

SYNTHESIS AND CHARACTERIZATION OF NEW FLUORESCENCE POLYAMIDES

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Abstract. In this study, a series of novel fluorescent polyamides (PA2, PB2 and PC2) were synthesized and characterized through a multi-step process involving the preparation of monomeric intermediates and subsequent polycondensation reactions. Spectroscopic techniques, including FT-IR, UV-Vis, fluorescence emission and thermal analysis (TGA), besides the use of FESEM to identify the surface morphology, were used to confirm the successful formation of the target polyamides and investigate their structural and thermal properties. The synthesized polyamides exhibited strong green fluorescence and variable thermal stability, with PB2 showing the highest stability among the series. These findings highlight the potential of such materials for applications in optoelectronic devices and smart polymer systems.

Keywords: Polyamides, Pentaerythritol, Bischalcon, fluorescence.

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1. Introduction

Polyamides are one of the most important polymers, the amide group (-CO-NH-) was the repeating unit of polymers. It can be found in natural materials (such as proteins, silk, wool, etc.) as well as in synthetic compounds (such as nylons and aromatic polyamides or aramids) (Miriam *et al.*, 2018). In the presence of an amide group in the repeating monomer units, strong hydrogen bonds will be present, which is responsible for its strength, stability, crystallinity, melting points and mechanical strength, overall, polyamides have good performance in all areas, including textiles, mechanical, electronic, biological materials and water treatment (Xubao *et al.*, 2023). Also, related to their attractive mechanical strength, chemical resistance and thermal stability, polyamides are widely used in industries differing from automotive and electronics to textiles and packing, considerate that the thermal behavior of polyamides is essential, as their response to heat, including melting, crystallization and thermal deterioration, frequently determine how well they conduct in applications (Simin, 2006).

Based on their molecular structure, polyamides can be commonly classified into three categories, aliphatic, aromatic (aramid) and semi-aromatic, each one exhibiting distinct properties depending on their molecular structure, aliphatic polyamides such as Nylon 6 and Nylon 6,6, are frequently utilized in commonplace products like carpets, clothing and packaging films. Due to their exceptional tensile strength and heat resistance, aromatic polyamides such as Kevlar and Nomex are used in high-performance applications (Mohammad & Tatsuo, 2015).

The investigation of fluorescence in polyamides creates new opportunities for their use in: Innovative light-emitting nanodevices, biosensors, sustainable energy technologies, explosive sensing, pH/temperature sensors, biological imaging, life science, material science and ultrasensitive molecular diagnosis (Jignasa, 2023).

Fluorescence in polyamides can be introduced either from the polyamide structure or produced by chemical modification. The notable photophysical activity may result from the presence of aromatic groups, conjugated systems, or electron-donating or electron-withdrawing substituents within the polymer backbone. Like aromatic polyamides (aramids), for example, those that have para-phenylene groups, generally exhibit fluorescence related to $\pi \rightarrow \pi^*$ transitions in the aromatic rings (David *et al.*, 2000; Mousa & Raouf, 2011). Furthermore, their emission properties can be further improved or tuned by adding fluorophores or chromophores to the polyamide chain (Marioara, 2009).

Further mechanisms affect fluorescence in polyamides, such as aggregation-induced emission (AIE), which limits intramolecular movement in the solid or aggregate state to increase fluorescence. Also, the interactions of hydrogen bonding that are inherent in the amide groups can further be involved in adjusting photophysical properties by affecting the molecule packing, rigidity and energy transfer (Nicholas *et al.*, 2015; Ju *et al.*, 2015).

Despite studies on fluorescent polyamides, there remains a significant gap in the development of structurally defined polyamides derived from monomers (DA2, DB2 and DC2) that exhibit both strong fluorescence and thermal stability. Most existing works focus on traditional fluorophores without integrating environmentally conscious or structurally novel components. In this study, we introduce a new class of fluorescent polyamides synthesized from monomers (DA2, DB2 and DC2), aiming to enhance both fluorescence intensity and thermal resistance. The novelty of this research lies in the combination of structurally diverse polyamide backbones, which has not been thoroughly explored in recent literature. Our findings contribute to the development of advanced fluorescent materials suitable for optoelectronic and sensing applications.

2. Experimental

2.1. Material

Fluka, Merck, B.D.H, J.T. Baker, Sigma-Aldrich, HIM, fluoro chem and SRL companies provided all the chemicals and solvents used in this study. The melting point of all prepared compounds was taken in open capillaries and recorded on Cole-Parmer by Bio COTE, a digital melting point device located at the Department of Chemistry-College of Science-University of Basrah. Infrared spectra for all compounds were measured as KBr disks using an FTIR spectrophotometer Shimadzu 8400S in the range $400 - 4000 \text{ cm}^{-1}$. $^1\text{H-NMR}$, $^{13}\text{C-NMR}$ spectra were recorded using a 100 MHz Bruker AVANCE NEO 400 spectrometer in deuterated DMSO- d_6 solvent at the Department of Chemistry- College of Education for Pure Science - University of Basrah. Thermo Gravimetric Analysis was recorded using TA Q600 at a constant heating rate ($20 \text{ }^\circ\text{C/min}$) at the University of Tehran in (IRAN). Fluorescent measurement by Fluo Time300 Fluorescence spectrometer, PICO QUANT (Germany), College of Science, University of Basrah, Iraq. FESEM was photographed on TESCAN Tehran University (IRAN). UV-visible measured by UV-19001 SHIMADZU in College of Science, University of Basrah.

2.2. Synthesis methods

2.2.1. Synthesis of monomers DA2, DB2 and DC2

The monomers were prepared according to the literature (Muhanad *et al.*, 2022) with some modifications, from the reaction of isophthaloyl dichloride with pentaerythritol, dipentaerythritol and tripentaerythritol, respectively:

Monomer [DA2]: (Figure 1) A solution of pentaerythritol (0.004 mol, 0.544g) dissolved in 50 mL of dry dimethylformamide(DMF) in a 100 mL round-bottom flask equipped with a magnetic stirrer, the solution was stirred continuously at room temperature until solubility is completed, then triethylamine (0.008 mol, 1.11mL) was added to the solution above and stirred continuously for 90 min. at temperature 10°C under a nitrogen atmosphere to prevent oxidation, after that (0.02 mol, 4.06 gm) of isophthaloyl dichloride dissolved in 50 mL of dry dimethylformamide(DMF), dropped into the mixture reaction for an hour at 10°C with constant stirring, then the mixture was left with stirring at 10°C for 24 hours. The resulting solution was poured into (300 mL) of cold distilled water, the precipitated product was filtered, washed several times with water and ethanol and dried at room temperature, the resultant was solid white powder labeled as DA2. Yield 80%. M. p. > 300 °C. Its structure was confirmed by FT-IR, ¹H-NMR and ¹³C-NMR. FT-IR: ν 1793 (C=O stretching of acid chloride), 1689(C=O stretching of ester), 1423(CH=CH stretching of aromatic), 3066(CH stretching of aromatic), 2989(CH stretching of aliphatic). ¹H NMR δ =4.88(s,8H, CH₂ aliphatic), 7.61(t,4H, aromatic), 8.15-8.17 (2d,8H, aromatic), 8.74 (t,4H, aromatic). ¹³C NMR: δ = 166.57(C3 C=O ester), 169.18(C10 C=O acid chloride), 44.58 (C1), 64,14(C2), 128.7(C4), 131.15(C5), 129.12(C6), 136.74(C7), 133.36(C8), 129.93(C9).

