution was stirred for 24 hrs at 25 °C, and the separated solid product was filtered off washed with water dried in vacuum at room temperature. An analytical sample was obtained by recrystallization from benzene in yellow needles, yield 75%, m.p 219 °C. Calculated for C₃₁H₂₄O₅: C, 78.15; H, 5.04; %. Found: C, 77.76; H, 5.01%. IR (KBr, cm⁻¹): at 1670 (C=O of cycloheptanone), at 1700 (C=O of benzoyl group),

at 1600 (C=C group). ¹H-NMR (DMSO-d₆, ppm), at 7.2–8.5 (m, 4H, furfuryl moiety, 10H Ar-H, and 2H of 2CH=C), at 2.2–2.5 (d, 4H of 2CH₂); at 1.8–2.1 (d, 4H of middle 2CH₂ of cycloheptanone).

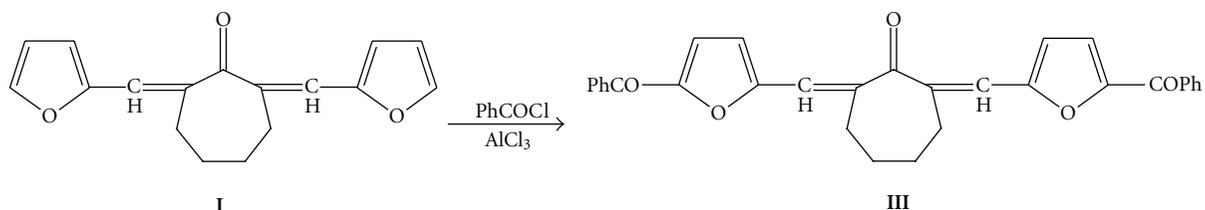
2.2.3. Synthesis of Polyketones IVa–d. Friedel-Crafts method was applied for the preparation of all the polymers. Typically, in a three necked flask equipped with a condenser, dry nitrogen inlet and outlet, and a dropper, a mixture of 6 mmole (1.596 g) of monomer **I** and 1.218 g (6 mmole) of isophthaloyl chloride in 25 mL of dry carbon disulphide were introduced. The flask was purged with nitrogen while stirring and a 2.66 g (20 mmole) of aluminum chloride was added during the nitrogen flash. The reddish brown reaction mixture was stirred for 24 hrs then it was filtered, off and the separated product was triturated three times with a large excess of methanol. The powdery material was again filtered off, washing with water, methanol, and acetone, and dried under reduced pressure (1 mmHg) at 70 °C for 2 days. This method was applied for the preparation of other polyketones; whose yields, elemental analyses, viscosity, and colors are listed in Table 1.

2.2.4. Synthesis of Copolyketones V and VI. The same method, which applied in the synthesis of the polyketones, was also applied in the synthesis of the copolyketones.

Typically, in a three necked flask equipped with a condenser, dry nitrogen inlet and outlet, and a dropper, a mixture of 0.798 g (3 mmole), 2,7-bis furfurylidene cycloheptanone **I** and 0.864 g (3 mmole) of 2,7-bis(benzylidene)-cycloheptanone **II** and 1.208 g (6 mmole) of terephthaloyl chloride, and 50 mL of dry carbon disulphide were introduced. The flask was purged with nitrogen while stirring, and 2.66 g (20 mmole) of aluminum chloride was added during the nitrogen flash. The reddish brown reaction mixture was stirred for 24 hrs then it was filtered off, and the separated product was triturated three times with a large excess of methanol. The powdery material was again filtered off, washing with water, methanol, and acetone and dried under reduced pressure (1 mmHg) at 70 °C for 2 days. This method was applied for the synthesis of copolyketone **VI**; whose yield, elemental analysis, viscosity, and colors are also listed in Table 1.



SCHEME 1: Synthesis of 2,7-bis furfurylidene cycloheptanone I.



SCHEME 2: Synthesis of model compound III.

