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Synthesis and Characterisation of Small Thiophene Analogues for Organic Photovoltaic Cell Applications

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Abstract

With an increase in the energy crises, it is vital to find substitute sources of energy. Various sources of alternative renewable sources are being developed. Due to its semiconductor properties, silicon is the metal of choice for manufacturing solar cells. Though silicon-based solar cells are commercialised globally, the cost involved in mining purified silicon is high, causing hindrances in the usage of silicon-based solar cells. Hence, second-generation solar cells, consisting of organic polymers are preferred. However, the stability and efficiency of polymer-based solar cells is still a challenge. Many inorganic semiconductor materials having greater efficiency are being manufactured. However, manufacturing processes, on the other hand, are both material and method-intensive. As a result, Organic-Based Photovoltaic (OPV) cells are gaining popularity because of their potential for low-cost solar energy collection and the abundance and ease of handling organic materials. The present research focuses on developing low-cost, flexible organic semiconductor materials with unique properties including flexibility, lightness, nontoxicity and transparency as well as large-area OPV panels that can be produced with inexpensive methods of solution processing. These benefits suggest that they have a wide range of applications and complement classic silicon-based OPV cells. Their excellent light harvesting, natural charge transport behaviour and structural adaptability, thiophene-based conjugated systems have sparked a lot of attention in the development of high-performance organic solar cells. As a result, fused thiophenes and their analogues have been synthesised for use as organic semiconductor materials in OPVs, and they have been characterised using[1] Proton Nuclear Magnetic Resonance (HNMR), Ultra Violet (UV)-Visible Spectrophotometer and the Tauc equation and graphs. The band gap of each analogue has been determined. Further, the synthesised compounds are characterised for photovoltaic applications using I-V characterisation techniques.

Keywords: *B Band Gap, I-V Characterisation, Photovoltaic Cells, Tauc Plots, Thiophenes*

1.0 Introduction

In one hour, the amount of energy received by the earth from the sun is greater than what people utilise in a whole year. On a clear day, the American Society for Testing and Materials (ASTM) G173-03 standard reference states that 1000 W of solar energy are received by the Earth per square meter (ASTM Standard G173 2008). Meeting people's

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energy needs today and in the future, can be achieved in part by making use of this dependable and sustainable energy source. Thus, Photovoltaic (PV) devices are widely used to directly convert solar energy into electricity. In a lab setting, conventional silicon solar cells demonstrated an efficiency of roughly 25% (Green *et al*. 2011). While silicon is abundant, obtaining high-purity silicon suitable for PV cell fabrication can be challenging and expensive for silicon mining industries[1]. The primary hindrance is related to the purification process required to achieve the desired level of purity. Producing high-grade silicon (such as electronic-grade or solar-grade silicon) involves multiple purification steps, which can be energy-intensive and costly[2]. Thus, amorphous silicon, Cadmium Telluride (CdTe) and Copper Indium-Gallium di Selenide (CIGS) emerged as the main material systems for second-generation PV, with CIGS having the highest efficiency at 19.6%. Many techniques, including Dye-Sensitised Solar Cells (DSSC), polymer solar cells and small molecule organic solar cells, are employed to attain improved energy efficiency. The newest organic compounds are used in the production of third-generation solar cells.

OPV which is a kind of solar cell uses Organic Semiconductors (OSCs), which are often polymers or small molecules, as the absorbing layer[3-5]. Organic materials require a significant amount of conjugation (alternating single and double bonds) to become conducting or semiconducting. The electrons attached to the double bonds of an organic molecule become delocalised throughout the entire conjugation process. These electrons are akin to the valence electrons found in inorganic semiconductor materials and are more energetic than the other electrons in the molecule. However, in organic materials, these electrons belong to the "highest occupied molecular orbital" rather than a valence band[5-8]. Higher energies have unoccupied energy levels, just like inorganic semiconductors. The energy difference between the Lowest Unoccupied Molecular Orbital (LUMO) and the Highest Unoccupied Molecular Orbital (HOMO) determines the material's band gap of the OSC. The energy difference between the LUMO and the HOMO determines the material's band gap of the OSC[8-9]. The band gap will become so tiny as conjugation increases that an electron can be excited from HOMO to LUMO by visible light. Thus, with an increase in conjugation and a decrease in the bandgap energy, highly efficient energy-producing solar cells can be synthesised[9-11].