Monomer [DB2]: (Figure 2) Prepared from dissolving (0.004 mol, 1.017 gm) of dipentaerythritol in 100 mL of dry dimethylformamide (DMF) in a 200mL round-bottom flask equipped with a magnetic stirrer, the solution was stirred at room temperature to enhance the solubility after that triethylamine (0.008 mol, 1.11mL) was added with continuous stirring for (90min.) to the solution at a temperature of 10°C and under an inert atmosphere of nitrogen to prevent oxidation, then add a solution of (0.024 mol, 4.872 gm) isophthaloyl dichloride dissolved in 50mL of dry dimethylformamide (DMF) dropwise and stirred for an hour. The reaction mixture remained under the stirrer for 24 hours and at 10°C, then the product mixture was added to (300mL) of cold distilled water, the resulting sediment was filtered, washed with water and ethanol for some time and then dried at room temperature, the white solid product obtained labeled as DB2. Yield 70%. M. p. > 300 °C. The structure of DB2 was verified by FT-IR, ¹H-NMR and ¹³C-NMR. FTIR: ν 1793 (C=O stretching of acid chloride), 1708(C=O stretching of ester), 1419 (CH=CH stretching of aromatic), 3039 (CH stretching of aromatic), 2962 (CH stretching of aliphatic). ¹H NMR: δ =3.6 (s,4H, aliphatic), 4.36 (s,12H, aliphatic), 7.6 (t,6H, aromatic), 8.13 (2d,6H, aromatic), 8.31(2d,6H, aromatic), 8.46 (t,6H, aromatic). ¹³C NMR: δ = 167.07(C6 C=O ester), 169.73(C13 C=O acid chloride), 45.38 (C1), 64,58(C2 ,C3,C5), 87.50(C4,C14), 129.12(C7), 131.64(C8), 129.52(C9), 137.16(C10), 133.36(C11), 130.45(C12).

Monomer[DC2]: (Figure 3) (0.002 mol, 0.744gm) from tripentaerythritol dissolved in a mixture of dry dimethylformamide(DMF): dry benzene (3:2 ml) (Al-Mughaid & Grindley, 2006) in a 200mL round-bottom flask equipped with a magnetic stirrer, to complete the solubility the solution stirred at room temperature, then (0.004 mol, 0.55mL) from triethylamine was added to the solution with stirring for 90min. at temperature 10°C

and under a nitrogen atmosphere to prevent oxidation, after that a solution of isophthaloyl dichloride (0.02mol, 4.060gm) dissolved in 50mL of dry dimethylformamide (DMF) was dropped to the mixture for an hour and at 10°C, the reaction mixture left at 10°C and under stirring for 24hours, the resultant solution poured into (300mL) of cold distilled water to obtain the precipitate which was filtered and washed several times with water and ethanol, then dried at room temperature to gained white sold product labeled as DC2. Yield 72%. M.p > 300°C. The structure was confirmed by FT-IR, ¹H-NMR and ¹³C-NMR. FTIR: ν 1793 (C=O stretching of acid chloride), 1712(C=O stretching of ester), 1423(CH=CH stretching aromatic), 3271(CH stretching aromatic), 2893(CH stretching aliphatic). ¹H NMR δ = 3.87(s, 4H, aliphatic), 3.91 (s, 4H, aliphatic), 4.06(s, 4H, aliphatic), 4.22 (s, 12H, aliphatic), 7.62 (t, 8H, aromatic), 8.15(2d, 8H, aromatic), 8.32(2d, 8H, aromatic) 8.47(t, 8H aromatic). ¹³C NMR: δ = 166.71(C6, C17 C=O ester), 170.38(C13 C=O acid chloride), 70.74(C1, C26), 45.84(C2), 61.07(C3, C4, C5), 129.26(C7, C18), 133.50(C8, C19), 130.07(C9, C20), 138.58(C10, C21), 134.98(C11, C22), 131.28(C12C23), 70.87(C14, C25), 45.91(C15), 61.21(C16, C24).

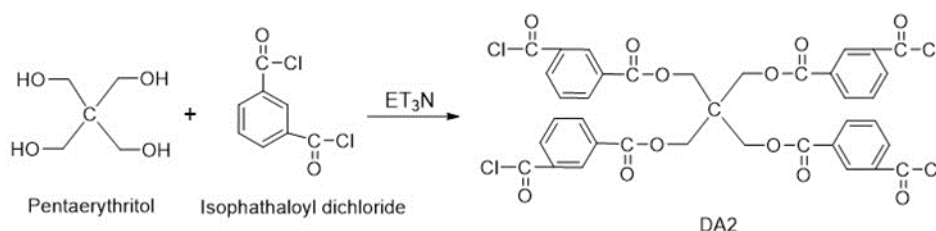


Figure 1. Represent the synthetic route for the preparation of monomer DA2 through the condensation reaction of pentaerythritol and isophthaloyl dichloride

The reaction was carried out in DMF at 10°C under a nitrogen atmosphere.

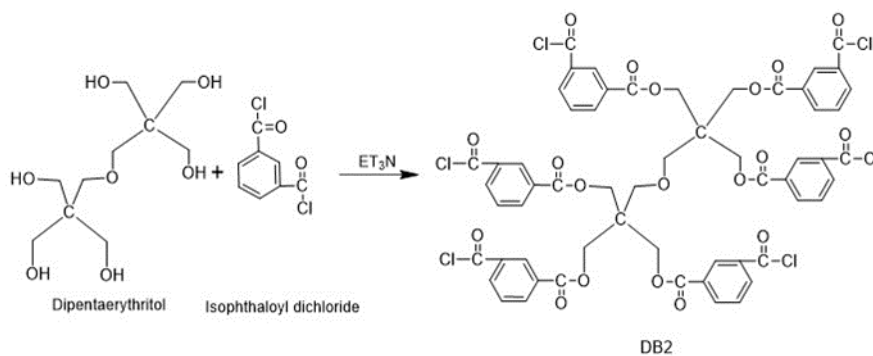


Figure 2. Represent the synthetic route for the preparation of monomer DB2 through the condensation reaction of dipentaerythritol and isophthaloyl dichloride. The reaction was carried out in DMF at 10°C under a nitrogen atmosphere

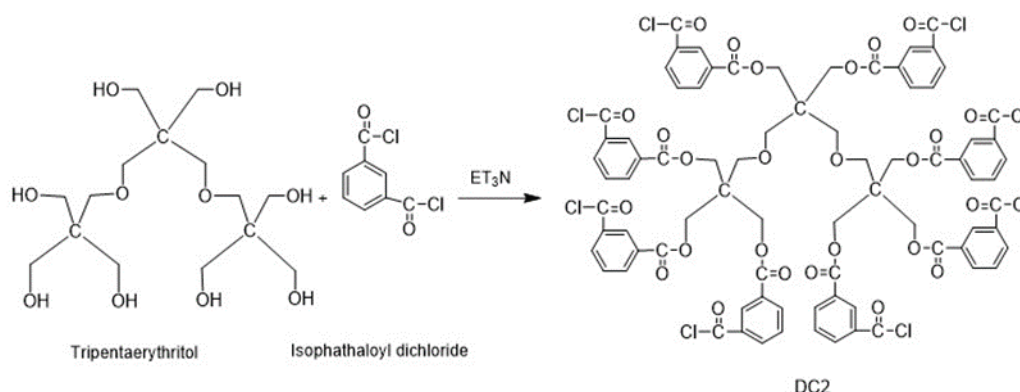


Figure 3. Represent the synthetic route for the preparation of monomer DC2 through the condensation reaction of triptaerythritol and isophthaloyl dichloride. The reaction was carried out in DMF at 10°C under a nitrogen atmosphere

2.2.2. Synthesis of Bis-chalcone

Bis-Chalcone 3,3'-(1,4-phenylene) bis (1-(4-aminophenyl) prop-2-en-1-one) was prepared as described in the literature (Abdullah *et al.*, 2022) (Figure 4). In a 100mL round-bottom flask equipped with a magnetic stirrer, a mixture of 4-aminoacetophenone (0.015 mol, 2.013884 gm) and terephthalaldehyde (0.00750 mol, 1.005975 gm) dissolved in absolute ethanol 25mL, the reaction mixture was stirred at ambient temperature for 20 minutes, then 10 mL from an aqueous sodium hydroxide solution 60% NaOH was added dropwise with constant stirring. Finally, the mixture was stirred for 24 hours at room temperature until the mixture became thick (i.e., the stirring was no longer effective) until a dark yellow mass was obtained. Then it was diluted with 100 mL of cold distilled water. TLC was used to track the reaction utilizing (benzene: methanol) (8:2) mL (R_f 0.66). After filtering, the product was rinsed with water until the filtrate was litmus-neutral. The desired product dark yellow (yield: 79%, m.p.: 273-277°C decomposition) was achieved by recrystallizing from absolute ethanol and distilled water (1:1) to give the required product.