Polymer Identification. Infrared spectra of the prepared polymers were measured on a Nicolet 6700 FT-IR Thermo Fisher Scientific Incorporation. (USA). The powder samples were of similar weight and mixed with KBr. All spectra were recorded within the wave number range of $4000\text{--}400\text{ cm}^{-1}$ at 25°C . Elemental analyses of the prepared monomers, model compound, and polymers were performed at the Micro Analytical Unit, Assiut University, Egypt.

Polymer Characterization

Viscosity. Inherent viscosity measurements were carried out on 0.5% solutions of the polymers in NMP at 30°C using a suspended-level Ubbelohde viscometer with negligible kinetic energy correction. Flow times were measured at five different concentrations of the polymer sample. All the plots obtained were linear. Inherent viscosity was determined by usual extrapolation of gsp/c to zero concentration and expressed in deciliter per gram (dL g^{-1}).

Solubility. The solubility of polymers in various solvents such as DMF, DMSO, and NMP was determined at room temperature (30°C). It was performed by gradual addition of the polymer to the solvent and stirred well till saturation. The maximum solubility of the polymers were calculated as percent weight of the polymer per hundred milliliter of solvent (% w/v).

Scanning Electron Microscopy Measurements. The morphology of polyketone A as an example was examined by scanning electron microscopy (SEM) using a Jeol JSM-5400 LV instrument.

Thermogravimetric Analysis. Thermogravimetric analysis (TGA) curves were recorded on a Shimadzu TGA-50 H in air atmosphere at a heating rate of $10^\circ\text{C min}^{-1}$ and a heating

range from room temperature to 700°C . The sample weights ranged from 3 to 5 mg, and the gas flow rate was 30 mL min^{-1} .

