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Synthesis and Characterization of Novel ***N-(2-Benzoyl-benzofuran-3-yl)-acrylamide*** **monomer**

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In this study, a new acrylamide monomer was synthesized and characterized. The new substance synthesized in this study was the *N*-substituted acrylamide monomer *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide monomer (NBZA). This substance was synthesized through the reaction of (3-Amino-benzofuran-2-yl)-phenyl-methanone (which was synthesized by the reaction of 2-hydroxybenzonitrile with 2-bromoacetophenone under basic conditions) with acryloyl chloride at a temperature of 0-5 ^{o}C. Nuclear magnetic resonance technique (^{1}H-NMR) and infrared (FT-IR) studies were utilized to spectroscopically characterize the chemical structure of *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide monomer (NBZA). In addition, the maximum absorption wavelength value of the substance was determined by UV-VIS spectrum.

Keywords: *Benzofuran, acrylamide, N-substituted acrylamide monomer.*

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1. Introduction

Because of their biological activity and applications in material science, benzofurans have an important role in chemistry. In general, they are used as antitumor [1], anti-microbial [2], kinase inhibitory and antihyperglycemic [3,4], anti-cancer, antiviral [5], analgesic [6], antihyperglycemic [7], antiparasitic [8], anti-inflammatory [9], antibacterial, antifungal and anticonvulsant agents [10] in pharmaceutical chemistry. Moreover, compounds that include benzofuran moieties demonstrated a potential to suppress Alzheimer's activity according to literature surveys based on the framework of ACHE (American College of Healthcare Executives) [11-13]. Due to their properties of blue-light emitting [14], electrochemical behavior and thermal stability [15, 16], benzofurans are commonly used in material science as materials for organic electroluminescence (OEL is an emerging display technology allowing the manufacture of efficient, low-voltage multicolor displays), [17] and layered organic light-emitting diodes (OLEDs are used to create digital displays in devices such as television screens, computer monitors, and portable systems such as smartphones and handheld game consoles) [18]. Benzofuran and its derivatives are still being widely studied. For example; Trang et al. studied the physicochemical and structural properties of four compounds containing benzofuran. Among the compounds studied, it was stated that non-planar compound (E)-5-(2-(2-(4-hydroxyphenyl)benzofuran-5-yl)vinyl)benzene-1,3-diol had the most superior properties. In particular, it was found that the antioxidant capacity of the aforementioned compound was greater than other compounds [19]. Tudos et al. produced two new composites based on benzofuran derivatives and graphene oxide. They reported that these composites had mild toxicity on tumor cells and emphasized that these composites can be developed and used in biomedical applications [20]. Ganea et al. synthesized a functional polymer called poly(benzofuran-co-arylacetic acid) and characterized it by producing a composite of the compound with the magnetic nanoparticle Fe3O4. Ganea et al. investigated the adsorption property of this composite. According to the results, it was stated that the composite can be used in waste water treatment [21].

Acrylamide (2-Propenamide) is utilized to synthesize polyacrylamide and it is the monomer form [22]. It is difficult to analyze acrylamide individually in complex matrixes by employing classical analytical techniques since it is a polar, hydrophilic, and unsaturated amide [23, 24]. At high temperature and low moisture, acrylamide naturally gets produced as a byproduct of the cooking process of foods which are rich in carbohydrate [25].

A great number of studies have been done on benzofuran in the literature. However, studies on acrylamide containing benzofuran derivatives are limited in number. Both benzofuran and acrylamide structures exhibit various properties. Therefore, the synthesis of structures containing both of these compounds is important. By doing so, these substances can be examined more deeply and they can be used in some areas. This will also ease the further synthesis of similar compounds.

2. Experimental

2.1. Materials and analytical techniques

For the synthesis of the monomer, 2-bromoacetophenone, potassium carbonate, 2-hydroxybenzonitrile, acryloyl chloride were procured from Sigma Aldrich company. Triethylamine (Sigma-Aldrich), acetone (Sigma-Aldrich) and ethanol (Sigma-Aldrich) were purified adequately.

A Perkin Elmer Spectrum 100 model Fourier-transform infrared (FT-IR) spectrometer was utilized for structural characterization of substances. The monomer's ^{1}H-NMR spectrum was recorded in $CDCl_3$ using an AGILENT 400 MHz NMR spectrometer. UV-VIS spectrum of the monomer was recorded in ethanol using a PerkinElmer Lambda 25 UV/VIS spectrometer.