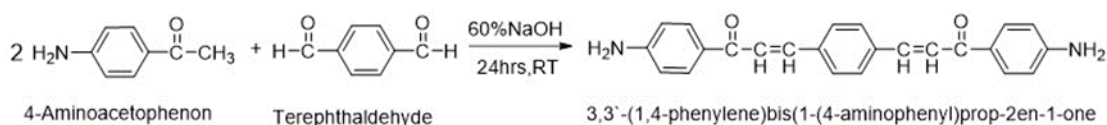


Figure 4. Represent a synthetic route for the preparation of bis-chalcone from the reaction of 4-aminoacetophenone and terephthalaldehyde, the reaction was carried at room temperature with stirring and in the presence of sodium hydroxide solution

2.2.3. Synthesis of polyamides PA2, PB2 and PC2

Polyamides PA2, PB2 and PC2 were prepared by polycondensation reaction of monomers DA2, DB2 and DC2 with bis-chalcone according to the literature with some modification (Abdullah *et al.*, 2022):

Polyamide [PA2]: (0.003 mol, 1.104gm) from bis-chalcone dissolved in 25mL of absolute ethanol was added to a solution of (0.0015 mol, 1.185 gm) of DA2 in 50 mL of

dry dimethylformamide (DMF) in a 100mL round-bottom flask equipped with a magnetic stirrer, the mixture stirred for 20 minutes, then 20 mL of 10% aqueous sodium hydroxide solution was added dropwise to the mixture and then the mixture was refluxed at 80-90 °C for about 6 hours in an oil bath with constant stirring. The reaction was monitored by TLC using an eluent of (8 benzenes:2 methanol) mL. The resultant mixture was cooled to room temperature and then poured into cold distilled water (150 mL). The precipitate was filtered off and washed with distilled water until the filtrate was litmus-neutral and then dried at room temperature. The resultant solid product was recrystallized using a 1:1 mixture of distilled water and absolute ethanol to give a brightly orange powder (Rf 0.59), labeled as PA2. Yield 75%. M. p. 188°C (Figure 5). FTIR: ν 3475(N–H stretching of amide), 1630(C=O stretching of ester), 1597(C=O stretching of amid), 1438(CH=CH stretching of aromatic), 1573(CH=CH stretching of aliphatic alkene), 3325(CH stretching of aromatic), 2901(CH stretching of aliphatic), 1543(N–H bending of amide).

Polyamide [PB2] : (0.0045 mol, 1.656 gm) bis-chalcone dissolved in 25mL of absolute ethanol mixed with a solution of (0.0015 mol, 1.879 gm) from DB2 dissolved in 50mL of dry dimethylformamide (DMF) in a 100mL round-bottom flask equipped with a magnetic stirrer, by following the same procedure of preparing polyamide PA2, the brightly orange powder was obtained after recrystallized resultant product with a (1:1) mixture of distilled water and absolute ethanol, Rf (0.61) and labeled as PB2. Yield 72%. M. p. 191°C (Figure 6). FTIR: ν 3475(N–H stretching of amide), 1620(C=O stretching of ester), 1600(C=O stretching of amid), 1438(CH=CH stretching of aromatic), 1570(CH=CH stretching of aliphatic alkene), 3329(CH stretching of aromatic), 2901(CH stretching of aliphatic), 1543(N–H bending of amide).

Polyamide [PC2]: Prepared in the same manner by mixing the solution of (0.0018 mol, 0.6624 gm) of bis-chalcone dissolved in 25mL of absolute ethanol and a solution of (0.0003 mol, 0.5112 gm) from DC2 dissolved in 50mL of dry dimethylformamide(DMF), also the same way of preparing PA2 and PB2, PC2 was gained as a brightly orange powder product after recrystallized, Rf (0.52), the product labeled as PC2. Yield 68%. M.p. 186°C (Figure 7). FTIR: ν 3471(N–H stretching of amide), 1620(C=O stretching of ester), 1597(C=O stretching of amid), 1438(CH=CH stretching of aromatic), 1570(CH=CH stretching of aliphatic alkene), 3329(CH stretching of aromatic), 2893(CH stretching of aliphatic), 1543(N–H bending of amide).

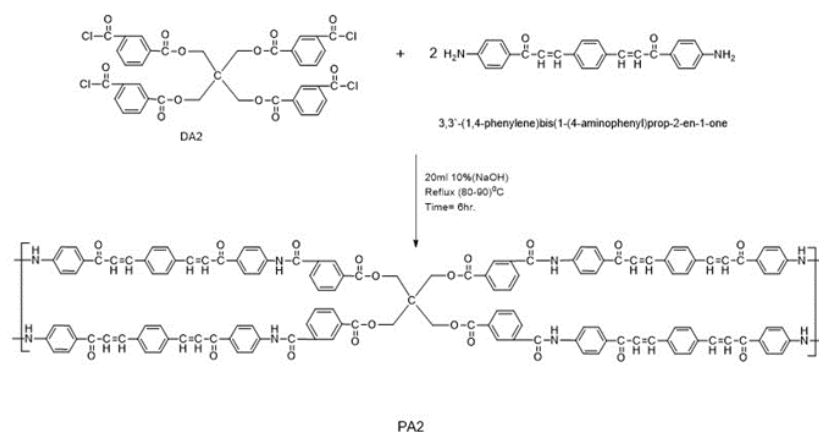


Figure 5. Proposed mechanism for the polycondensation of DA2 with bis-chalcone to yield the fluorescent polyamide PA2. The reaction proceeds via step-growth polymerization

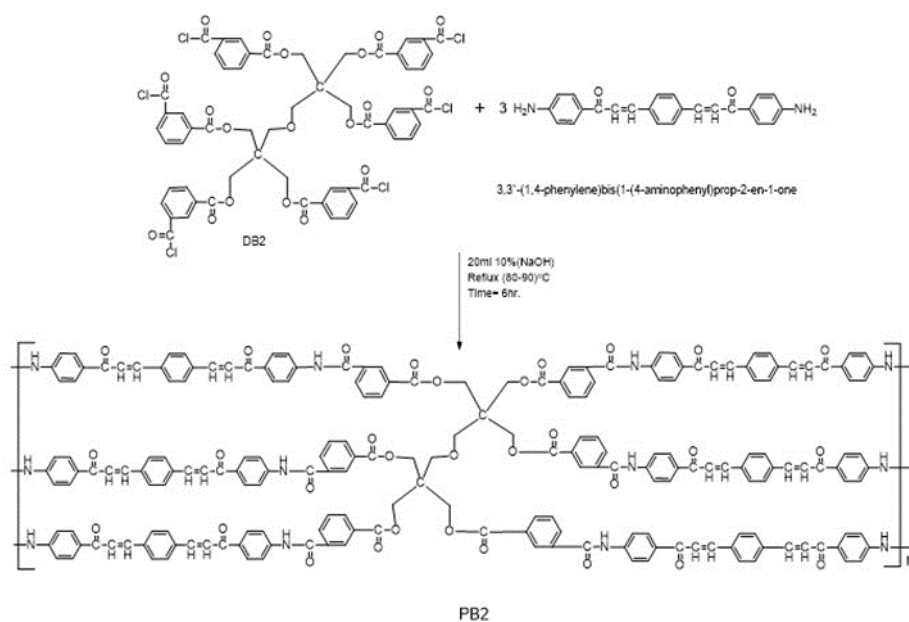


Figure 6. Proposed mechanism for the polycondensation of DB2 with bis-chalcone to yield the fluorescent polyamide PB2. The reaction proceeds via step-growth polymerization