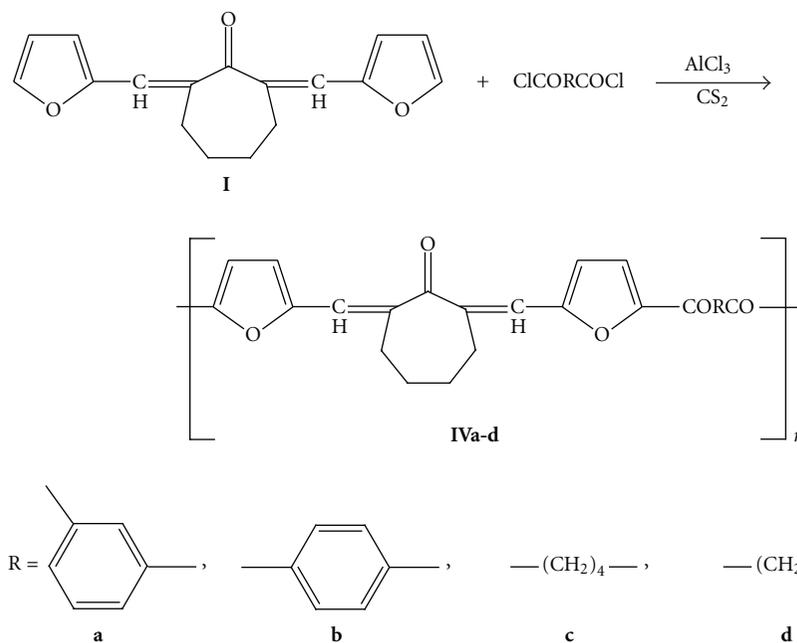
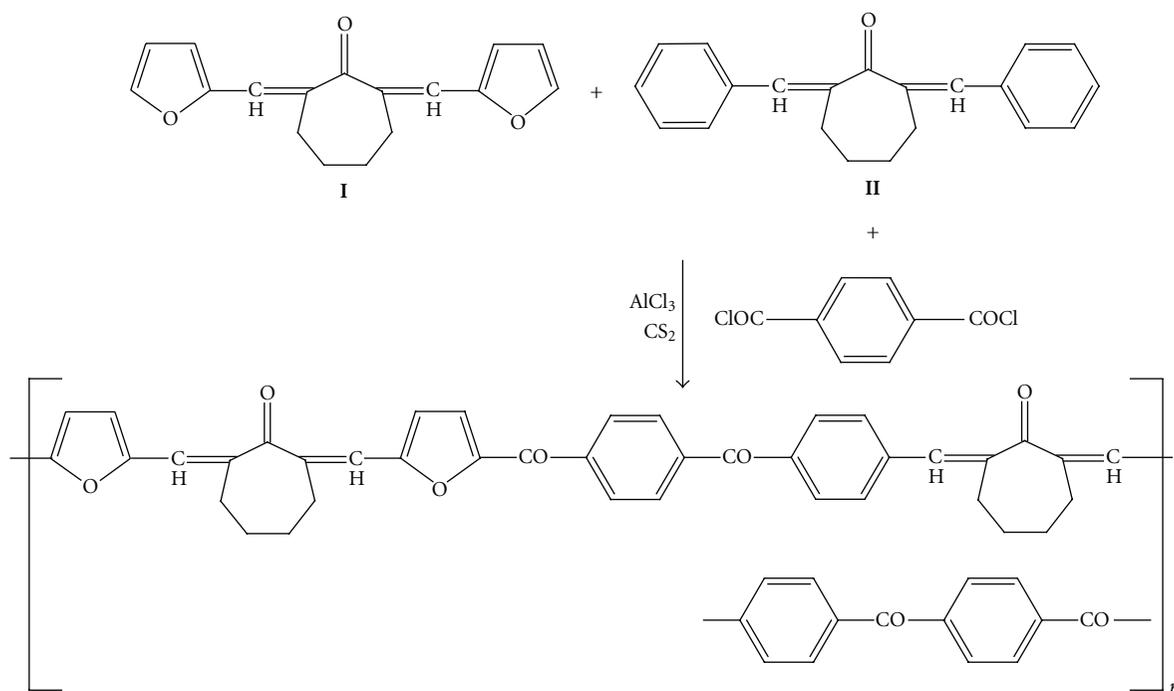
Antimicrobial Properties. The antimicrobial screening of polyketones and copolyketones (IV-a-d, V, VI) were performed using the standard agar diffusion method, against different organisms (fungal and bacterial species) including: *Aspergillus flavus*, *Aspergillus niger*, *Candida albicans*, *Geotrichum candidum*, *Trichophyton rubrum*, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Staphylococcus aureus*. The fungal species were maintained on sabourand dextrose agar (SDA) whereas the bacterial species were maintained on nutrient agar (NA).

3. Results and Discussion

3.1. Synthesis of Monomer I. 2,7-bis furfurylidene cycloheptanone I monomer was synthesized in good yields by the basic catalyzed condensation of two moles of furfural with one mole of cycloheptanone, as shown in Scheme 1.

3.2. Synthesis of Model Compound III. Before attempting the polymerization, model compound was prepared by the reaction of monomer I with two equivalents benzoyl chloride. A typical reaction is depicted in Scheme 2.

3.3. Synthesis of Polyketones IVa-d. In order to determine an adequate catalyst for the synthesis of polyketones based on furfurylidene cycloheptanone in the main chain, Friedel-Crafts polycondensation of 2,7-bis furfurylidene cycloheptanone I with terephthaloyl chloride (as an example) (polymer IVb) was carried out in the presence of various Lewis acids such as FeCl_3 , SbCl_5 , and AlCl_3 . It appeared that anhydrous AlCl_3 gave the best result with respect to yield and degree of polymerization. The favorable mole ratio of catalyst to each reactant was ca. 2.0.

