

# Synthesis, characterization polymerization and antibacterial properties of novel thiophene substituted acrylamide

Maher Z. Elsabee<sup>a</sup>, Eman A. Ali<sup>a</sup>, Samia M. Mokhtar<sup>b,\*</sup>, Mohamed Eweis<sup>c</sup>

<sup>a</sup> Department of Chemistry, Faculty of Science, Cairo University, Cairo 12613, Egypt

<sup>b</sup> Department of Chemistry, Faculty of Science, College of Women, Ein Shams University, Cairo, Egypt

<sup>c</sup> Department of Botany, Faculty of Science, Cairo University, Cairo 12613, Egypt

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## ABSTRACT

Ethyl 2-acrylamido-4,5,6,7-tetrahydrobenzo [b] thiophene-3-carboxylate (ETTCA) has been synthesized and its structure has been elucidated by elemental analysis and spectral tools. Free radical polymerization of (ETTCA) has been conducted in several solvents using azobisisobutyronitrile (AIBN) as an initiator. The kinetic parameters of polymerization of the ETTCA were investigated, and it was found that the polymerization reaction follows the conventional free radical scheme. The overall activation energy of polymerization  $\Delta E$  was determined ( $\Delta E = 45.11 \text{ kJ mol}^{-1}$ ). The copolymerization of ETTCA with three conventional monomers was carried out in dioxane at 65 °C. The monomer reactivity ratios for the copolymerization of ETTCA with methyl methacrylate (MMA), vinyl acetate (VA) and vinyl ether (VE) were calculated. Thermal stability of the ETTCA polymer and its copolymers were investigated by thermogravimetric analysis. It has been found that the prepared polymer (PETTCA) and its copolymers with VA have moderate biological activity and highly dependent on the copolymer composition.

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## 1. Introduction

The synthesis of polymers with well-defined compositions, architectures, and functionalities has become an important topic of contemporary polymer science. N-substituted acrylates are very interesting monomers because they have acquired prime importance in various avenues of application. Recently acrylamide derivatives were found to have antiviral activity as inhibitors of hepatitis B virus replication [1]. Moreover, the N-substituted acrylamides are used to prepare thermosensitive materials. These thermoplastic polymers present also great potential in application as drug delivery system [2], as glycogen phosphorylase inhibitors [3] human gene vectors [4] and biocatalysts [5]. It is possible to obtain N-acryloyl and N-methacryloyl derivatives of human serum albumin (HSA), in which acryloyl fragments are bound to asparagines and lyzin fragments [6]. The development of antimicrobial macromolecules holds a good promise for novel therapeutics and new materials to prevent the spread of infectious disease [7].

The reaction of acryloyl chloride or methylacryloyl chloride with the corresponding amines to prepare new functional monomers has been reported [8–10]. Many investigations have focused

on the synthesis and characterization of functional copolymers with physical, chemical, and electrical stimuli properties which can respond to different environmental conditions. The present work deals with the preparation of a novel acrylamide derivatives which contains a thiophene moiety which could have a biological activity. The new monomer has been characterized and copolymerized with different conventional monomers, the antifungal and antibacterial behavior of one copolymer with vinyl acetate has been investigated against several fungi and bacteria.

## 2. Experimental

### 2.1. Materials

Cyclohexanone, ethyl cyanoacetate, elemental sulfur, triethylamine and acryloyl chloride were used as received. Methyl methacrylate (MMA) was purified by washing with 50 ml of 5% NaOH several times followed by distilled water. Finally, the washed methyl methacrylate was dried with anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ ), filtered and distilled before use. Vinyl ether (VE) and vinyl acetate (VA) (Aldrich) were distilled before use. All the reagents and solvents were purified by conventional methods. Azobisisobutyronitrile (AIBN) was purified by recrystallization from methanol and then dried in the dark.

\* Corresponding author. Mobile: +20 2 0106609863.

E-mail address: [samiamokhtar@hotmail.com](mailto:samiamokhtar@hotmail.com) (S.M. Mokhtar).

## 2.2. Monomer synthesis

### 2.2.1. Preparation of ethyl 2-amino-4,5,6,7-tetrahydrobenzo [b] thiophene-3-carboxylate

A mixture of 39.2 g (0.4 mol) of cyclohexanone, 45.2 g (0.4 mol) ethylcyano acetate, 12.8 g (0.4 mol) elemental sulfur and 40.4 g (0.4 mol) triethylamine was refluxed in absolute ethanol for two hours. The reaction mixture was poured into cold water. The solid product was filtrated and recrystallized from ethanol. The yield was 80%, and the golden crystals with melting point 112 °C were collected (Scheme 1).

Elemental analyses (%) were: found (theoretical), C = 58.66 (58.64%), H = 6.97 (6.71%), N = 6.17 (6.22)% and S = 14.15 (14.23)%.