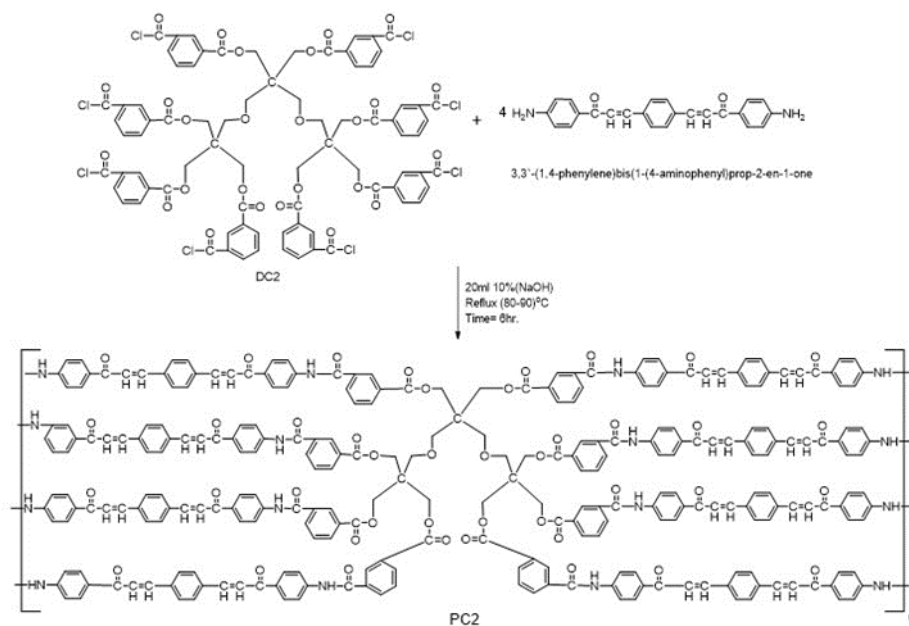


Figure 7. Proposed mechanism for the polycondensation of DC2 with bis-chalcone to yield the fluorescent polyamide PC2. The reaction proceeds via step-growth polymerization

3. Result and discussion

3.1. FTIR analysis

Monomers DA2, DB2 and DC2 were prepared from the condensation reaction of pentaerythritol, dipentaerythritol and tripentaerythritol, respectively, with isophthaloyl dichloride, they were characterized by FTIR spectroscopy and it showed the appearance

of absorption bands at 1793cm^{-1} , which was related to CO–Cl stretching of acid chloride group, peaks in the range of $1689\text{--}1712\text{ cm}^{-1}$ due to C=O stretching of the ester group, peaks at $2893\text{--}2989\text{ cm}^{-1}$ were attributed to CH aliphatic stretching, while peaks at $3039\text{--}3271\text{ cm}^{-1}$ related to CH aromatic, another peaks in the range of $1419\text{--}1423\text{ cm}^{-1}$ associated with CH=CH cm^{-1} aromatic stretching.

Polyamides PA2, PB2 and PC2 were prepared by step-growth polymerization of monomers DA2, DB2 and DC2 with different ratios of bis-chalcone, characterized by FT-IR, demonstrating the disappearance of CO–Cl and appeared a new stretching band of NH amide at $3471\text{--}3474\text{ cm}^{-1}$ and stretching bands of C=O for amide in the range $1597\text{--}1600\text{ cm}^{-1}$, besides the presence of C=O stretching bands of ester in the range $1620\text{--}1630\text{ cm}^{-1}$, also in the range of $1570\text{--}1573\text{ cm}^{-1}$ stretching bands of CH=CH alkene were observed, the absorption stretching bands of C=O amide, ester and CH=CH of alkene were shifted to a lower frequency that due to the high conjugation that can occur in the presence of these functional groups, leading to electron density distribution out of the molecule, that leads to lowering the frequency and bands combinedly in one region, beside the effect of hydrogen bonding (Suha *et al.*, 2019; Robert *et al.*, 2015).

3.2. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ analysis

The $^1\text{H-NMR}$ spectra of prepared monomers DA2, DB2 and DC2 were measured in deuterated dimethyl sulphoxide (DMSO- d_6), the chemical structure and spectra were shown in (Figures 8-10).

The spectrum of DA2 (Figure 8) exhibits the appearance of a singlet signal at (4.88ppm) related to aliphatic protons in position 1, a triplet signal at (7.61-7.65ppm) belonging to protons of the aromatic ring in position 3. Two doublet signals from (8.15ppm) to (8.17ppm) related to protons of aromatic rings in positions 4 and 2 respectively, while a triplet signal appeared at (8.74ppm) belonging to aromatic protons at positions 5.

While spectra of DB2 (Figure 9) observed the appearance of two singlet signals at (3.6ppm) related to aliphatic protons in position 1, another singlet signal observed at 4.36 ppm related to aliphatic protons in position 2, a triplet signal at (7.60-7.63) ppm due to aromatic protons in positions 4. Four doublet signals from (8.13ppm) to (8.38ppm) belong to aromatic protons 5 and 3 respectively, while a triplet signal at (8.46ppm) is related to aromatic protons in position 6.

The $^1\text{H-NMR}$ of DC2 (Figure 10) exhibited two singlet signals at (3.87ppm) and (3.91ppm) related to aliphatic protons in positions 9 and 1 respectively and another singlet signal at (4.06ppm) due to aliphatic protons in position 10 and 15, while a singlet signal at (4.24ppm) related to aliphatic protons in positions 2,3,4 respectively. Triplet signal at (7.62ppm) attributed to protons of aromatic rings in position 6. Four doublet signals from (8.15ppm to 8.32ppm) belong to aromatic protons at positions (7,13) and (5,11), while the triplet signal at (8.47ppm) is related to protons in aromatic rings in positions 8,14 (Robert *et al.*, 2015; Abbas, 2015).

While Figures 11-13 show the $^{13}\text{C-NMR}$ of the prepared monomers.