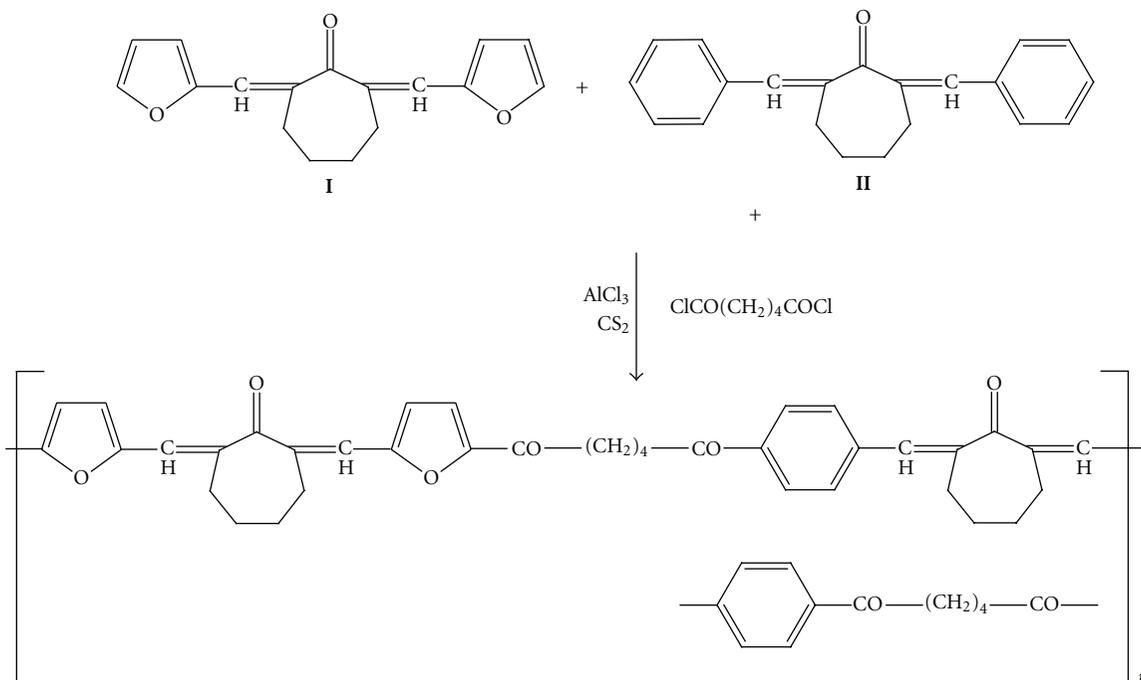
SCHEME 3: Synthesis of polyketones **IVa-d**.SCHEME 4: Synthesis of copolyketone **V**.

On the basis of these results, Friedel-Crafts polycondensation of dicarbonyl chlorides including: isophthaloyl, terephthaloyl, adipoyl and sebacoyl dichlorides with 2,7-bis furfurylidene cycloheptanone **I** was carried out at room temperature for 24 hrs. These reactions are shown in Scheme 3.

3.4. Synthesis of Copolyketones V and VI. Unreported copolyketones **V** and **VI** containing two different moieties

for example bis furfurylidene cycloheptanone, and bis-benzylidene cycloheptanone in the polymer main chain. These two copolyketones were synthesized from the copolymerization of monomer **I** and monomer **II** with two different diacid chlorides, for example, terephthaloyl or adipoyl as shown in Schemes 4 and 5.

The polymerization and copolymerization reactions were carried out at room temperature in carbon disulphide as a



SCHEME 5: Synthesis of copolyketone VI.

solvent and anhydrous aluminum chloride as a catalyst. The reaction times varied from 20 to 24 hr and the polymers and copolymers were immediately isolated when the reaction mixtures were poured into methanol/water mixture, with yield in the range of 68–85%. These polymers were characterized by elemental analysis, IR spectroscopy, solubility, viscometry, thermal analysis, and morphological properties. The elemental data of all the polymers and copolymers coincided with the characteristic repeating units (Table 1).

Spectral analyses in (KBr disks) showed the appearance of new carbonyl absorption at $1690\text{--}1720\text{ cm}^{-1}$, characteristic for the C=O of ketonic group besides the original C=O of cycloheptanone at $1650\text{--}1670\text{ cm}^{-1}$, and at C=C at $1595\text{--}1610\text{ cm}^{-1}$. Other characteristic absorption peaks for the rest of the groups in the molecules were also present.

