ICC
Iranian Chemical Communication
Original Research Article
Payame Noor University http://icc.journals.pnu.ac.ir

Synthesis, characterization and polymerization of a novel acrylate monomer containing both 4*H*-pyran-4-one and 1,2,3-triazole moiety and evaluation of their antibacterial activity

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Received: 25 July 2016, Accepted: 15 May 2017, Published: 15 May 2017

Abstract

A novel acrylate monomer containing 4*H*-pyran-4-one and 1,2,3-triazole ring, {1-[4-(4-oxo-6-phenyl-4*H*-pyran-2-yl)benzyl]-1,2,3-triazol-4-yl}methyl acrylate was synthesized by the reaction of 2-{4-[(4-(hydroxymethyl)-1,2,3-triazol-1-yl)methyl]phenyl}-6-phenyl-4*H*-pyran-4-one with acryloyl chloride in the presence of triethylamine. The structure of the acrylate monomer was established on the basis of FT-IR, ^{1}H NMR, ^{13}C NMR, MS and elemental analysis. This monomer was polymerized using 2,2´-azobisisobutyronitrile (AIBN) as the initiator in *N*,*N*-dimethylformamide solution. Thermal stability of the polymer was investigated by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The synthesized compounds were evaluated for their antibacterial activity against Gram-positive and Gram-negative bacteria using the disk diffusion method. The results of antibacterial assay indicated that these compounds exhibited moderate bactericidal activity.

Keywords: 4*H*-pyran-4-one; 1,2,3-triazole; acrylate monomer; antibacterial activity; free radical polymerization; thermal properties.

Introduction

1,2,3-Triazoles and their derivatives can be considered as an important class of heterocyclic compounds that have attracted much attention due to their wide range of biological properties such as antibacterial, antiallergic, antifungal, antimicrobial, anti-HIV, antiviral, anticancer, antiinflammatory, and also their applications in drug discovery, bioconjugates and medicinal chemistry as well as materials chemistry [1-11]. In addition, a number of compounds containing 1,2,3-triazole moiety have found industrial applications as dyes, agrochemicals, photostabilizers, and corrosion inhibitors [12]. There are several methods for the synthesis of these compounds in the literature [13-15]. Huisgen's 1,3-dipolar cycloaddition reaction of organic azides with alkynes is mostly used for the synthesis of 1,2,3-triazoles [16]. The Cu(I) catalyzed cycloaddition reaction known as "click chemistry" was introduced by Sharpless and Meldal [17,18].

Meanwhile, 4*H*-pyran-4-one and its derivatives constitute a useful class of heterocyclic compounds, that are

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widely distributed in a variety of natural and synthetic biologically active compounds [19-21]. They have shown to possess anticancer, anti-HIV, antileishmanial, anticoagulant, antimicrobial and anticonvulsant activities [22-27].

Functional polymers have become increasingly important due to their various applications such as polymeric reagents and crosslinking resins, etc. [28,29]. These polymers can be prepared either by synthesizing new functional monomers and their subsequent polymerization or by converting functional groups on the polymer into the desired functional groups [30-33]. Functional groups give the polymer structure special characters substantially different from the inherent properties of the basic polymer chain [34]. Functional polymers derived from acrylates have attracted attention because of their wide applications in leather, adhesives, printing inks, lithography, coating, medical, paper and textiles industry [35-42].

There are many reports on the synthesis of functional monomers and polymers containing 1,2,3-triazole ring [43, 44]. At the best of our knowledge, the synthesis of monomers containing 4*H*-pyran-4-one and 1,2,3-triazole ring has not been reported. Herein, we wish to report the synthesis and characterization of a novel acrylate monomer containing both 4*H*-pyran-4-one and 1,2,3-triazole moiety which could have a biological activity. By the polymerization of this monomer, 4*H*-pyran-4-one and 1,2,3-triazole moiety were introduced into the polymer side chain. The thermal stability of the polymer was studied by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Antibacterial activity of the synthesized compounds were investigated against two Gram-positive and two Gram-negative bacteria.