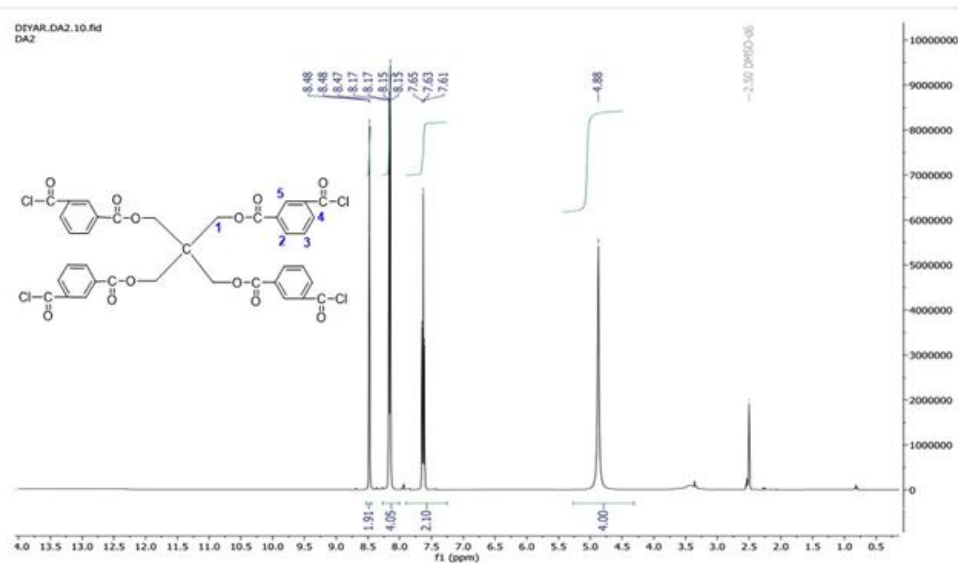


Figure 8. ¹H-NMR spectrum of DA2 monomer in DMSO-d₆

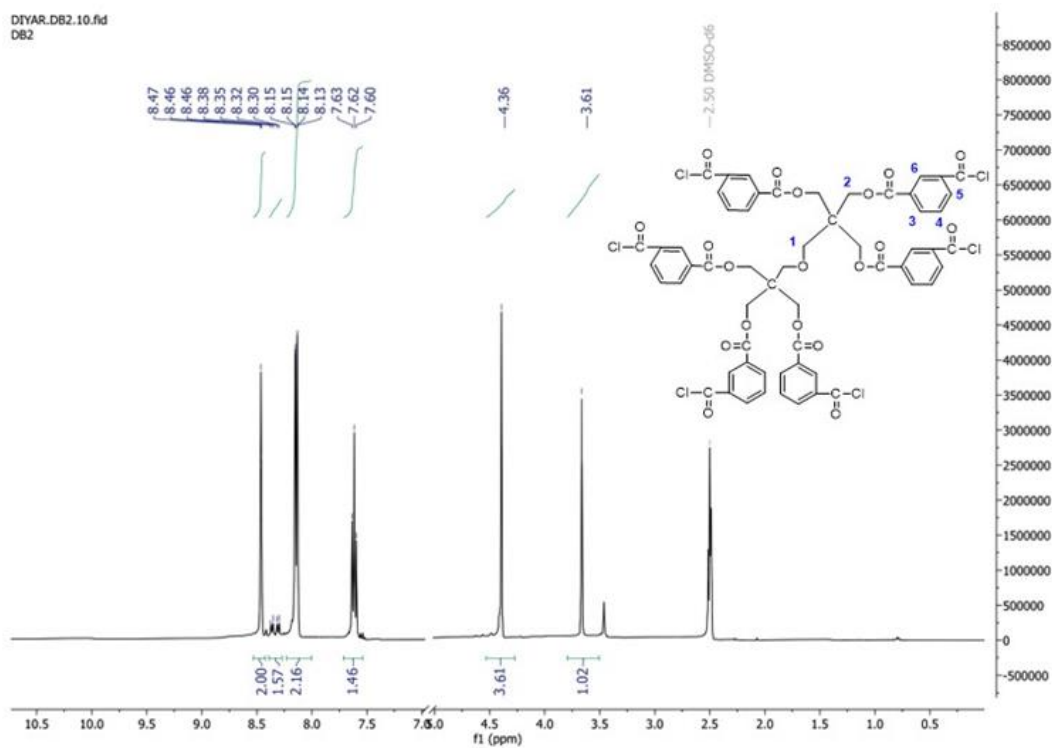


Figure 9. ¹H-NMR spectrum of DB2 monomer in DMSO-d₆

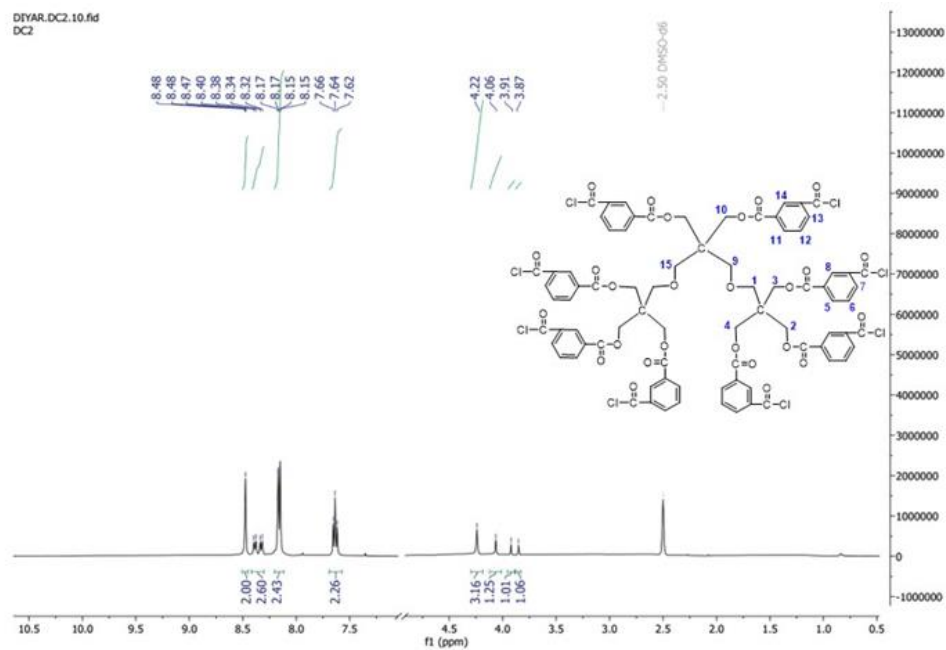


Figure 10. ^1H -NMR spectrum of DC2 monomer in DMSO-d_6

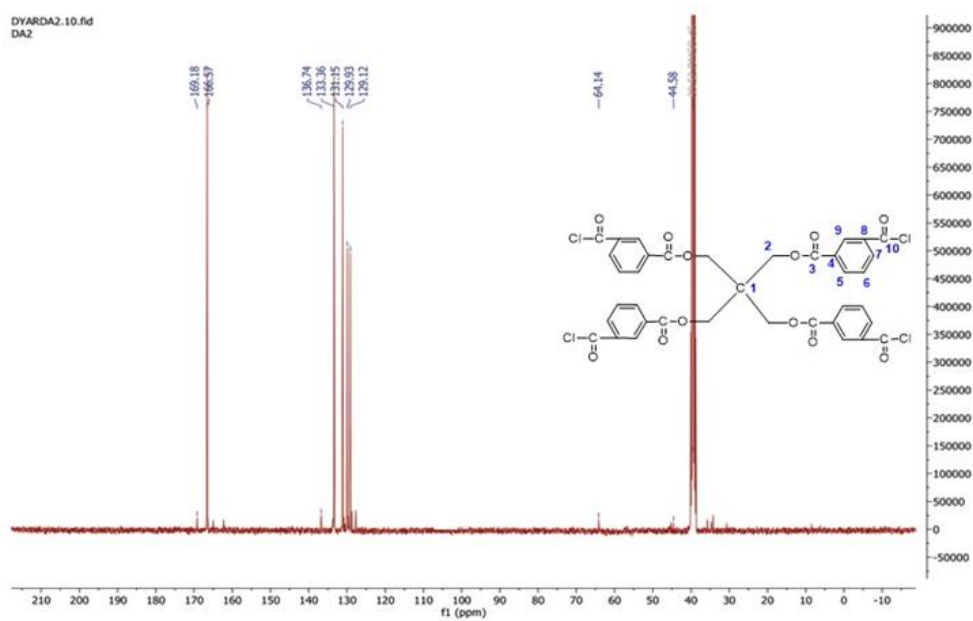


Figure 11. ^{13}C -NMR spectrum of DA2 monomer in DMSO-d_6

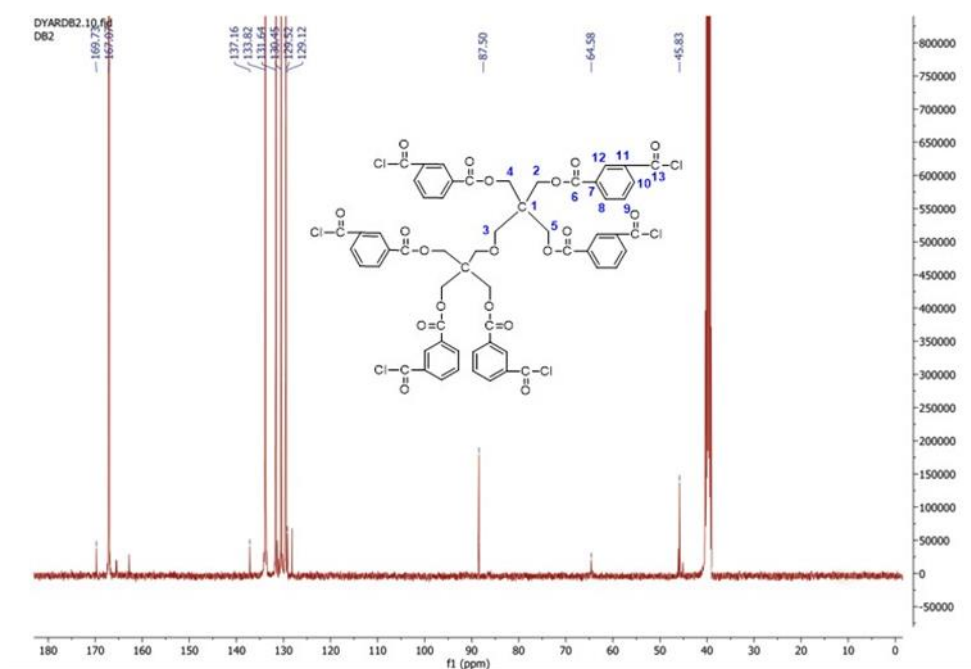


Figure 12. ^{13}C -NMR spectrum of DB2 monomer in DMSO-d_6

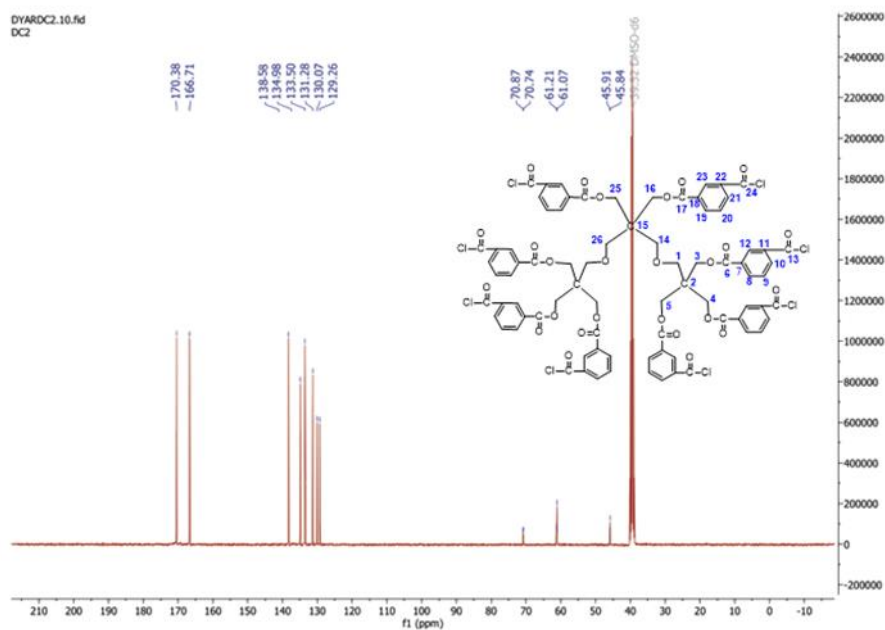


Figure 13. ^{13}C -NMR spectrum of DC2 monomer in DMSO-d_6

3.3. Thermal analysis:

Thermal stability is regarded as one of the main characteristics of polymers, hence their properties are changed as a function of temperature (Muhanad *et al.*, 2022). The partial fragmentation of polymeric chains brought on by exposure to high temperatures has resulted in the thermal degradation of polymers. The nature and composition of

polymers determine pyrolysis. When subjected to heat, polymers' great thermal stability allows them to resist changes in their chains (Baquer *et al.*, 2017).

Many essential functions were measured to understand the behavior and thermal stability of polyamides PA2-PC2, among these functions, are the decomposition temperature, categories to initial degree of dissociation (T_i) and the final degree of dissociation (T_f), also it can calculation of the weight loss at any temperature and measure the remaining from polymer after the char residual process, also calculation the activation energy (Table 1).

The thermogravimetric analysis (TGA) of polyamides PA2-PC2 (Figure 14) was measured at a heating rate of 20°C and under a nitrogen atmosphere, it was revealed that all synthesized polyamides exhibited reasonable thermal stability, the decomposition temperatures ranging from 234°C to 271°C, each polyamide give on decomposition state. Among the series, PB2 displayed the highest thermal stability and high residual polymer. The structural rigidity contributes to greater resistance to thermal degradation. In contrast, the differences in decomposition rates between these polyamides highlight the influence of chemical structure on the thermal behavior of the polyamides (Hadi *et al.*, 2017).