Thiophene-based-conjugated systems have garnered a lot of interest in the creation of high-performance organic solar cells due to their effective light collection, structural plasticity and natural charge transport behavior[12-14]. These kinds of polymer solar cells can serve as emergency backup power sources for critical systems in mining operations, such as emergency lighting, communication systems or ventilation in case of power outages or emergencies.

2.0 Problem Definition

Traditional solar cells are made up of inorganic material-based semiconductors such as silicon which exhibits efficiency of nearly 25% and are widely used. Particularly, the silicon obtained from mining industries needs to be purified which involves high energy consumption, time and rigorous purification methods and paves an alternate route to use polymer-based solar cells[12-14]. However, they are expensive and require high reaction conditions to synthesise. Thus inorganic semiconductor materials are replaced by organic compounds such as thiophene and their analogues which have both electron-donating and accepting properties, are available in nature, are halogen-free and do not produce ozone-depleting chlorofluorocarbons[15]. In this research fused thiophene and their analogues are synthesised and characterised by PV applications to check the energy efficiency. Such polymer solar cells can be integrated into wearable technology worn by mining personnel, such as safety vests or helmets, to harvest ambient light and convert it into electrical energy for powering communication devices or sensors, enhancing worker safety and efficiency[3-4].

3.0 Methodology

3-bromo-2-methyl thiophene[1] is used as a starting material to produce thieno[3,2-b] 2-carboxylic acid thiophene. It is then combined with the appropriate amines, bases such as Hexafluorophosphate Azabenzotriazole Tetramethyl Uranium (HATU) and Diisopropylethylamine (DIPEA) and solvents such as Dichloromethane (DCM) and agitated under nitrogen conditions overnight at room temperature. By using column chromatography, the product is filtered and purified[16-19].

3.1 To Synthesise Thienothiophene Analogues from 3-Bromo-2-Methyl Thiophene(1)

Intermediate methyl thieno is formed when 3-bromo-2-methyl thiophene[1] is combined with ethyl sulfonyl acetate, potassium carbonate as a base and N, N-Dimethylformamide as the solvent at 70°C at room temperature for two hours [3,2-b] is hydrolysed for two hours at room temperature with LiOH, water, and ethanol to yield thieno[3,2-b] thiophene-2-carboxylic acid[4], which is then utilised as a starting material[21] to create other thiophene analogues.

Characterisation of 2-carboxylic acid thiopheno [3,2-b][5] is performed by Liquid Chromatography-Mass Spectrometry (LCMS).

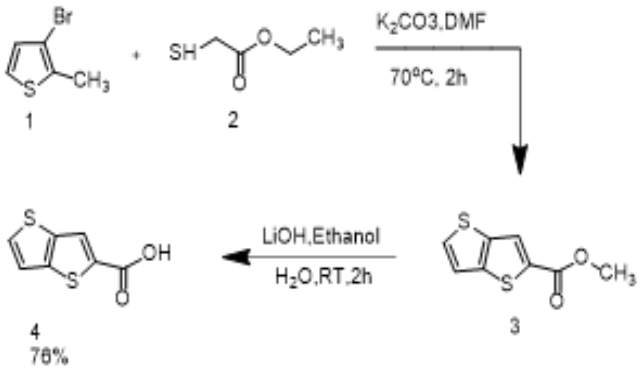


Figure 1. Synthesis of thieno[3,2-b] thiophene-2-carboxylic acid (4).

3.2 Derivatisation of Thienothiophene Compounds

3.2.1 *Synthesis of 2-(thieno[3,2-b] thiophen-2-yl)-1H-benzimidazole*

Thieno[3,2-b] thiophene-2-carboxylic acid is coupled along with O-phenylenediamine in the presence of HATU and N, N-(DIPEA) bases and Dichloromethane as solvent at room temperature.