Experimental

General

All reagents were purchased from Merck and Fluka companies and were used without further purification. Solvents were dried and distilled prior to use. The monomer was purified by preparative layer chromatography (PLC; Merck, silica gel 60 F_{254}, CAMAG, Switzerland) using n-hexane: acetone (4:3) as eluent. Melting points were determined on an Electrothermal Barnstead 9200 apparatus (Barnstead, UK) and are uncorrected. FT-IR spectra were obtained using KBr pellets on a tensor 27-Bruker spectrometer (Shimadzu, Japan). ^{1}H NMR and ^{13}C NMR spectra were recorded with a FT-NMR-Bruker spectrometer (Germany), at 400 and 100 MHz, respectively, in $CDCl_3$ and DMSO-d_6. Mass spectra were recorded using a direct insert probe Agilent Technologies 5975c mass spectrometer. Elemental analyses were carried out on Perkin-Elmer CHNS-O Analyzer, Model 2400 Series II. The thermogravimetric analysis (TGA) of the polymer was performed under nitrogen atmosphere at a heating rate of 10 ^{o}C min^{-1} using a Mettler-Toledo thermal analyzer. The glass transition temperature (T_g) of the polymer was measured using a differential scanning calorimeter (DSC), TGA/SDTA 851, under nitrogen atmosphere at a heating rate of 10 ^{o}C min^{-1}.

Synthesis of {1-[4-(4-oxo-6-phenyl-4*H*-pyran-2-yl)benzyl]-1,2,3-triazol-4-yl}methyl acrylate (3)

Method 1: To a solution of 2-{4-[(4-(hydroxymethyl)-1,2,3-triazol-1-yl)methyl]phenyl}-6-phenyl-4*H*-pyran-4-one 2 (0.1 g, 0.28 mmol) in 5 mL dry THF under nitrogen atmosphere was added triethylamine (0.14 g, 1.4 mmol)

and the reaction mixture was cooled to 0 °C using ice bath. Then, acryloyl chloride (0.127 g, 1.4 mmol) in 1 mL THF was added dropwise to above solution under 0 °C. The reaction mixture was stirred at 0 °C for 1 h and at room temperature for 24 h. The reaction progress was monitored by TLC. The precipitated triethylamine hydrochloride was filtered off and the solvent was removed under reduced pressure. The residue was purified by preparative layer chromatography (PLC) on silica gel with n-hexane: acetone (4:3) as an eluent to give the pure monomer. Cream solid, 0.05 g (43%), mp 155-157 °C; FT-IR (KBr): υ 3128, 2966, 1730 (ester C=O), 1647 (pyrone C=O), 1585, 1382, 1234, 1186, 948, 846, 783 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz): δ = 5.31 (s, 2H, -CH_2N-), 5.63 (s, 2H, -CH_2O-), 5.85 (dd, 1H, *J* = 0.8 and 12.0 Hz, alkene-H), 6.12 (dd, 1H, *J* = 12.0 and 16.0 Hz, alkene-H), 6.43 (dd, 1H, *J* = 0.8 and 16.0 Hz, alkene-H), 6.80 (d, 1H, *J* = 1.6 Hz, pyrone-H), 6.82 (d, 1H, *J* = 2.0 Hz, pyrone-H), 7.43 (d, 2H, *J* = 8.4 Hz, Ar-H), 7.53-7.55 (m, 3H, Ar-H), 7.66 (s, 1H, triazole-H), 7.83-7.88 (m, 4H, Ar-H) ppm; ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 53.6, 57.6, 111.5, 111.8, 124.0, 125.9, 126.7, 127.9, 128.7, 129.2, 131.2, 131.6, 131.7, 131.9, 137.8, 143.5, 162.4, 163.5, 166.0 (ester C=O), 180.1 (pyrone C=O) ppm; EIMS *m/z* 413 $[M]^+$ (3), 233 (100), 77 (50). Anal. Calcd. for $C_{24}H_{19}N_3O_4$: C, 69.72; H, 4.63; N, 10.16. Found: C, 69.89; H, 4.52; N, 10.07.