The Broido 1965 model was used to calculate the activation energy (E_a), as described in equations (1) and (2) (Michael *et al.*, 2015):

$$\ln \ln \left[\frac{1}{y} \right] = -\frac{E_a}{RT} \quad (1)$$

$$y = \frac{W_t - W_\infty}{W_i - W_\infty} \quad (2)$$

W_i represents the polyamide's initial weight, while W_t represents the weight of polyamides at any temperature, W_∞ is the final weight of the polyamides, T is the temperature, R is a general constant of gases, a straight line obtained from plotting a graph between $\ln \ln[1/y]$ versus $1/T$ and the slope represent the activation energy.

Table 1. Values obtained from TGA curves of prepared polyamides

SYM.	T_i (°C)	T_f (°C)	T_{50} (°C)	Rate of Decomp. %/min	Activation Energy KJ.mol ⁻¹	Weight Loss %	Char Residue
PA2	251	653	382	3.68	22.11	75.08	24
PB2	271	370	317	7.6	26.6	41.16	59
PC2	234	392	315	11.69	8.56	87.32	14

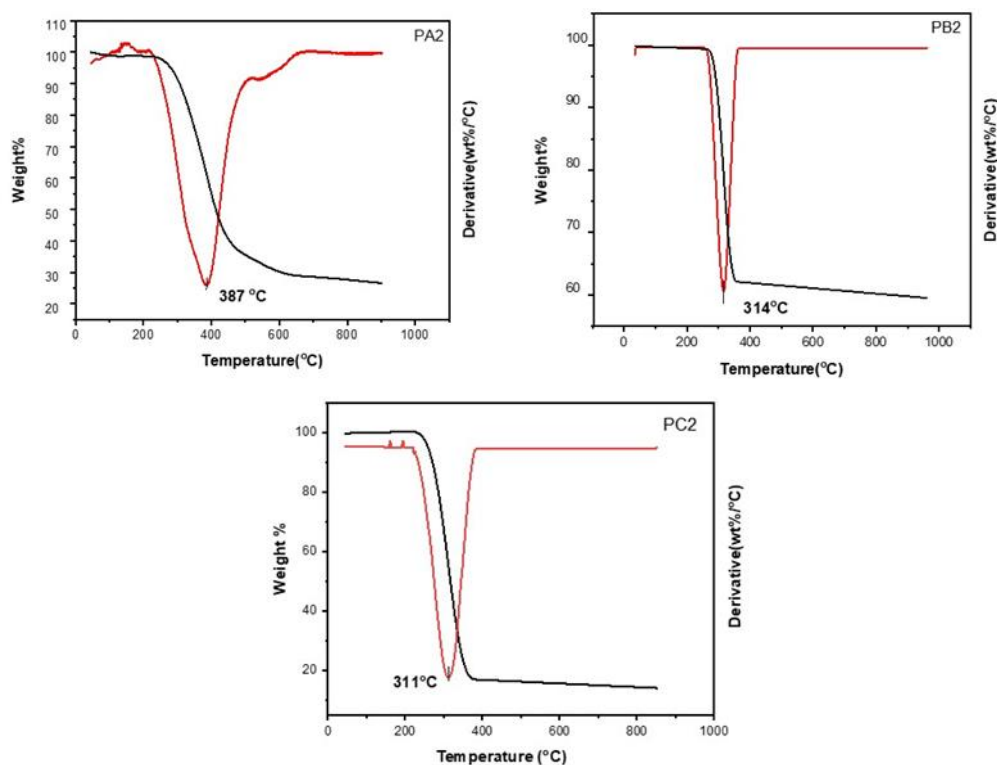


Figure 14. Thermogravimetric analysis (TGA) curves of PA2, PB2 and PC2 indicate their thermal stability. PB2 exhibited the highest decomposition temperature, suggesting enhanced rigidity and intermolecular interactions

3.4. Field emission scanning electron microscopy(FESEM)

The surface morphology of prepared polyamides and by a specific magnification, the particle size of the sample can be determined via FESEM analysis (Field emission scanning electron microscopy), which was used to produce high-resolution images of the material's surface that are less distorted (Safaa *et al.*, 2020) from both (Figures 15 and 16) which represent the FESEM images of two polyamides PA2 and PB2 respectively at low and high magnification can be noted that at lower magnification the two polymers exhibited irregular platelet structure having smooth surfaces, at high magnification, both of them shows nanoparticles, the size and morphology were comparatively spherical particles with narrow size distribution about (55.13 nm 71.11 nm) for PA2 and (45.87 nm and 46.51 nm) for PB2 (Leander *et al.*, 2016). While (Figure 17) shows different scale magnifications of polyamides PC2, it can be seen from it that the polymer gives a sheet-like flower shape structure with non-uniform ends at low magnification (2 μ m-5 μ m), while at high magnification (20 μ m-200 μ m) rod-like morphology was observed (Kutalkova *et al.*, 2018).