2-carboxylic acid thiopheno[3,2-b], O-phenylenediamine, HATU, DIPEA and DCM are added to a round bottomed flask and stirred overnight at room temperature. The progress of reaction is monitored by Thin Layer Chromatography (TLC) and the product is purified using Column Chromatography with hexane and ethyl acetate as eluents.

The purified product is 2-(thieno[3,2-b] thiophene-2-yl)-1H-benzimidazole[8], first analogue of thiophene synthesised is then characterised by ^{1}HNMR.

3.2.2 *Synthesis of N-phenyl 2-carboxylic acid thiopheno[3,2-b]*

At ambient temperature, compound (B) and 2-aminophenol are coupled in the presence of hexafluorophosphate Az benzotriazole tetramethyl uranium (HATU), diisopropylethylamine (DIPEA) bases, and dichloromethane as the solvent.

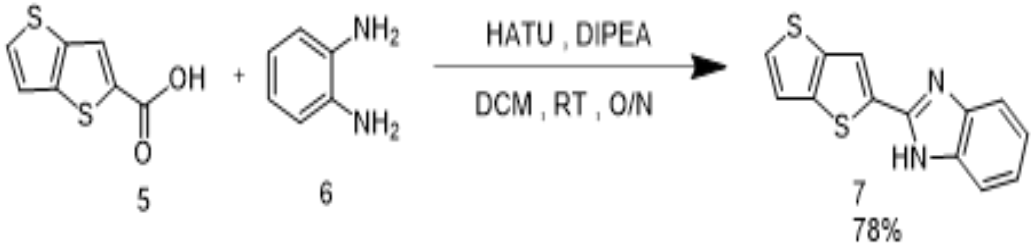


Figure 2. Creation of 2-(thiophen-2-yl] thieno[3,2-b])-1H-benzimidazole(7).

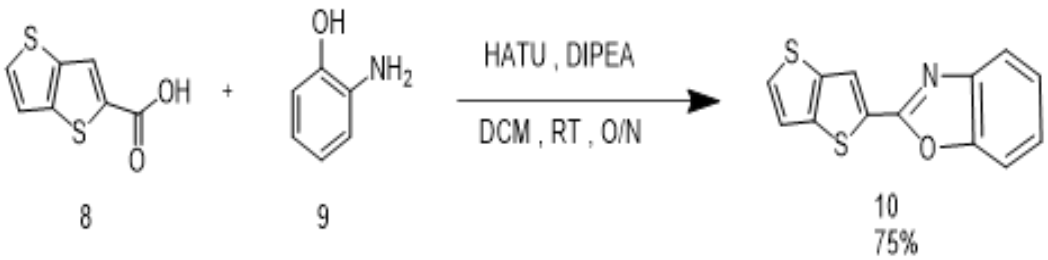


Figure 3. Synthesis of N-phenyl 2-carboxylic acid thiopheno[3,2-b] (10).

A round-bottomed flask is filled with thieno[3,2-b] thiophene-2-carboxylic acid[4], 2-aminophenol, HATU, DIPEA, and DCM. The mixture is then agitated overnight at room temperature. TLC is used to assess the reaction's progress, and column chromatography which uses hexane and ethyl acetate as solventsis used to purify the end product.

The purified product is N-phenyl 2-carboxylic acid thiopheno[3,2-b][10], the second analogue of thiophene synthesised is then characterised by ^{1}HNMR.

3.2.3 Synthesis of 2-(thieno[3,2-b] thiophen-2-yl) benzo[d]thiazole

HATU, N, N-(DIPEA) bases and dichloromethane as the solvent are present when 2-Aminothiophenol is mixed with Thieno[3,2-b] thiophene-2-carboxylic acid at room temperature.



Figure 4. Synthesis of 2-(thieno[3,2-b] thiophen-2-yl) benzo[d]thiazole (13).

