

# Synthesis, Characterization, and Photoluminescence Study of Copolymer Derived From 2-Amino 6-nitrobenzothiazole and Oxamide with Formaldehyde

Punam G. Gupta<sup>1</sup>, R. H. Gupta<sup>2</sup>, W. B. Gurnule<sup>3</sup>

Department of Chemistry, Kamla Nehru Mahavidyalaya, Sakkardara, Nagpur, Maharashtra, India<sup>1</sup>

Department of Chemistry, K. Z. S. Science College, Kalmeshwar, Nagpur, Maharashtra, India<sup>2,3</sup>

punamgupta04@gmail.com<sup>1</sup> and wbgurnule@yahoo.co.in<sup>3</sup>

**Abstract:** *The current research article describes the synthesis of BOF-III copolymer from 2-amino 6-nitrobenzothiazole, oxamide, and formaldehyde used as a monomer in 3:1:5 molar ratios using polycondensation polymerization method in presence of 2M HCl as acid catalyst. The structure of the synthesized copolymer was characterized using elemental analysis and spectral techniques such as UV-Visible, FT-IR, and <sup>1</sup>H-NMR spectroscopy. The molecular weight of the copolymer was measured using a non-aqueous conductometric titration method. Scanning electron microscopy was used to investigate the surface morphology of a copolymer. The RF-501 (PC) S CE (LVD) MODEL PL spectrometer was used to evaluate the photoluminescence properties of newly synthesized copolymer. With significant input from current researchers in the field, the overall purpose of this development is to produce new polymeric material and analyze their photo luminescent properties.*

**Keywords:** Copolymer, Elemental Analysis, Spectroscopy, Photoluminescence

## I. INTRODUCTION

Organic polymers are attractive materials for use as the active layer in optoelectronic devices including field-effect transistors (FETs) [1], light-emitting diodes (LEDs) [2], and photovoltaic cells[3], as well as in flexible displays[4], solar panels[5], and smart materials [6].The majority of organic semiconductors are made up of  $\pi$ -conjugated molecules of various sizes, ranging from tiny molecules to polymers. The present challenge for the research world as well as industrial areas is to design innovative conjugated polymers with substantial optoelectronic capabilities [7].Conjugated polymers with a large  $\pi$ -conjugated structure have gained a lot of attention as multifunctional materials in recent decades because of their potential uses in chemical and biological devices [8] Copolymers containing  $\pi$ -conjugated bridges show significant promise as building blocks for carbon-rich networks, which could be useful in the creation of new optical materials and optoelectronic devices [9]. Because of their organic nature, they can be formed into flexible, lightweight materials that can be processed rapidly, making them ideal for low-power, low-cost applications[10]. Organic materials have several advantages over inorganic semiconductors, including inexpensive production and processing costs, flexibility, and light weight.

For full-color displays, high-efficiency red, green, and blue emissions are required [11]. Blue colour purity and consistency remain a challenge. However, compared to red or green emitting materials, blue emitting materials have major challenges with lower efficiency and shorter lifetime due to difficulties in hole and electron injection with greater band gaps [12]. Chemical and thermal stability of doped conjugated polymers are key concerns when evaluating possible long-term uses of organic conducting polymers [13]. Our ongoing exploration of several classes of  $\pi$ -conjugated polymers has been inspired by the promise of  $\pi$ -conjugated polymers as sophisticated materials for applications in electronics, optoelectronics, and nonlinear optics [14].One of the most intriguing features of organic materials is the able to develop a wide range of emission colour, notably in the blue region of EL displays, according to molecular design [15] [16]. Photoluminescence spectroscopy (PL) is a nondestructive, noncontact way of probing materials in which light energy or photons are passed through the sample, absorbed, and excess light energy is imparted into the sample. It is a strong approach for characterization and analysis of the electrical structure of intrinsic and extrinsic semiconducting and

semiinsulating materials. Many copolymers have been produced and successfully employed as electron-transporting and emitting materials in OLEDs, selective luminescent sensors, and nonlinear optical materials throughout the last three decades. W. B. Gurnule and co-workers studied the photoluminescence spectra of co-polymeric metal complexes 8-hydroxyquinoline-1,6-diamino hexane-formaldehyde with  $\text{Cu}^+$ ,  $\text{Ni}^{+2}$ , and  $\text{Zn}^{+2}$  [17]. P. Kumari and coordinates studies the luminescence properties of poly(3-hexyl thiophene) and poly(N-isopropylacrylamide) copolymers and this it was observed that synthesized copolymers are promising material for sensor, fluorescence thermometer, optoelectronics, and bioelectronics devices [18]. The photoluminescent properties of poly(styrene-*b*-2-(N,N-dimethylamino)ethyl methacrylate) diblock copolymers were studied by S. Basu and et.al. [19]. Using the Horner-Emmons condensation polymerization technique, G. Vidya and colleagues created a PPV block copolymer for blue-light emitting diodes [20]. S.B. Raut and co-workers study the photoluminescent properties of polymeric compound synthesized from trichloro-DDQ with PMMA, the light emitting and optoelectronic property make them useful material for electroluminescence, sensors and OLED applications [21].