3.5. Properties of the Polyketones IVa–d and Copolyketones V, VI. The solubility characteristics of polyketones and copolyketones (IVa–d, V, VI) were tested in various solvents (Table 2) including: dimethylsulfoxide (DMSO), N,N-dimethylformamide (DMF), N-methylpyrrolidone (NMP), tetrahydrofuran (THF), methylene chloride, CHCl_3 -acetone (1:1), and concentrated sulphuric acid. It was found that all the polyketones and copolyketones dissolved readily in concentrated H_2SO_4 at room temperature giving deep red color due to the instability of the polymers. For halogenated solvents like CH_2Cl_2 and CHCl_3 -acetone mixture, polymers IVa–c and copolymers V, VI were partially soluble. More particularly, polymer IVd and copolymer VI (with $n = 8$ or 4 resp.) showed good solubility in most organic solvents, due to the high flexibility of these polymers through the polymethylene spacers.

The morphology of the synthesized polyketone IVa in Figures 1(a) and 1(b) was examined by SEM (Jeol-SM-5400 LV instrument). The SEM sample was prepared by putting a fine powder of polyketone on a smooth surface of aluminum foil and coating it with gold-palladium alloy. The SEM (Camera) with Ilford film at an accelerating voltage of 15 kv using a low-dose technique [17]. The SEM study of polyketone IVa in Figures 1(a) and 1(b) showed that the polymer had a polymorph globular and subglobular structures which (with magnification) the globular structure has a “cauliflower” appearance.

The thermal behavior of polyketones IVa,d and copolyketones V and VI were evaluated by thermogravimetric analysis (TGA) in air at a heating rate of $10^\circ\text{C}/\text{min}$. The thermographs of these polymers are given in Figure 2, also Table 3 gives the temperature of various percentages of weight loss. In Figure 2, TGA curves of polyketones IVa (as an example) show a small weight loss in the range 2–4% starting at 160°C until 200°C , which may be attributed to loss of absorbed moisture and entrapped solvents. The thermographs also indicate the polymers decompose in two stages. The first stage between 205°C and 310°C depends upon the nature of the polyketones. This result is in good agreement with the decomposition of ketone-linkage observed by Swedo and Marvel [18]. The second stage of degradation of polyketones occurred between 330°C and 540°C . The rate of degradation in the first stage is somewhat faster than in the second stage. A comparison of the T_{10} values of polyketones IVa,b containing aromatic moiety showed better thermal stability than others based on aliphatic IVc,d. For copolyketones V and VI, a small

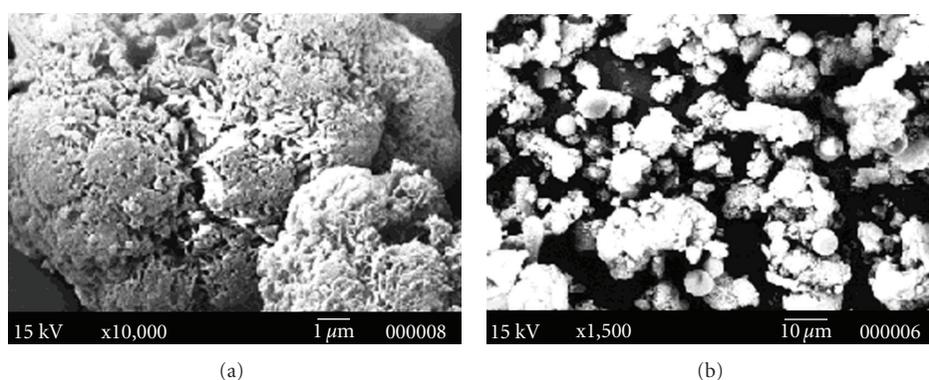
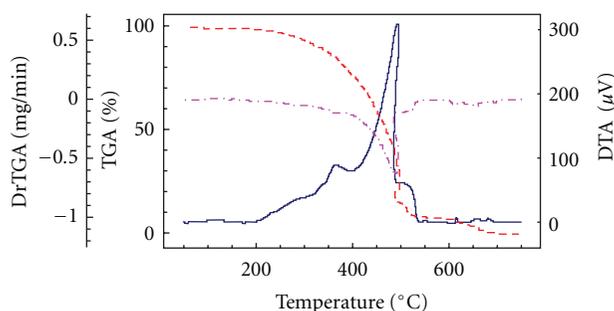
TABLE 2: Solubility characteristics of polyketones **IVa–d** and copolyketones **V** and **VI**.