A round-bottomed flask is filled with thieno[3,2-b] thiophene-2-carboxylic acid (4), 2-aminothiophenol, HATU, DIPEA, and DCM, and the mixture is agitated overnight at room temperature. Thin Layer Chromatography (TLC) is used to track the progress of the reaction, while column chromatography is used to purify the result.

The third produced analogue of thiophene, 2-(thieno[3,2b] thiophene-2-yl) benzo[d]thiazole (13) is then identified by ^{1}HNMR from the purified product.

3.3 Synthesis of Cobalt Ferrite Nanoparticles

Iron nitrate (7g) and Cobalt hexahydrate (2.52g) were added to the minimal amount of water and sonicated for 15 minutes. In a water bath glycine of 1.936g was taken and dissolved using distilled water and to this iron nitrate and cobalt hexahydrate solution were added and one hour of stirring was continued at 50^{0}C. Later the bead was taken out and stirred manually at maximum temperature till the formation of a puffy-like material. The puff was removed and finely ground into the nanoparticles[22].

The thiophene analogues synthesised were ground along with cobalt ferrite nanoparticles at 2%, 4% and 6%. Further PV cell studies of these hybrid compounds were conducted.

4.0 Results and Discussions

4.1 LCMS of Acid

In Figure 5, the mass of acid is observed at 185.20 which helps to elucidate the structure and chemical properties of the compound.

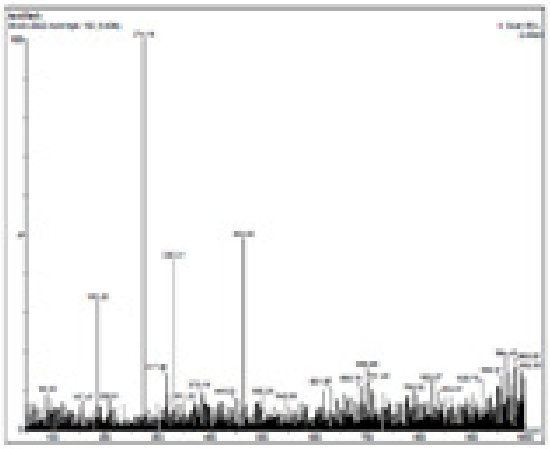


Figure 5. LCMS of 2-carboxylic acid thiopheno[3,2-b] (4).

4.2 ^{1}HNMR

All of the generated compounds' structures were verified using proton nuclear magnetic resonance.

4.3 UV-Visible Spectrophotometer Study

The synthesised thiophene analogues' optical absorptivity was examined with a UV-visible spectrophotometer that

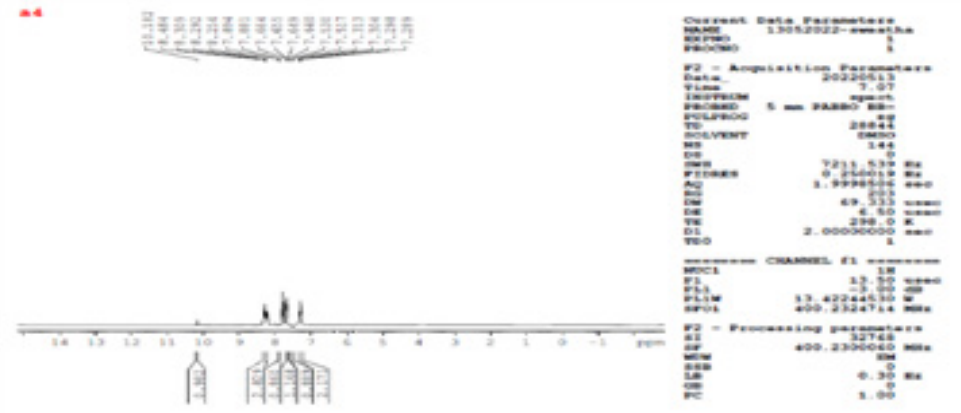


Figure 6. ^{1}HNMR of 2-(thieno[3,2-b] thiophen-2-yl)-1H-benzimidazole (7).