Method 2: To a solution of 2-{4-[(4-(hydroxymethyl)-1,2,3-triazol-1-yl)methyl]phenyl}-6-phenyl-4*H*-pyran-4-one 2 (0.1 g, 0.28 mmol) and acrylic acid (0.04 g, 0.56 mmol) in 10 mL CH_2Cl_2 under nitrogen atmosphere was added a solution of *N,N'*-dicyclohexylcarbodiimide (DCC) (0.064 g, 0.31 mmol) in 2 mL CH_2Cl_2. Then, a solution of 4-(*N,N*-dimethylamino)pyridine (DMAP) (0.034 g, 0.28 mmol) in 1 mL CH_2Cl_2 was added. The reaction mixture was heated at 70 °C for 7 days. After completion of the reaction, which was monitored by TLC, the precipitated dicyclohexyl urea was filtered off and the solvent was removed under reduced pressure. The residue was purified by PLC on silica gel with n-hexane: acetone (4:3) as an eluent to give the pure monomer in 42% yield.

Radical polymerization of monomer 3

A typical procedure for the synthesis of the polymer is described below:

To a solution of {1-[4-(4-oxo-6-phenyl-4*H*-pyran-2-yl)benzyl]-1,2,3-triazol-4-yl}methyl acrylate 3 (0.148 g, 0.358 mmol) in dry DMF (5 mL) under nitrogen atmosphere was added AIBN (0.012 g, 8% of the monomer concentration), and the reaction mixture was heated at 90 °C for 7 days. The polymer was precipitated by adding the reaction mixture into methanol. The polymer was purified by repeated reprecipitation of methanol from a solution of the polymer in DMF. The polymer was filtered off and dried in vacuum. Brown solid, 0.05 g (34%) FT-IR (KBr): υ 3006, 2960, 1741, 1650, 1517, 1429, 1363, 1263, 1218, 676 cm^{-1}; ^1H NMR (DMSO-d_6, 400 MHz) δ 1.57 (br, 2H, -CH_2- backbone), 2.22 (br, 1H, -CH- backbone), 2.72 (s, 2H, -CH_2N-), 2.88 (s, 2H, -CH_2O-), 6.89 (s, 2H, pyrone-H), 7.41-7.50 (m, 5H, Ar-H), 7.92-8.03 (m, 4H, Ar-H), 8.20 (s, 1H, triazole-H) ppm.

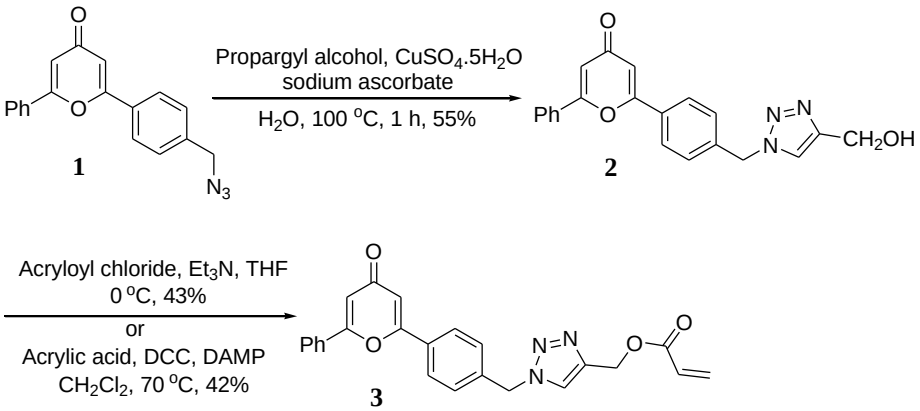
Antibacterial activity assay

Antibacterial activity of the synthesized compounds **2-4** was determined against two Gram-positive bacteria, *Staphylococcus aureus* and *Bacillus subtilis* and two Gram-negative

bacteria, *Escherichia coli* and *Salmonella typhi* by the standard disk diffusion method. Muller- Hinton agar (oxoid) was used for preparation of the media. The filter paper discs (6.4 mm indiameter) were individually impregnated with 10 µl of stock solution of the extracts (2 mg/disc) and then placed onto the agar plates which had previously been inoculated with the tested bacteria. The plates were inoculated with bacteria incubated at 37 °C for 18-24 h. The diameters of inhibition zones were measured in millimeters. All the tests were performed in duplicate. Gentamicin (30 µg) served as positive control.