2.2. Synthesis of N-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

Firstly, (3-Amino-benzofuran-2-yl)-phenyl-methanone to be utilized in the monomer synthesis was synthesized as follows: In solvent of absolute acetone (100 mL), 2-bromoacetofenone (0.005 mol), 2-hydroxybenzonitrile (0.005 mol) and potassium carbonate (0.0075 mol) were refluxed for 12 hours. Then, the mixture was stirred at room temperature for 24h. The reaction mixture was added into water. The title compound was filtered, washed with water and then dried and crystallized with ethanol. Later, (3-Amino-benzofuran-2-yl)-phenyl-methanone (0.01 mol) and triethylamine (0.011 mol) were dissolved in 50 ml of dry acetone in a 100 mL three-necked flask and the mixture was cooled to 0 ºC. Then, acryloyl chloride (0.011 mol) was added to the acetone mixture drop-wise while stirring constantly at 0 ºC. The mixture was stirred continuously for 2 hours at 0 ºC and at room temperature for 12 hours. After, the reaction mixture was added into cold water. The title compound was filtered and the monomer was dried in vacuum for 24 hours. In order to obtain shining light yellow crystals, the compound was crystallized from ethanol (Melting point: 125 ºC). Fig. 1 demonstrates the reaction schemes of this synthesis.

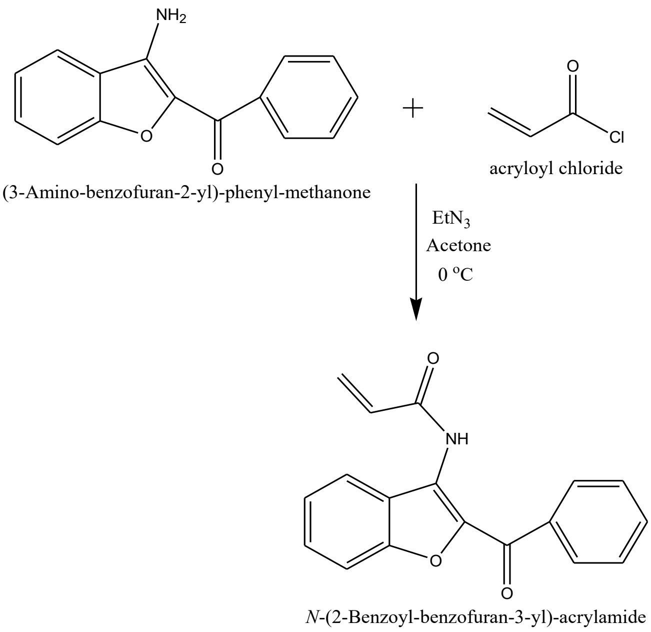

Fig.1. The synthesis scheme of *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

3. Results and Discussion

3.1. FT-IR and ^{1}H-NMR analysis of N-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

The structural characterization of the NBZA monomer synthesized in this study was conducted through FT-IR and ^{1}H-NMR (Fig. 3) spectroscopic methods. In the FT-IR spectrum presented in Fig. 2, peaks at 3285 cm^{-1}, 1671 cm^{-1}, 1627 cm-1, 1610 $cm^{-1}$and 1074 cm^{-1} that belong to symmetric NH stretching, symmetric C=O stretching (in the amide group), ketone carbonyl stretching, C=C stretching, and C-O-C stretching, respectively, are the characteristic peaks of the monomer.

The ^{1}H-NMR spectrum of the monomer was taken in $CDCl_3$ solution (Fig. 3). Peaks of ^{1}H NMR (400 MHz,) δ 10.94 (1H, N*H*), 8.62-7.25(9H, Ar H), 6.52-6.37 (2H, CO-C*H* and =C*H*), 5.86(1H, =C*H*) are the peaks that characterize the monomer [26, 27].