### 2.2.2. Preparation of ethyl 2-amino-4,5,6,7-tetrahydrobenzo [b] thiophene-3-carboxylate acrylamide (ETTCA)

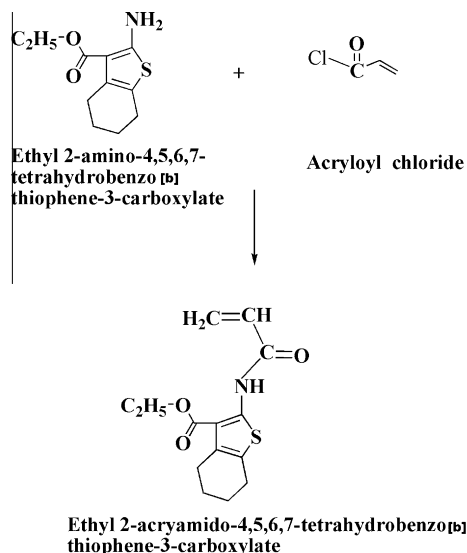
24.7 g (0.1 mol) ethyl 2-amino-4,5,6,7 tetrahydrobenzo thiophene-3-carboxylate was dissolved in diethyl ether, and in the same solvent acryloyl chloride 9.05 g (0.1 mol) was added drop wise. The mixture was maintained at low temperature 4 °C using an ice bath and stirred for 5–7 h. The resulting product was poured into water, stirred and washed with diethyl ether, then dried at room temperature. The yield was 70%, and the pale brown crystals melting point was 115 °C. Scheme 2 describes the synthesis of ETTCA monomer.

Elemental analysis: found (theoretical) C = 59.34 (60.19)%, H = 6.44 (6.13)% N = 5.06 (5.01)% and S = 11.09 (11.48)%.

## 2.3. Homopolymerization and copolymerization

Radical solution polymerization of ETTCA was carried out in sealed glass tubes with the proper solvent (tetrahydrofurane THF or 1,4-dioxane) and initiator AIBN at 65 °C. The polymerizations were homogeneous in all cases.

Copolymerization of ETTCA with MMA, VA and VE was carried out following the above procedure. The composition of the copolymers was determined by elemental analysis.



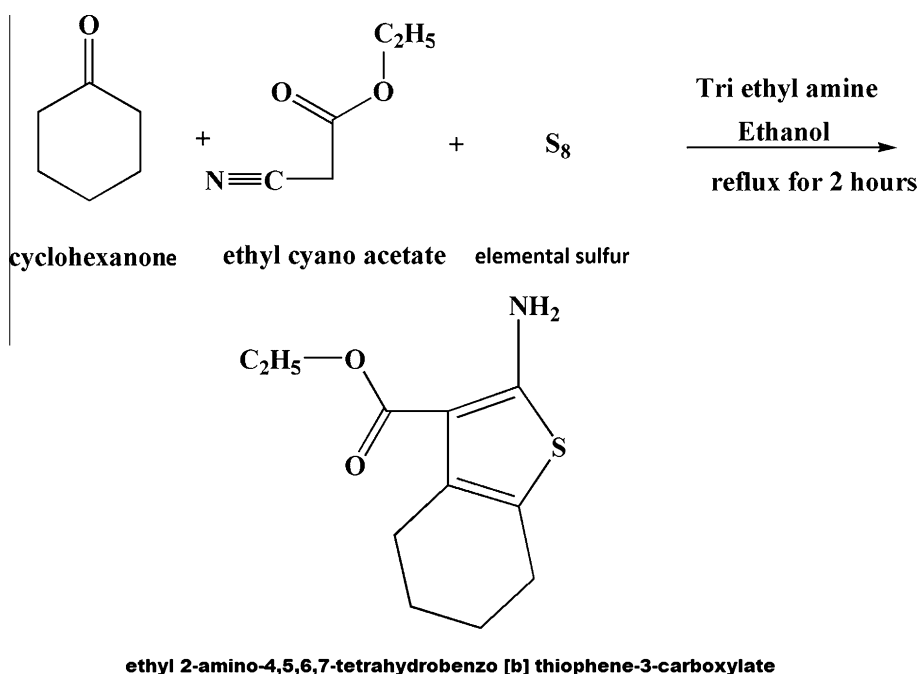
**Scheme 2.** Preparation of Ethyl 2-acrylamido-4,5,6,7-tetrahydrobenzo [b] thiophene-3-carboxylate (ETTCA).

## 2.4. Thermal analysis, NMR, FTIR

Thermogravimetric analysis TGA studies were performed on a Shimadzu TGA-50H instrument. Typically 7–10 mg samples were heated at a rate of 10.0 °C/min in nitrogen atmosphere. FTIR infrared measurement was obtained using a Perkin–Elmer 398 FTIR spectrophotometer between 400 and 4000  $\text{cm}^{-1}$ .  $^1\text{H}$  NMR spectra of the samples were obtained on a Bruker AC-400 at 20 °C in  $\text{CDCl}_3$  solution.

## 2.5. Molecular weight determination

The viscosity measurements were carried out using an Ubbelohde viscometer suspended level dilution viscometer. Dioxane



**Scheme 1.** Preparation of ethyl 2-amino-4,5,6,7-tetrahydrobenzo [b] thiophene-3-carboxylate (EATTC).

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