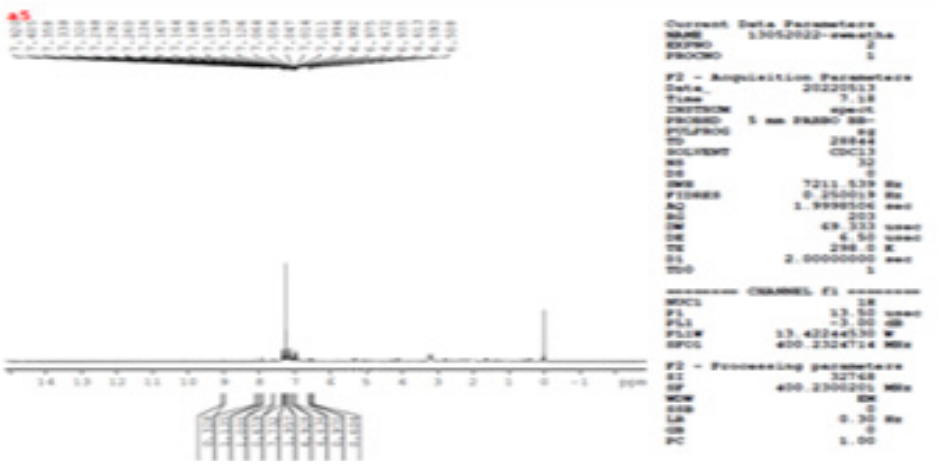


Figure 7. ^{1}HNMR of N-phenyl thieno [3,2-b] thiophene-2-carboxamide (10).

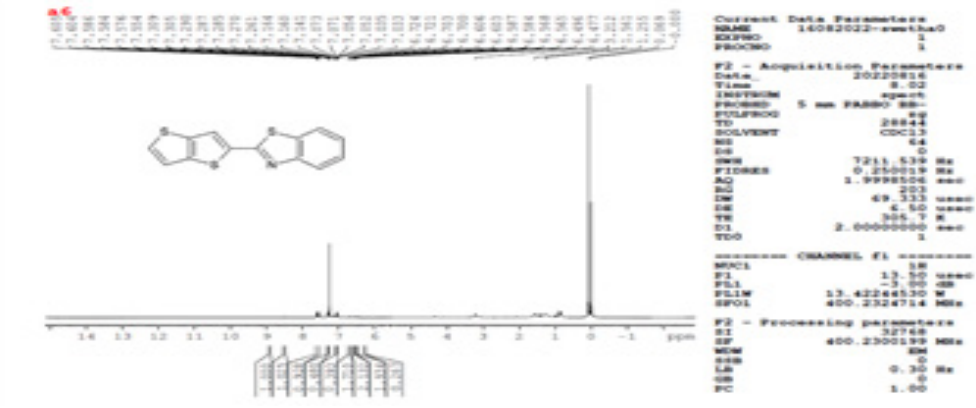


Figure 8. ^{1}HNMR of 2-(thieno[3,2-b] thiophen-2-yl) benzo[d]thiazole (13).

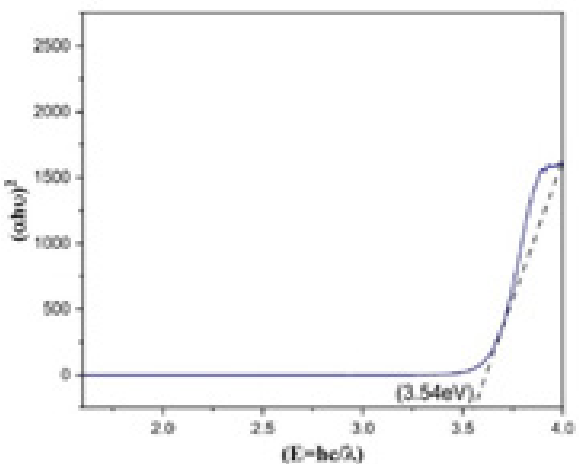


Figure 9. Tauc plot of 1-H-benzimidazole 2-(thieno [3,2-b] thiophen-2-yl) (7).