Results and discussion

In this work, a novel acrylate monomer **3** was synthesized by the reaction of 2-{4-[(4-(hydroxymethyl)-1,2,3-triazol-1-yl)methyl]phenyl}-6-phenyl-4*H*-pyran-4-one **2** with acryloyl chloride or acrylic acid in 43% and 42% yields, respectively. For this purpose, 4*H*-pyran-4-one was prepared through cyclization of related 1,3,5-triketone under acidic conditions [45]. 2-(4-Bromomethylphenyl)-6-phenyl-4*H*-pyran-4-one was prepared by the reaction of 4*H*-pyran-4-one with *N*-bromosuccinimide (NBS) in dry CCl_4 [46], which was converted to 2-(4-azidomethylphenyl)-6-phenyl-4*H*-pyran-4-one **1** using sodium azide in dry DMF [47]. 1,4-Disubstituted 1,2,3-triazole **2** was prepared by the reaction of 2-(4-azidomethylphenyl)-6-phenyl-4*H*-pyran-4-one **1** with propargyl alcohol in the presence of $CuSO_4.5H_2O$ and sodium ascorbate in water at 100 ^oC for 1 h in 55% yield according to the literature (Scheme 1) [48].



1 **2** **3**

Scheme 1. Synthesis of 1-[4-(4-oxo-6-phenyl-4*H*-pyran-2-yl)benzyl]-1,2,3-triazol-4-yl}methyl acrylate **3**

The structure of the monomer was investigated by FT-IR, ^1H NMR, ^{13}C NMR, MS and elemental analyses. In the FT-IR spectrum of the monomer, a strong absorption band at 1730 cm^{-1} is assigned to the ester C=O stretching vibration. The absorption band at 1647 cm^{-1} is assigned to the pyrone C=O stretching vibration. In the ^1H NMR spectrum of monomer, the signals at 5.31 and 5.63 ppm are assigned to the -CH_2N- and -CH_2O- protons, respectively (Figure 1). The signals corresponding to the vinyl protons (CH_2=CH-) from acrylate unit are exhibited at 5.85, 6.12 and 6.43 ppm. The doublet at 6.80 and 6.82 ppm are assigned to the pyrone protons. Due to the aromatic protons the signals are exhibited at 7.43, 7.53-7.55 and 7.83-

7.88 ppm. A signal at 7.66 ppm is assigned to the triazole proton. The above results confirmed that the monomer was successfully synthesized.

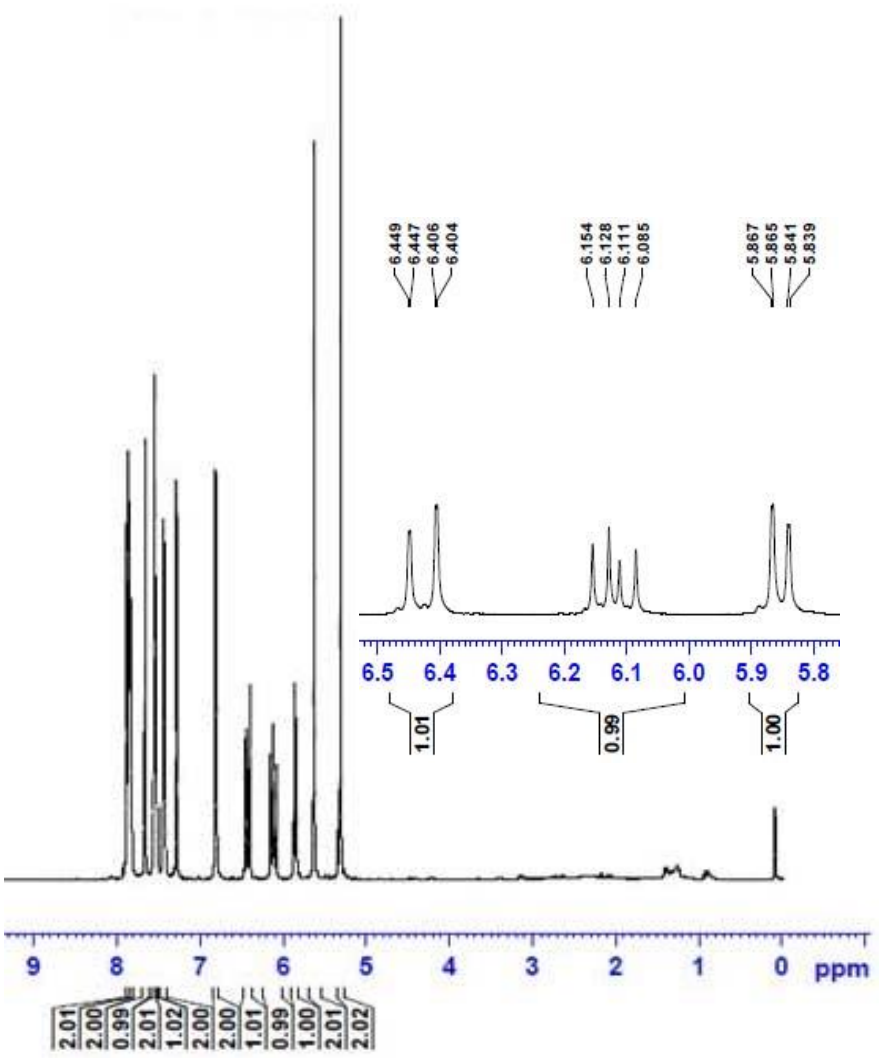
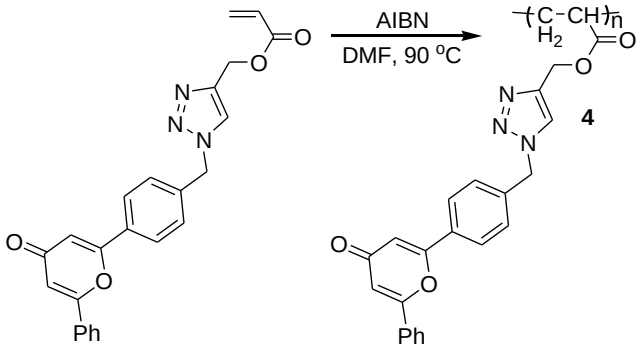