Polymer code	DMSO	DMF	NMP	Chloroform Acetone (1 : 1)	THF	Methylene chloride	H ₂ SO ₄
IVa	±	±	±	±	±	±	+
IVb	±	±	±	±	±	–	+
IVc	±	±	±	±	±	–	+
IVd	+	+	±	+	±	±	+
V	±	±	±	±	±	±	+
VI	+	±	±	±	±	±	+

(+) Soluble at room temperature RT.

(±) Partially soluble at RT.

(–) Insoluble.

FIGURE 1: SEM image of polyketone **IVa**.FIGURE 2: Thermogravimetric curves of polyketones **IVa**.

weight loss in the range 2–4% starting at 180°C until 220°C, which may be attributed to loss of absorbed moisture and entrapped solvents. In contrary to the decomposition of the polyketones, the decomposition of copolyketones occurred in one step only, and the T_{10} values are in the range 350–370°C. Moreover, the data indicated that the copolyketone **V** is more thermally stable than the copolyketone **VI**, and this may be attributed to the presence polymethylene groups (CH₂)₄ in as flexible spacers in the polymer main chain in the later copolyketone, and this decreases the stability.

TABLE 3: Thermal properties of Polyketones **IVa–d** and **V**, **VI**.

Polymer code	Temperature (°C) for various decomposition levels				
	10%	20%	30%	40%	50%
IVa	300	350	385	445	470
IVb	250	265	280	310	370
IVc	210	255	280	310	355
IVd	205	250	275	305	340
V	370	420	455	470	495
VI	350	405	435	440	470

* Heating rate: 10°C min⁻¹.

Antimicrobial Screening. The antimicrobial screening of polyketones **IVa–d** and copolyketone **V** were performed using the standard agar diffusion method, against different organisms (fungal and bacterial species) including: *Aspergillus flavus*, *Aspergillus niger*, *Candida albicans*, *Geotrichum candidum*, *Trichophyton rubrum*, *Escherichia coli*, *Pseudomonas aeruginosa*, and *Staphylococcus aureus*.

The fungal species were maintained on sabourand dextrose agar (SDA) whereas the bacterial species were maintain on nutrient agar (NA). Similar inhibitory effects were obtained by several authors who dealt with antifungal and

TABLE 4: Effect of polyketones **IVa,c,d**, and copolyketones **V, VI** on the growth of fungal and bacterial species.

Type of Organism	Polyketone inhibition number zone (mm)					
	IVa	IVc	IVd	V	VI	Control
<i>Aspergillus niger</i>	11	8	10	0	8	19
<i>Candida albicans</i>	0	0	0	0	0	30
<i>Trichophyton rubrum</i>	15	18	0	0	8	23
<i>Aspergillus flavus</i>	6	7	11	18	0	25
<i>Escherichia coli</i>	0	0	0	0	0	26
<i>Pseudomonas aeruginosa</i>	0	0	0	0	0	18
<i>Geotrichum candidum</i>	0	10	21	9	8	32
<i>Staphylococcus aureus</i>						
<i>Staphylococcus aureus</i> , yeast, and bacteria including: <i>Aspergillus niger</i> ,	13	9	17	15	0	24

Control: (antibacterial: chloramphenicol, antifungal: trosyd).

antibacterial activities of different polyketone derivatives. Patel and Patel [11] pointed out that chlorine containing polyketones exerted a very closed an inhibitory effect against some fungi, yeast, and bacteria including: *Aspergillus niger*, *Penicillium chrysogenum*, *R. minuta*, *S. cerevisiae*, *P. stipitis*, *P. fluorescens*, *B. subtilis* and *E. coli* species.