This work focuses on the photoluminescence analysis of a copolymer synthesized by polycondensation polymerization of 2-amino 6-nitrobenzothiazole and oxamide with formaldehyde in 3:1:5 molar ratios. The molecular weight was calculated using a non-aqueous conductometric titration method, and scanning electron microscopy was utilised to analyse the surface morphology of a copolymer. UV-Visible, proton NMR, FTIR, and photoluminescence spectra were all discussed in the results and discussion section.

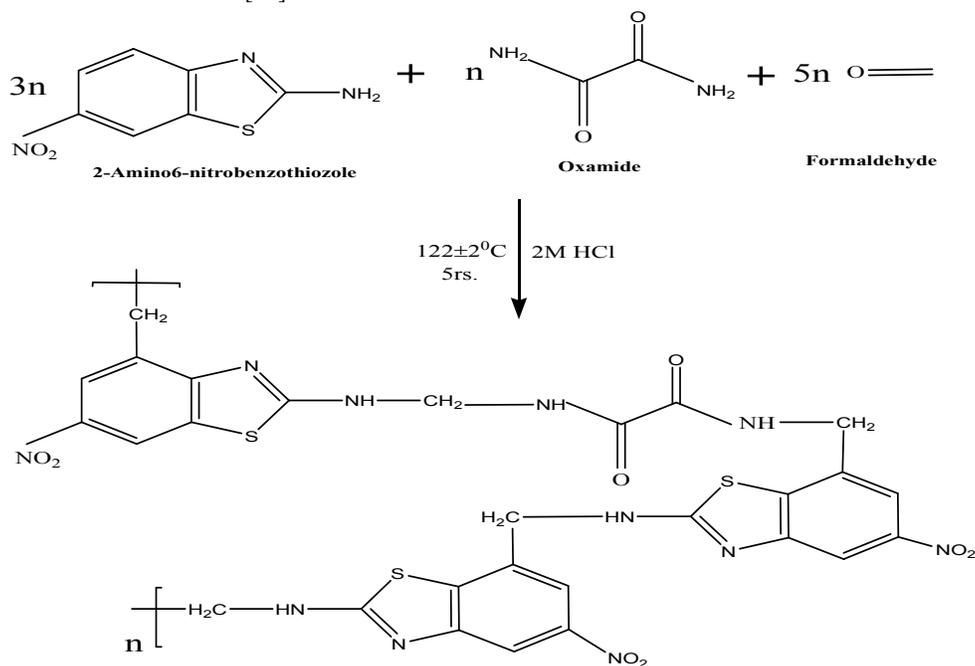
## II. MATERIALS AND METHODS

### 2.1 Materials

For the synthesis chemically pure and analytical grade chemicals and solvents were used. 2-amino-6-nitrobenzothiazole (Sigma Aldrich), Oxamide (Loba), Formaldehyde (Merk 37%), Dimethyl sulphoxide (99.8%, Fisher Scientific), Dimethyl formamide (Sigma Aldrich) (99 percent Genni Chem). All of the experiments were done with double-distilled water.

### 2.2 Synthesis of BOF-III Copolymer

The BOF-III copolymer was obtained by condensing 2-amino 6-nitrobenzothiazole (4.99g, 0.3 mole), oxamide (0.88g, 0.1 mole), and formaldehyde (18.75 mL, 0.5 mole) in 3:1:5 molar ratios in the presence of 2 M HCl (200 mL) as a catalyst at  $122 \pm 2^\circ\text{C}$  in an oil bath for 5 hours [21].