Figure 1. ^{1}H NMR spectrum of the acrylate monomer **3** in $CDCl_3$

The polymerization of the acrylate monomer **3** was carried out at 90 ^{o}C in dry DMF *via* free radical polymerization using AIBN as an initiator under nitrogen as shown in Scheme 2. By the polymerization of the monomer, 4*H*-pyran-4-one and 1,2,3-triazole unit were introduced into the polymer side chain. The structure of the resulting polymer was confirmed by FT-IR and ^{1}H NMR spectra. The FT-IR spectrum of the polymer **4** showed the absorption band at 1741cm^{-1} corresponding to the ester carbonyl group stretching vibration.



Scheme 2. Synthesis of the polymer **4**

In the ^{1}H NMR spectrum of the polymer, the signals at 1.57 and 2.22 ppm are assigned to -CH_2- and -CH- protons in the polymer chain, respectively. Due to the vinyl protons the signals at 5.85, 6.12 and 6.43 ppm have disappeared and the chemical shifts are well consistent with the polymer structure. The spectral data corresponding to other protons were in agremeent with the structure of the polymer.

The solubility of the polymer was tested qualitatively in various solvents. The polymer was soluble in *N*,*N*-dimethylformamide and dimethyl sulfoxide, but insoluble in chloroform, dichloromethane, tetrahydrofuran, 1,4-dioxane, methanol, ethanol, n-hexane, acetone, ethyl acetate, acetonitrile and toluene. The thermal behavior of the polymer was investigated by thermogravimetric analysis (TGA). TGA curve of the polymer is shown in Figure 2. The curve clearly indicates that weight loss of the polymer occurs in three stages. A weight loss in the temperature range of 130-240 °C may be due to the absorbed solvent. The second step of weight loss in the temperature range of 240-490 °C depends on the polymer structure and may be attributed to the decomposition of the polymer and the ester linkage. The final stage of weight loss in the temperature range of 490-850 °C may be attributed to the decomposition of the heterocyclic and the aromatic rings. The glass transition temperature (T_g) of the polymer was determined by DSC. The polymer was indicated as a single T_g at 123 °C.