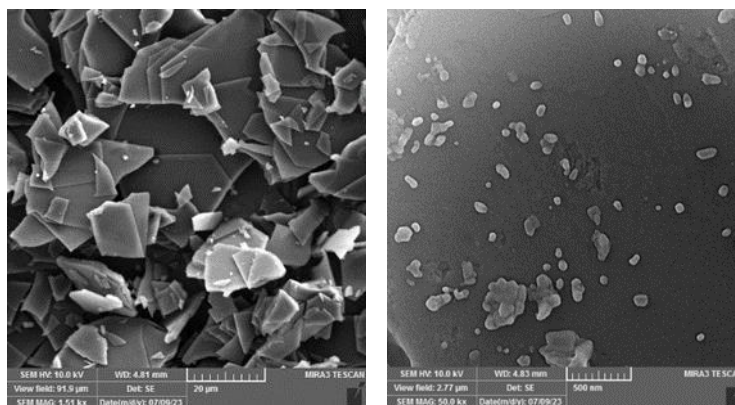


Figure 15. FESEM micrographs of PA2 at low and high magnification

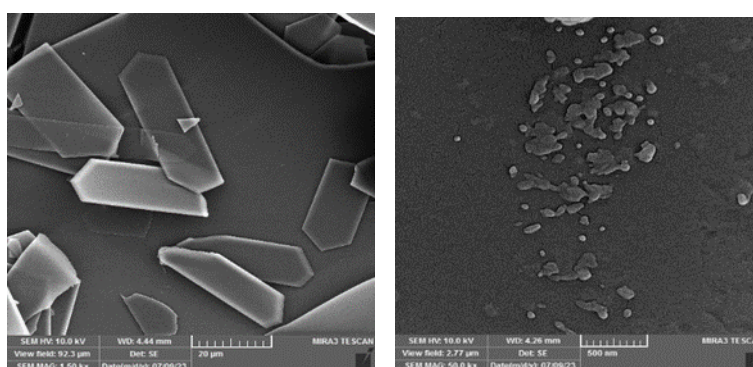


Figure 16. FESEM micrographs of PB2 at low and high magnification

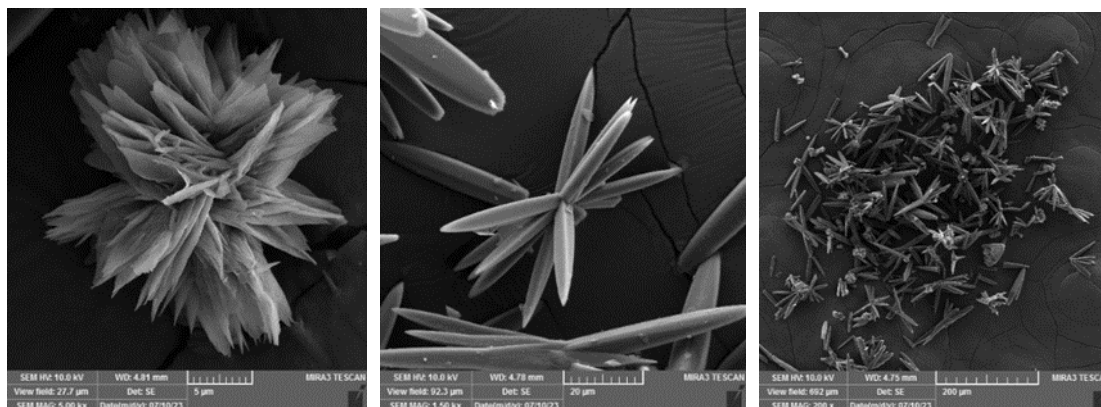


Figure 17. FESEM micrographs of PC2 at different magnification

3.5. Ultraviolet-visible and Fluorescence emission studies

The UV-visible and photoluminescence(PL) properties of the synthesized polyamides were examined at room temperature in DMF solvent (Figure 18), the UV-visible was recorded at (200-800) nm and it was observed from the absorption curves that polymers exhibited low-energy(high wavelength), whereas the absorption maximum at (402-406) nm due to the ($n \rightarrow \pi^*$) bond transition for the carbonyl groups, also the presence of extended π -conjugation systems within the backbone ($\pi \rightarrow \pi^*$) introduced through the aromatic group's monomers, so increasing of wavelength was related to the

increasing of many consecutive double bonds in the prepared polyamides (Hammed *et al.*, 2013; Seonja *et al.*, 2008).

The fluorescence intensity of all prepared polyamides was recorded at (410-700) nm, these polymers exhibited strong green fluorescence light, (Figure 19) and the emission intensity was at (505-511) nm, which matches the ranges of green light, the relation between the structure and fluorescence emission for these prepared polymers was observed clearly. In contrast, many factors affect the decrease of fluorescence intensity such as the restriction in the polymeric chains movement which results from the increasing molecular weight of the polymer and increase in the chain length. The molecular structure and composition, as well as intramolecular or intermolecular reactions, excited-state reactions and energy dispersion, may also cause a decrease in fluorescence intensity (David *et al.*, 2000; Xiaobing, 2016).

Also, the energy gap (E_g) has been calculated and its values were (3.05, 3.06 and 3.08) eV for PA2, PB2 and PC2 respectively, which agree very well with the range of conjugated polymers (Tekalign & Fiseha, 2017), the fluorescence properties observed and the energy gap values enhance that these materials can be promising candidates for applications in optoelectronic devices, fluorescent sensors and imaging materials. Further studies involving quantum yield measurements and photostability evaluations could provide deeper insights into their practical potential.

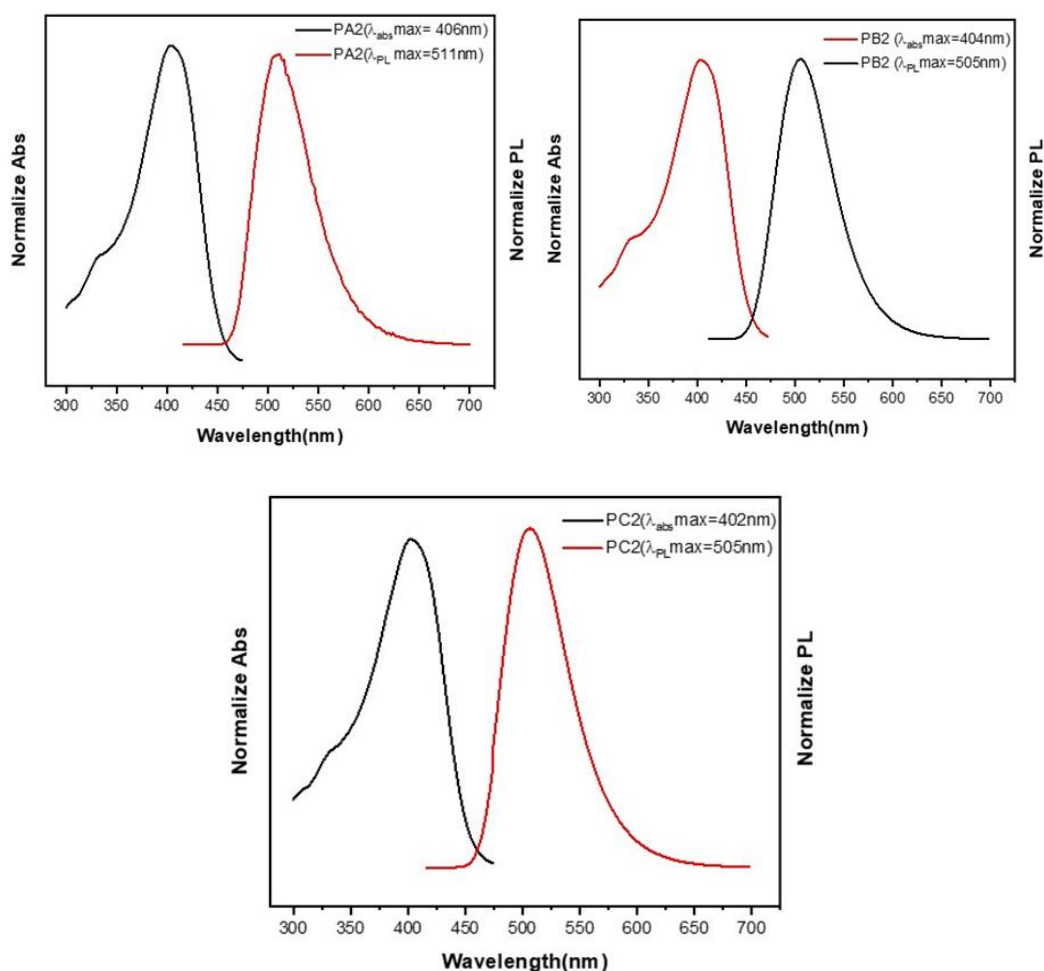


Figure 18. UV-vis absorption and Fluorescence spectra of polyamides PA2, PB2 and PC2