Scheme 1: Schematic representation of the synthesis of BOF-III copolymer

The pale yellow solid product was removed from the flask as soon as the reaction period was over. It was cleaned, dried, and powdered in cold water. The powder was rinsed many times with cold water to remove unreacted monomers. It was purified further by dissolving it in 8% NaOH and then filtering it. The copolymer was then precipitated by adding 1:1 (v/v) conc. HCl/water drop by drop with constant stirring and filtration. To obtain the pure copolymer, the procedure was repeated multiple times. The polymer sample was then rinsed in hot water, air dried, powdered, and kept in a vacuum desiccator with silica gel. The synthesis of BOF-III copolymer is depicted schematically in Scheme 1.

### III. CHARACTERIZATIONS

#### 3.1 Physicochemical and Elemental Analysis

The elemental analyzer Vario EL III (Elementar, Germany) was used to perform the elemental analysis and determine the percentage of components present in BOF-III copolymers, including carbon (C), hydrogen (H), nitrogen (N), and sulphur (S). Using conductometric titration in DMSO medium with ethanolic KOH as the titrant and 50 mg of sample, the average molecular weight was obtained. A graph of specific conductance vs. the milliequivalents of KOH required to neutralize 100 g of polymer was made. There were a lot of breaks in the plot. This graph was used to find the first and last breaks. The calculations for this method are based on the following principles. Based on the average degree of polymerization, the average molecular weight must be computed.

$$\overline{DP} = \frac{\text{Total milliequivalents of base required for complete neutralization}}{\text{Milliequivalents of base required for smallest interval}}$$

$$\overline{Mn} = \overline{DP} \times \text{Repeat unit weight}$$

#### 3.2 Spectral and Surface Analysis

A Shimadzu twin beam spectrophotometer UV-1800 was used at Shivaji Science College in Nagpur to scan the UV-Visible spectra of prepared samples copolymer in the 200–800 nm range. The infrared spectra of BOF-III copolymer were recorded in the range of 500-4000 $\text{cm}^{-1}$  using a Bruker Alpha –E spectrophotometer at RUSA Centre for Bio-Actives and Natural Products, Rashtrasant Tukadoji Maharaj Nagpur University in Nagpur. Sophisticated Analytical Instrumentation Facility at Punjab University, Chandigarh, scanned the proton nuclear magnetic resonance ( $^1\text{H-NMR}$ ) spectra of the copolymer using a Bruker Avance-II 400 MHz NMR Spectrometer with DMSO- $d_6$  as the solvent. At STIC, Cochin University, Cochin, the surface morphology of the BOF-III copolymer was studied at various magnifications using Jeol 6390 LV. The photoluminescence properties of copolymer were recorded using the Shimadzu Model RF-501 (PC) S CE (LVD) LS55.

### IV. RESULTS AND DISCUSSION

#### 4.1 Physicochemical and Elemental Analysis

The newly blended BOF-III copolymer was found to be yellow in color. The copolymer is soluble in solvents such as DMF, DMSO, THF and conc.  $\text{H}_2\text{SO}_4$  while insoluble in almost all inorganic and organic solvents. The yield of the copolymer was found to be 82 %.

**Table 1:** The Physicochemical and Analytical Data of the BOF-III Copolymer

Copolymer	The empirical formula of repeating unit	Empirical formula weight	%C Found (Cal.)	% H Found (Cal.)	% N Found (Cal.)	% S Found (Cal.)
BOF-III	$\text{C}_{28}\text{H}_{21}\text{N}_{11}\text{O}_8\text{S}_3$	735	46.07(46.85)	3.05(3.09)	20.14(20.57)	10.18(11.84)

The percentages of carbon, nitrogen, sulphur, and hydrogen in the sample were determined by elemental analysis, as indicated in Table I. The empirical formula and empirical weight of repeating unit of BOF-III copolymer were found to be  $\text{C}_{28}\text{H}_{21}\text{N}_{11}\text{O}_8\text{S}_3$  and 735 correspondingly, and the composition of copolymer derived using elemental analysis data was found to be in good agreement with the calculated values[22].

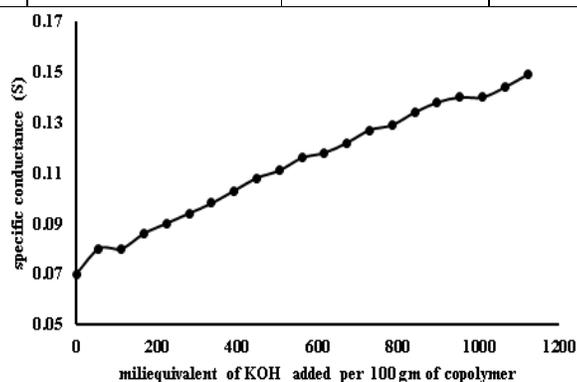
The nonaqueous conductometric titration method in DMSO medium employing standard potassium hydroxide (0.05M) in absolute ethanol as a titrant was used to measure the number average molecular weight ( $\overline{Mn}$ ) of this copolymer