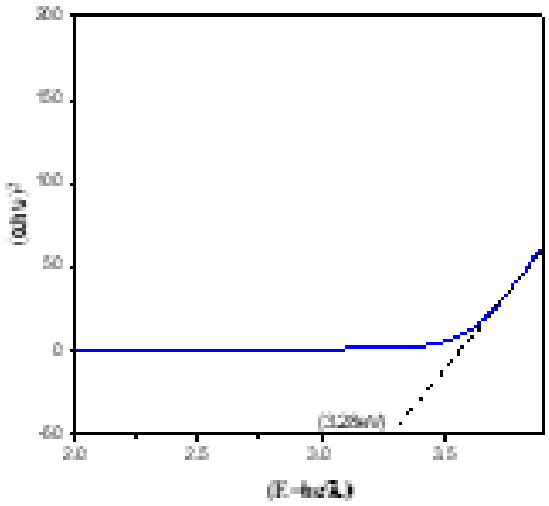


Figure 10. Tauc plot of [3,2-b] N-phenyl thieno 2-carboxamide thiophene (10).

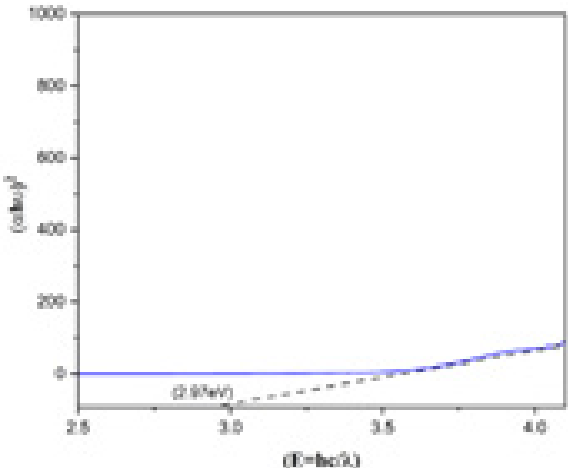


Figure 11. Tauc plot of benzo[d]thiazole 2-(thieno [3,2-b] thiophen-2-yl) (13).

measured wavelengths between 200 and 800nm. The UV-visible absorbance data and the Tauc plot were used to calculate the band gap values[23-26].

$(\alpha h\nu)^{\gamma} = A\ (h\nu - E_g)^n$

α = absorption coefficient,

h = Planck's constant,

ν = photon frequency,

A= Proportionality constant, which is set at 1 for amorphous materials, is found by measuring the index of refraction of electrons in hole-effective masses.

E_g= Energy of band gap

γ = Electronic transition nature

4.4 I-V Characterisations

Solar cell devices with all three analogues were fabricated and tested. A solar simulator was used to carry out the

Table 1. Depicts the optical bandgap (E_g) and λ_{max} of all three analogues.

Analogues	Bandgap Energy(eV)	λ_{max}	Spectral Range	Absorption Region
Analogue (7)	3.54	302.5nm	251.18-376.47nm	Ultraviolet
Analogue (10)	3.28	300.6nm	240.49-364.81nm	Ultraviolet
Analogue (13)	2.97	245.0nm	240.62-359.95nm	Ultraviolet