Antifungal and antibacterial activities of the tested polymers were determined as described before [19, 20]; the size of the resulting inhibition zone was determined in Table 4 It can be seen from the Table 4 that all the selected polyketones and copolyketone showed no significant influence antifungal activity against the selected fungi.

On the other hand, it can be clarified from the data shown in Table 4 and Figure 3 that the majority of the selected polyketones and copolyketone has a moderated antibacterial activity against *Trichophyton rubrum*, *Bacillus cereus*, *Serratia marcescens*, and *Staphylococcus aureus*. The exception for polyketones **VI**d and **VI** against *Trichophyton rubrum*, polyketone **V** against *Aspergillus flavus*, polyketone **IVa** against *Geotrichum candidum*, and polyketone **V** against *Staphylococcus aureus* which have no significant influences. Whereas only polyketones **IVa** and **IVb** showed a strong antibacterial activity against *Trichophyton rubrum* compared with the controlled values (15,18/resp.), polyketones **IVb**, **IVc**, **IVd**, **V** showed strong antibacterial activity against *Geotrichum candidum* (10/21/9/8 resp.); and polyketones **IVa**, **IVb**, **IVc**, **IVd** showed strong antibacterial activity against *Staphylococcus aureus* (13,9/17/15 resp.), on the other hand, the rest of selected polymers and copolymers showed week significant influences.

These results indicate that the polyketones significantly inhibit the growth of microorganisms. It can be clarified from this figure that, the control culture (without polymer sample) generally exhibits maximum growth. On the other hand, the polymers sample giving different growth may be attributed to polymer structures. We believe that more work

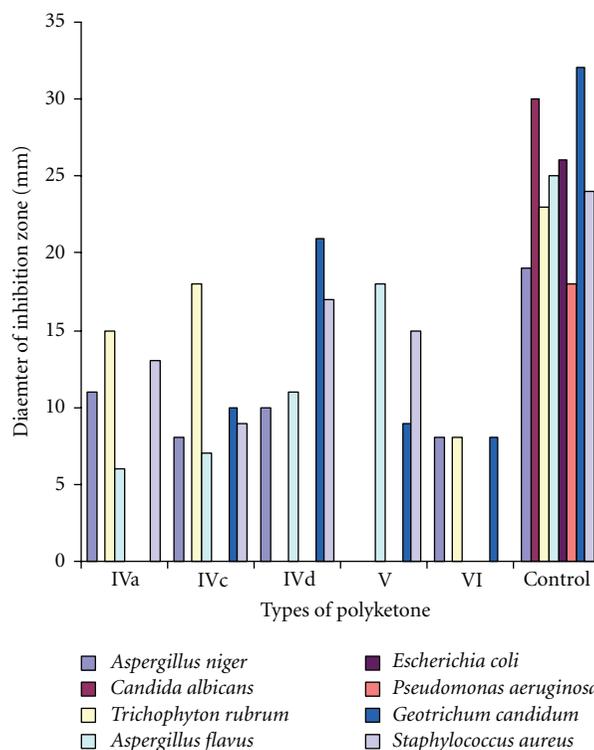


FIGURE 3: Effect of polyketones and copolyketones on the growth (%) of bacteria.

is needed to identify the moiety that is most important in the different structures for higher antimicrobial activity which will be done in our next work.

4. Conclusions

Two novel series of unsaturated polyketones and copolyketones based on 2,7-bis furfurylidene cycloheptanone have successfully been synthesized via Friedel-Crafts reaction. Polymers properties were strongly affected by their structural differences. In general, the introduction of the flexibilizing aliphatic linkages leads to polymer of a higher solubility in NMP. X-ray diffraction analyses indicated that most of the polymers are amorphous. All the prepared polymers are highly thermally stable and thermogravimetric analyses showed that the polyketones based aliphatic chains were somewhat less thermally stable than their polymers based aromatic. All the polymers showed antimicrobial activity.

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