(Table II). The milliequivalents of ethanolic KOH required to neutralize 100g of each copolymer were plotted against specific conductance. The number of plot breaks was found via plotting. Fig. 1 depicts the first break at 56 milliequivalents of base and the last break at 1008 milliequivalents of base. By multiplying the number average molecular weight ( $\overline{M}_n$ ) by the formula weight of the repeating unit, the average degree of polymerization ( $\overline{DP}$ ) of a copolymer can be calculated. Previous researchers have demonstrated that determining the number average molecular weight using non-aqueous conductometric titration is a straightforward and effective method [23] [24].

**Table 2:** Molecular weight determination of BDF-III copolymer

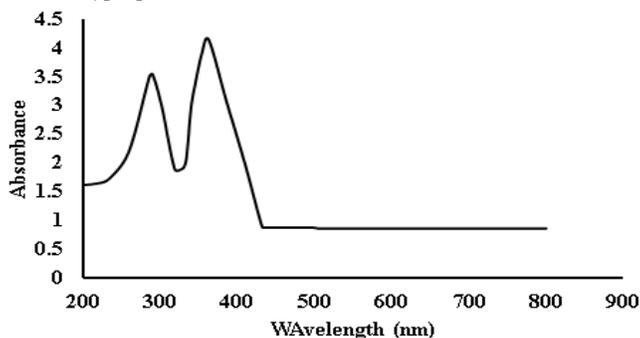
Copolymer	1 <sup>st</sup> phase of neutralization	The final phase of neutralization (Meq/100g sample)	Degree of polymerization ( $\overline{DP}$ )	Empirical weight (gm)	Number average molecular weight ( $\overline{M}_n$ )
BOF-II	56	1008	18.0	772	13896



**Figure 1:** Conductometric titration curve of BOF-III copolymer

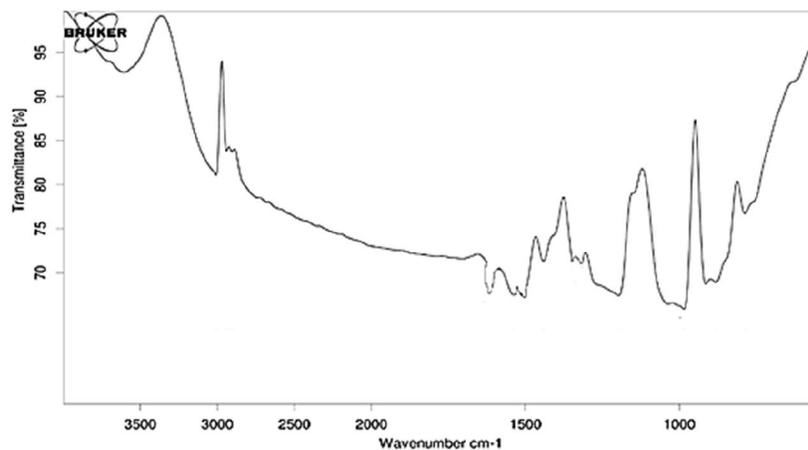
#### 4.2 Spectral and Surface Analysis

The UV-visible spectra of the BOF-III copolymer were recorded in DMSO solvent in the 200-800nm range. The newly produced BOF-III copolymer, which was displayed in Fig. 2, has two distinct bands at 280 nm and 370nm. At their observed positions, the absorption bands have different intensities. The band formed at 280 nm is less intense due to the allowed transition ( $\pi \rightarrow \pi^*$ ). The allowed  $\pi \rightarrow \pi^*$  transition is obtained due to the presence of a benzothiazole ring, which easily achieves coplanarity and shoulder merging (loss of fine structure), as well as chromophore groups such as C=O, >C=C, >C=N, and -NO<sub>2</sub> groups in conjugation with an aromatic nucleus (benzothiazole ring). At 370 nm, the more intense band could be due to the ( $n \rightarrow \pi^*$ ) transition, which shows the presence of -NH auxochrome. As a result, the  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions confirm the presence of aromatic nuclei and -NH groups, respectively. Bathochromic shifts (a shift toward longer wavelength) from the basic value, i.e. 240 and 310 nm, may be caused by a combination of conjugation (due to the chromophore) and -NH groups (auxochrome). A hyperchromic shift ( $\epsilon_{max}$  higher values) is caused by the presence of -NH groups (auxochromes)[25].