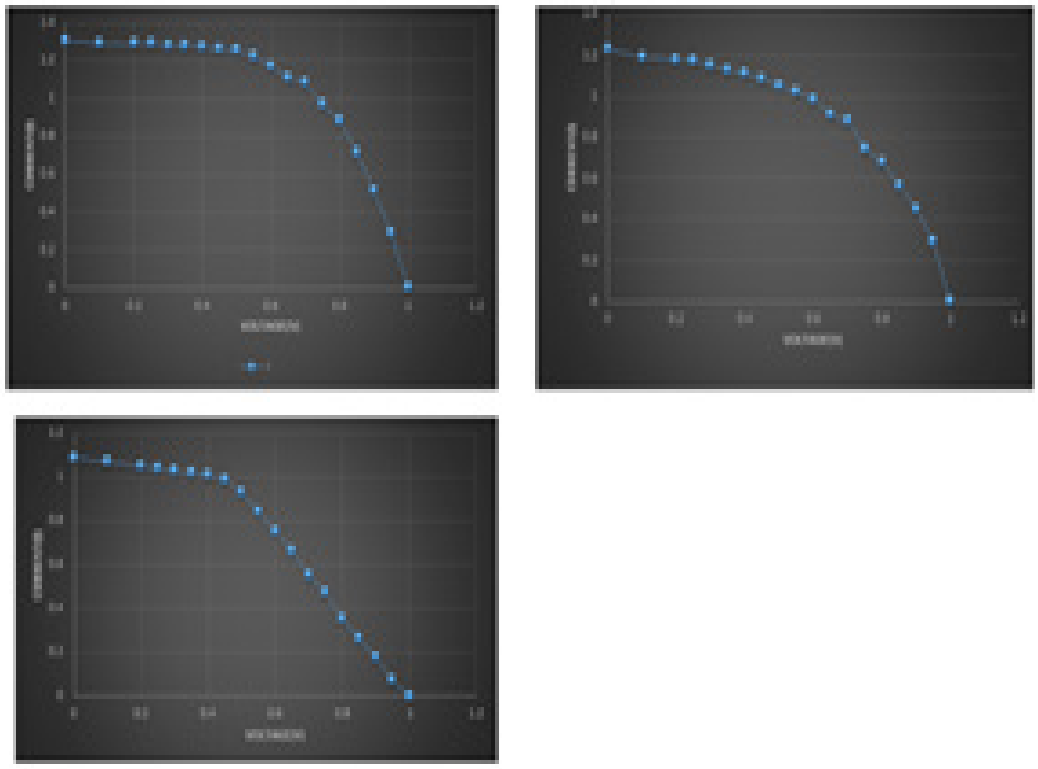


Figure 12. .I-V results of Figures 2,3 and 4 respectively.

I-V characterisation. Glass substrates coated with ITO were utilised to fabricate devices after being cleaned with acetone and isopropanol in succession. The whole manufacturing process took place in a glovebox. To finish the device's construction, a top electrode made of silver was employed[26-28].

5.0 Photovoltaic Applications

The three new synthesised analogues by acid-amine coupling were characterised by ^{1}HNMR. The optical bandgap of new compounds was obtained by Tauc plot which exhibited 3.54eV,3.28eV and 2. 97eV[25-28]. Further, the analogues were subjected to I-V characterisation. The Benzo[d]thiazole 2-(thieno[3,2-b] thiophene-2-yl) analogue showed greater PCE that is 0.99% whereas [3,2-b] N-phenyl thieno Two-carboxamide thiophene and 1-H-benzimidazole 2-(thieno[3,2-b] thiophene-2-yl) exhibited 0.7% and 0.66% respectively[30-33].

6.0 Conclusion

In the current research work, several bases and solvents were employed to synthesise fused thienothiophene and its analogues at room temperature**.** The optical absorptivity of compounds, which ranges from 2.97eV to 3.54eV, was investigated using a spectrophotometer with UV visibility with a range of wavelengths: 200–800nm. I-V characterisations are carried out for photovoltaic applications, with energy efficiencies exhibiting between 0.66% and 0.99%. All of these compounds were characterised using 1HNMR.

Organic semiconductors with cheap costs were created in this study to supplement inorganic semiconductors such as silicon-based materials, which are quite expensive. To increase energy efficiency, organic molecules with higher conjugation and a smaller band gap are synthesised. Thienthiophenes and their analogues are well-known chemicals with a narrower band gap. The compounds developed in this work have a bandgap

of around 3.26 eV, which may be further decreased by doping them with metal dopants, which enhances their optical properties. Thus, advancements in materials science and manufacturing techniques may address some of these challenges and make polymer solar cells more viable for use in mining applications in the future.

7.0 Acknowledgement

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