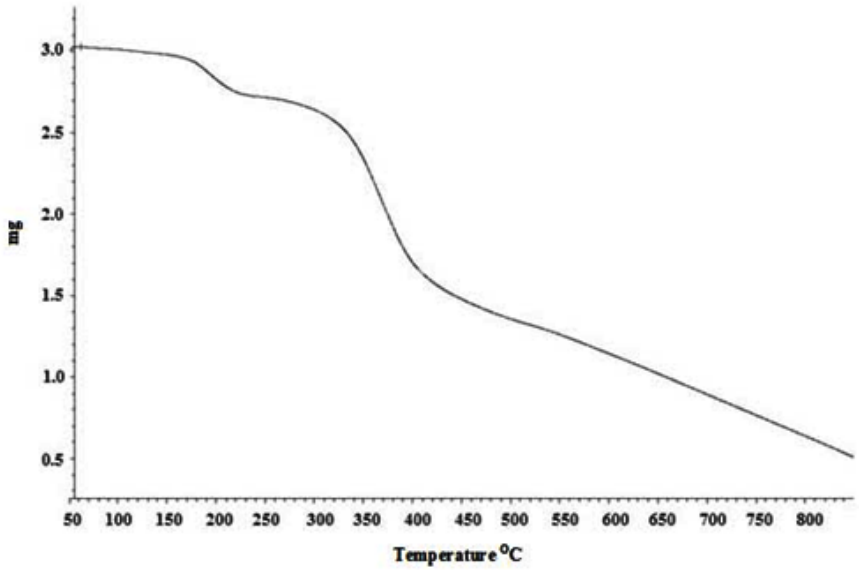


Figure 2. TGA curve of polymer **4**

The in-vitro antibacterial activity of the synthesized compounds **2-4** indicated moderate antibacterial activity on the Gram positive tested strains *Bacillus subtilis* (Figure 3) and *Staphylococcus aureus* with inhibition zones about (8-9 mm) and a weak antibacterial activity against the Gram negative tested strains *Escherichia coli* and *Salmonella typhi* with inhibition zones less than 8 mm. Our findings revealed that the difference in bactericidal activity in Gram positive and Gram negative bacteria may be related to the complex cell walls structure in Gram negative bacteria. Minimum inhibitory concentrations (MIC) values of the compounds were more than 1000 µg/mL.

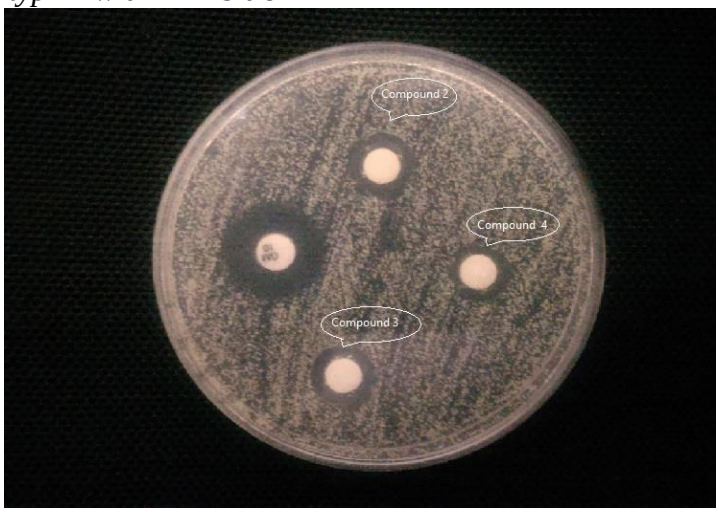


Figure 3. Inhibition zone of the synthesized compounds **2-4**, against *Bacillus subtilis* in disc diffusion method

Conclusion

In the present work, we have synthesized a novel acrylate monomer containing both 4*H*-pyran-4-one and 1,2,3-triazole moiety by the reaction of 2-{4-[(4-(hydroxymethyl)-1,2,3-triazol-1-yl)methyl]phenyl}-6-phenyl-4*H*-pyran-4-one with acryloyl chloride or

acrylic acid, and its structure was confirmed by FT-IR, NMR, MS and elemental analysis. The acrylate monomer was successfully polymerized by using AIBN as initiator. The thermal stability of the polymer was measured by TGA analysis and showed that the polymer has relatively high thermal stability. The antibacterial activity of the synthesized compounds was evaluated using the disk diffusion method and the results of antibacterial assay indicated that these compounds exhibited moderate bactericidal activity.

Acknowledgments

The authors thank the Research Council of Payame Noor University for financial support.

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