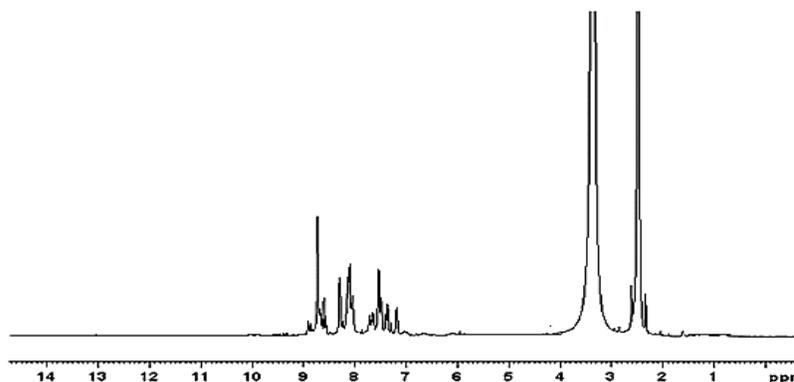
**Figure 2:** UV-Visible spectra of BOF-III copolymer

The FTIR spectra of the BOF-III copolymer is shown in Fig. 3. The band frequencies and specified groups of the copolymer are based on earlier studies[26][27]. The band that emerged at  $3619\text{cm}^{-1}$  is caused by the  $-\text{NH}$  asymmetric and symmetric vibrations. The benzothiazole ring stretching mode's  $-\text{NO}_2$  group is responsible for the emergence of a band at  $1507\text{cm}^{-1}$ . The  $1540\text{cm}^{-1}$  band is caused by the  $\text{C}=\text{O}$  stretching vibration. Sharp and medium absorption bands in the range of  $1197$  to  $915\text{cm}^{-1}$  are produced by the copolymer's 2,6,8-trisubstituted benzothiazole ring. The  $-\text{CH}_2$  asymmetrical and symmetrical vibrations in the BOF-III copolymers were confirmed by the absorption band that emerged at  $3004\text{cm}^{-1}$ . The aromatic ring's  $-\text{CH}$  stretching vibrations have a peak at  $2910\text{cm}^{-1}$ . The band appearing at  $1438\text{cm}^{-1}$  in the copolymer suggests that the  $-\text{CH}_2$  bending vibration in the  $\text{N}-\text{CH}_2-\text{N}$  Bridge is present. The presence of the  $\text{C}-\text{S}-\text{C}$  group is confirmed by the band at  $788\text{cm}^{-1}$ , whereas the band at  $1655\text{cm}^{-1}$  is attributable to the thiazole ring's  $\text{C}=\text{N}$  stretching mode.



**Figure 3:** FTIR spectra of BOF-III copolymer

Fig. 4 shows the NMR spectra of the BOF-III copolymer in  $\text{DMSO}-d_6$  solvent. The signals of the BOF-III copolymer's proton NMR spectra were interpreted using literature. The singlet signal at  $8.33\text{ppm}$  is caused by the  $-\text{NH}$  proton of the benzothiazole ring. A singlet was found at  $4.82\text{ppm}$  due to the presence of the  $-\text{NH}$ -proton in the  $-\text{CH}_2-\text{NH}-\text{CO}$  group. The methylene protons in the copolymer are responsible for the  $2.50\text{ppm}$  signal. The singlet signal at  $3.42\text{ppm}$  is caused by the  $\text{N}-\text{CH}_2-\text{N}$  bridge proton. The weak multiple signals in the range of  $7.19$ - $8.16\text{ppm}$  are due to all of the protons in the aromatic ring[28].



**Figure 4:**  $^1\text{H}$ -NMR spectra of BOF-III copolymer

Scanning electron micrographs at various magnifications were used to analyze the morphology of all of the BOF-III copolymer, as shown in Fig. 5, with the white bar at the bottom of the micrographs showing the scale. It provides information about the structure's surface topography and defects. The BOF-III copolymers have a closely packed structure with deep pits and more active sites, similar to irregular granular particles. The copolymer is porous, and SEM pictures

revealed a fringed depiction of the semicrystalline structure on the surface of the BOF-III copolymers. As evidenced by the fringes in the micrographs, the copolymers are transitioning between amorphous and crystalline states. A monomer's acidic nature determines the degree of crystalline property it has. The crystalline structure of the monomer is converted to the amorphous phase of the copolymer during polymerization. Visible holes and cracks could be caused by air voids[29].