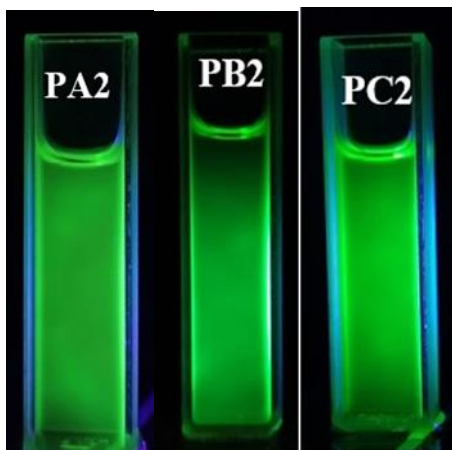


Figure 19. Photographs of polyamides PA2-PC2 in DMF at room temperature

4. Conclusion

In this study, a new series of fluorescent polyamides (PA2, PB2 and PC2) was successfully synthesized and characterized using FT-IR, TGA, FESEM, UV-Vis and fluorescence spectroscopy. These polymers demonstrated strong green fluorescence emission in DMF solvent and displayed high thermal stability, particularly PB2. The structural design integrating aromatic moieties with amide linkages contributed significantly to both fluorescence intensity and thermal performance. The results confirm the potential of these polyamides in applications such as fluorescence-based sensing, photonic devices and advanced materials with thermal resistance. Future studies may focus on modifying the chemical structure to tune the fluorescence behavior, exploring their interaction with metal ions and evaluating their performance in the solid state.

References

- Abbas, A.F. (2015). Synthesis, characterization and antibacterial studies of some new 1, 4-bis (3-phenyl-4, 5-dihydroisoxazol-5-yl) benzene derivatives. *Synthesis*, 7(8), 70-75.
- Al-Khalaf, A.A., Abbas, A.F. & Al-Lami, H.S. (2022). Synthesis and characterization of some new 3, 3'-(1, 4-phenylene) bis (1-(4-aminophenyl) prop-2-en-1-one) amide derivatives. *Basrah Journal of Sciences*, 40(2), 437-464.
- Al-Lami, H.S., Al-Mayyahi, B.A. & Haddad, A.M. (2017). Synthesis and thermal properties of poly (poly lactide-bn-hydroxyethyl acrylamide) nanostar-shape block copolymers. *Journal of Applied Polymer Science*, 1(3), 2.
- Al-Mayyahi, B.A., Haddad, A.M. & Al-Lami, H.S. (2017). Characterization and thermal stability of nano eight arm copolymers synthesized by atom transfer radical polymerization. *Karbala International Journal of Modern Science*, 3(2), 83-92.
- Almayyahi, M.T., Saleh, B.A. & Almayyahi, B.A. (2022). Synthesis and characterization of some new copolyester from curcumin mono-carbonyl analogues. *Biomedicine and Chemical Sciences*, 1(3), 147-159.
- Al-Mosawi, S.K., Al-Hazam, H.A. & Abbas, A.F. (2019). Microwave assisted synthesis, characterization and biological study of some chalcone compounds derived from phenyl isothiocyanate. *Chemistry and Materials Research*, 11(3), 26.
- Al-Mughaid, H., Grindley, T.B. (2006). Selective chemistry of tripentaerythritol Synthesis of acetals and their derivatives. *Canadian Journal of Chemistry*, 84(4), 516-521.

- David, T.M., Anthony, E.P. & Timothy, M.S. (2000). Conjugated polymer-based chemical sensors. *Chem. Rev.*, 100(7), 2537–2574.
- Ghaemy, M., Alizadeh, R. (2011). Synthesis, characterization and photophysical properties of organosoluble and thermally stable polyamides containing pendent N-carbazole group. *Reactive and Functional Polymers*, 71(4), 425-432.
- Hassan, H.H., Elhousseiny, A.F., Elkony, Y.M. & Mansour, E.S.M. (2013). Synthesis and characterization of thermally stable aromatic polyamides and poly (1, 3, 4-oxadiazole-amide) nanoparticles containing pendant substituted bezamides. *Chemistry Central Journal*, 7, 1-17.
- Hu, X. (2017). Novel fluorescent porous hyperbranched aromatic polyamide containing 1, 3, 5-triphenylbenzene moieties: Synthesis and characterization. *Journal of Applied Polymer Science*, 134(8).
- Jeong, S., Kwak, G., Takagi, A., Fujiki, M., Lee, D.H., Park, L.S. & Yoon, K.B. (2008). Luminous, fully aliphatic polyamides: Multicolor photoluminescence, their pH and solvent dependency. *European Polymer Journal*, 44(4), 1149-1156.
- Jiang, X., Wang, Q., Li, B., Li, S. & Kong, X. Z. (2023). Fluorescence behavior and emission mechanisms of poly (ethylene succinamide) and its applications in Fe³⁺ detection and data encryption. *Chinese Journal of Polymer Science*, 41(1), 129-142.
- Kasa, T., Gebrewold, F. (2017). Polymers and its application in light emitting diodes: A review article. *Advances in Physics Theories and Applications*, 2225-0638.
- Kutalkova, E., Plachy, T., Osicka, J., Cvek, M., Mrlik, M. & Sedlacik, M. (2018). Electrorheological behavior of iron (II) oxalate micro-rods. *RSC Advances*, 8(44), 24773-24779.
- McQuade, D.T., Pullen, A.E. & Swager, T.M. (2000). Conjugated polymer-based chemical sensors. *Chemical Reviews*, 100(7), 2537-2574.
- Mei, J., Leung, N.L., Kwok, R.T., Lam, J.W. & Tang, B.Z. (2015). Aggregation-induced emission: Together we shine, united we soar!. *Chemical Reviews*, 115(21), 11718-11940.
- Miriam, T.L., Jose, M.G., Jose, A.R.R., Felix, C.G. & Ramon, F. (2018). *Encyclopedia of Polymer Science and Technology*, 1st edition. John Wiley & Sons, 1-51.
- Modh, J.V. (2023). Synthetic protocols and significance of heterocyclic fluorescent-reinforcing polymers: A comprehensive review. *Journal of Advanced Scientific Research*, 14(2), 9-39.
- Mohamed, S. H., Hameed, A.S., Yousif, E., Alotaibi, M.H., Ahmed, D.S. & El-Hiti, G.A. (2020). New porous silicon-containing organic polymers: Synthesis and carbon dioxide uptake. *Processes*, 8(11), 1488.
- Mohammad, A.A., Tatsuo, K. (2015). Polyamide syntheses. *Encyclopedia of Polymeric Nanomaterials*. Springer-Verlag Berlin Heidelberg.
- Nechifor, M. (2009). Synthesis and properties of some aromatic polyamides with coumarin chromophores. *Reactive and Functional Polymers*, 69(1), 27-35.
- Ofem, M. I., Muhammed, M. & Umar, M. (2015). Thermal properties of chitin whiskers reinforced poly (acrylic acid). *International Journal of Scientific & Technology Research*, 4(9), 281-288.
- Robert, M.S., Francis, X.W., David, J.K. & David, L.B. (2015). *Spectrometric Identification of Organic Compounds*, 8th edition. John Wiley & Sons.
- Teo, N.K.S., Fan, B., Ardana, A. & Thang, S.H. (2024). Aggregation-induced emission polymers via reversible-deactivation radical polymerization: Special collection: Distinguished Australian researchers. *Aggregate*, 5(1), e414.
- Tjong, S.C. (2006). Structural and mechanical properties of polymer nanocomposites. *Materials Science and Engineering: R: Reports*, 53(3-4), 73-197.
- Verbelen, L., Dadbakhsh, S., Van den Eynde, M., Kruth, J.P., Goderis, B. & Van Puyvelde, P. (2016). Characterization of polyamide powders for determination of laser sintering processability. *European Polymer Journal*, 75, 163-174.