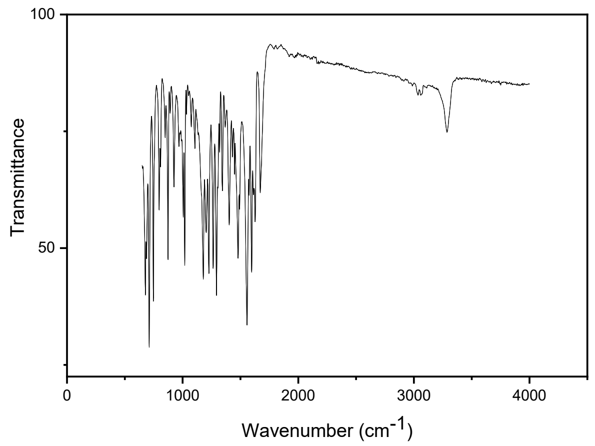

Fig.2. FT-IR spectrum of N-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

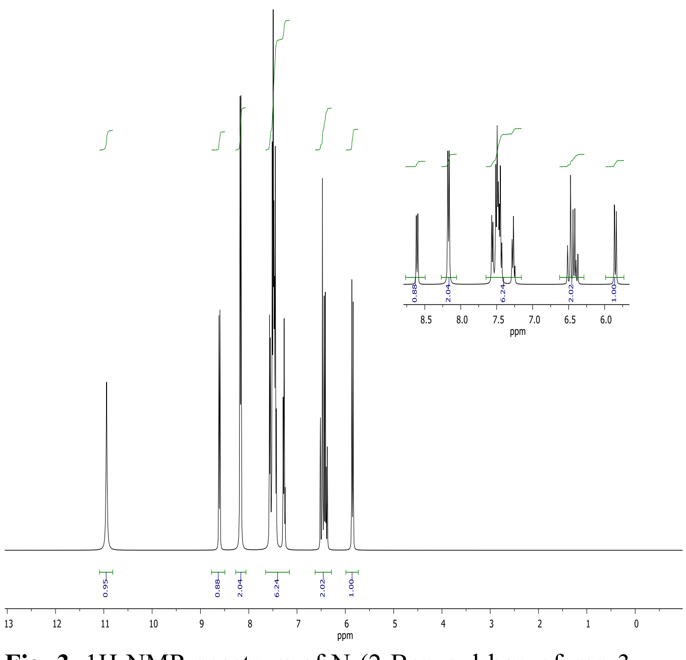

Fig. 3. 1H-NMR spectrum of N-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

3.2. UV-VIS analysis of N-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

The UV-VIS spectrum of the *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer was taken in the spectrum range of 200-500 nm and in ethanol solvent. In order to avoid a possible error, the analysis was repeated several times, and the resulting spectrum was presented in Fig. 4. The maximum wavelength value of the NBZA monomer determined via the UV-VIS spectrum was 332 nm. In a study conducted by A. Veeraiah, the absorption spectrum of 2-(bromoacetyl)benzo(b)furan was taken in methanol solution. The maximum wavelength value for this structure was found as 353 nm [28]. The wavelength value with the highest absorption may vary according to the groups included the compound's structure and the solvent used.

C.V. Maridevarmath et al. have synthesized and characterized the structures of (6-methyl-benzofuran-3-yl)-acetic acid hydrazide and (6-methoxy-benzofuran-3-yl)-acetic acid hydrazide. In their study, C.V. Maridevarmath et al. theoretically studied the absorption and fluorescence behavior of these benzofuran derivatives in different solvents. They emphasized that the studied substances can be used in the field of luminescence and optics in the future [29]. The examination of nonlinear optical properties of structures containing benzofuran is important for the use of benzofuran derivatives in the field of optics.

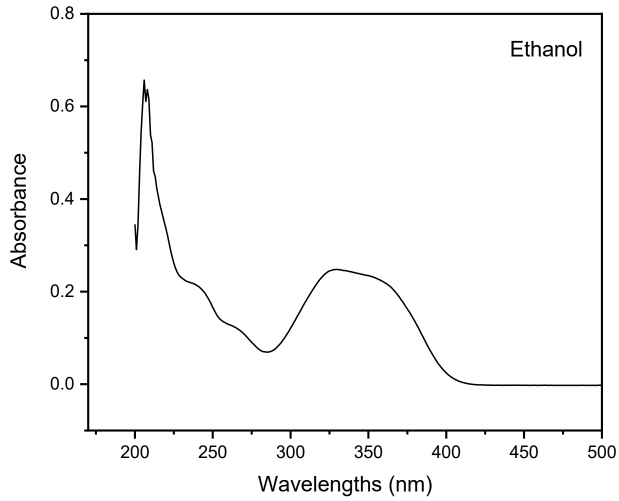

Fig. 4. UV-VIS spectrum of *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) monomer

4. Conclusion

In this study, a new acrylamide monomer named *N*-(2-Benzoyl-benzofuran-3-yl)-acrylamide (NBZA) that includes a benzofuran ring in its side chain was synthesized and characterized. For characterization, FT-IR and ^{1}H-NMR techniques were used. Peaks at 3285 cm^{-1}, 1671 cm^{-1}, 1627 cm-1, 1610 $cm^{-1}$and 1074 cm^{-1} representing NH stretching vibration, amide carbonyl stretching vibration, ketone carbonyl stretching vibration, C=C stretching vibration and C-O-C stretching vibration , respectively, which can be seen in the FT-IR spectrum presented, prove that the monomer has been successfully synthesized. When the ^{1}H-NMR spectrum of the compound is examined, it can be seen that the NH proton detected at 10.94 ppm and protons belonging to vinyl groups detected at 6.52-6.37 and 5.86 ppm are present. This further proves the success of the synthesis of the monomer. The maximum wavelength value of this acrylamide monomer containing a benzofuran side chain determined by UV-VIS absorption spectrum is compatible with the literature.

N-mono substituted acrylamide monomers and copolymers have an important place in organic polymer synthesis. When the various use cases of acrylamide monomers and the new properties that the benzofuran ring will add to this monomer is considered, it can be seen that it is likely this new monomer synthesized will contribute to the literature and perhaps find a use case.

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