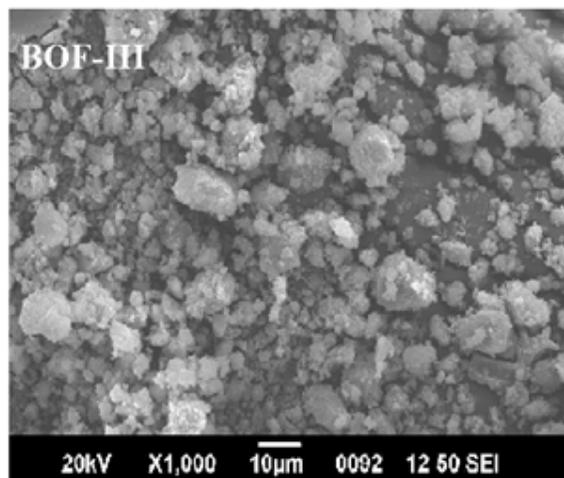


Figure 5: SEM images of BOF-III copolymer

#### 4.3 Photoluminescence Study of BOF-III Copolymer

A Shimadzu MODEL RF-5301(PC) S CE (LVD) LS55 spectrophotometer was used at Kamla Nehru College in Nagpur to measure the photoluminescence spectra of BOF-III copolymer. Organic compounds' luminescence is mostly determined by localized  $\pi$ -electron systems within individual molecules. Because of the conjugated side chains and the  $\pi$ - $\pi^*$  transition of the conjugated polymer chains, the copolymer emits light. Figs. 6 and 7 illustrate the excitation and emission spectra of BOF-III copolymer respectively. The excitation spectra of the copolymer at 365 nm are shown. When the copolymer is excited at 365 nm, it emits intense blue light with a wavelength of 440 nm, which is appropriate for OLEDs, as shown in Figs. 6 and 7. As the conjugation length shortens due to structural influences, the emission appears in the blue region. The PL spectra of the newly synthesized BOF-III copolymers reveal that the material obtained is of enough quality to be used in semiconductor device development and as a supporting material for photoluminescent liquid crystal displays (PLLCD) and solid state lighting applications.

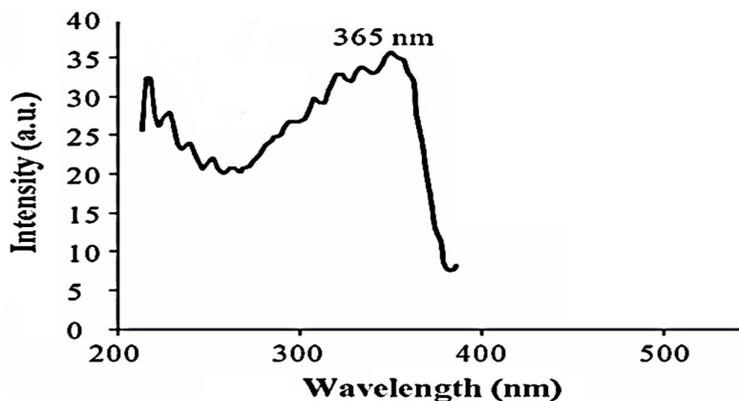


Figure 6: PL Spectra (Excitation) for BOF-III copolymer

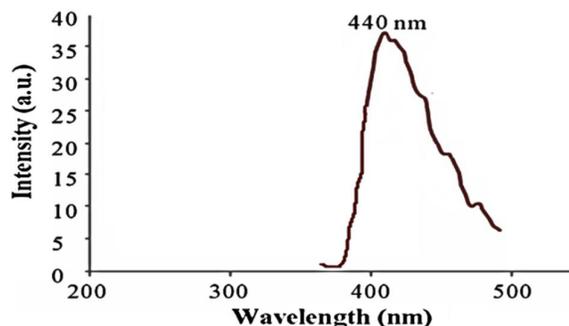


Figure 7: PL Spectra (Emission) for BOF-III copolymer

### V. CONCLUSION

The polycondensation polymerization of 2-amino 6-nitrobenzothiazole, and oxamide with formaldehyde produced a high yield of the BOF-III copolymer in 3:1:5 molar ratios in the presence of an acid catalyst. Elemental analysis, FTIR, <sup>1</sup>H-NMR, and UV-Visible spectral analyses were used to determine the probable structure of copolymer. The semicrystalline structure of the copolymer was confirmed by the surface morphology. The photoluminescence study of the BOF-III copolymer reveals a number of features that could be relevant in the testing of semiconductor devices and light-emitting materials.

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