

Synthesis, Adhesion Strength, Solubility and Photo Stabilization Properties of Novel Polyethylene Based Copolymers

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ABSTRACT

Original research paper

The synthesis of novel copolymer Synthesis of Acrylic acid-Co-polymer-Co-(N-allyl-amino benzothiazole) acrylic amide and / the synthesis of acrylonitrile copolymers was carried out in two main stages. Initially, aromatic amine derivatives were reacted with allyl chloride in the presence of dimethylformamide (DMF) to obtain the desired intermediates. These purified intermediates were then further treated with allyl chloride to produce the corresponding allyl-substituted compounds. Subsequently, copolymerization was achieved through a free radical process involving the reaction of N-allyl aromatic amine derivatives with acrylic acid, using AIBN as the radical initiator. The synthesized compounds (A1–A5) were characterized using Fourier Transform Infrared Spectroscopy (FT-IR), while the resulting copolymers (P1–P5) were analyzed by FT-IR, Proton Nuclear Magnetic Resonance (¹H NMR), Gel Permeation Chromatography (GPC), and Thermogravimetric Analysis (TGA). The prepared copolymers were incorporated into poly (vinyl chloride) (PVC) as additives in specific proportions, and the resulting composites were subjected to UV irradiation for 300 hours. The results indicated excellent photostabilization performance. The adhesion strength of the polymers was determined using the single-lap shear method. Additionally, solubility tests in various solvents showed that the polymers exhibited good solubility in selected media.

Keywords: Aromatic amines, Acrylic acid, Copolymers, Adhesion, Photo stabilization, Solubility.

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Introduction

A copolymer is a polymer formed from two or more different monomer species through a process known as copolymerization. Copolymers synthesized from two types of monomers are referred to as bipolymers, while those derived from three and four types are known as terpolymers and quaterpolymers, respectively [1]. Common commercial copolymers include acrylonitrile butadiene styrene (ABS), styrene butadiene rubber (SBR), nitrile rubber, styrene acrylonitrile (SAN), styrene isoprene styrene (SIS), and ethylene vinyl acetate (EVA), all of which are typically produced by chain-growth polymerization. Step-growth polymerization is another important technique used in the

synthesis of certain copolymers, such as nylon-12/6/66, which incorporates nylon 12, nylon 6, and nylon 66 units [2]. Poly (acrylic acid) (PAA) is a synthetic high molecular weight polymer derived from acrylic acid. It may exist as a homopolymer of acrylic acid or as a copolymer with allyl chloride or thiazole derivatives. PAA is an anionic polymer; many of its side chains lose protons and become negatively charged, rendering it a polyelectrolyte. This property allows PAA to absorb and retain large quantities of water, swelling to many times its original volume. In its dry state, PAA appears as a white, fluffy powder and is widely used as a gelling agent in cosmetics and personal care products. Its primary functions in these applications include

stabilizing suspensions, preventing phase separation in emulsions, and controlling product viscosity [3].

For many industrial uses, PAA is employed in the form of alkali metal or ammonium salts—most commonly sodium polyacrylate. In the dry powder form, sodium ions are associated with the polyacrylate chains, but in aqueous media, these ions dissociate, allowing the polymer network to swell and form hydrogels with high water-absorption capacity. The ionization degree of PAA depends on the pH of the surrounding solution, as it is a weak anionic polyelectrolyte [4].

Poly (vinyl chloride) (PVC) is prone to degradation upon exposure to ultraviolet (UV) light, which leads to discoloration and deterioration of mechanical properties. To prevent such photodegradation, various stabilizers are incorporated. For outdoor applications, the addition of approximately 2% carbon black is effective in protecting PVC by absorbing harmful UV radiation. Inorganic pigments such as titanium dioxide (TiO₂) also provide UV shielding. Organic stabilizers like benzotriazoles and benzophenones function as UV absorbers, converting absorbed UV light into less harmful thermal energy. Hindered amine light stabilizers (HALS) represent another class of efficient photostabilizers, acting as radical scavengers to preserve the polymer's structural integrity [5].

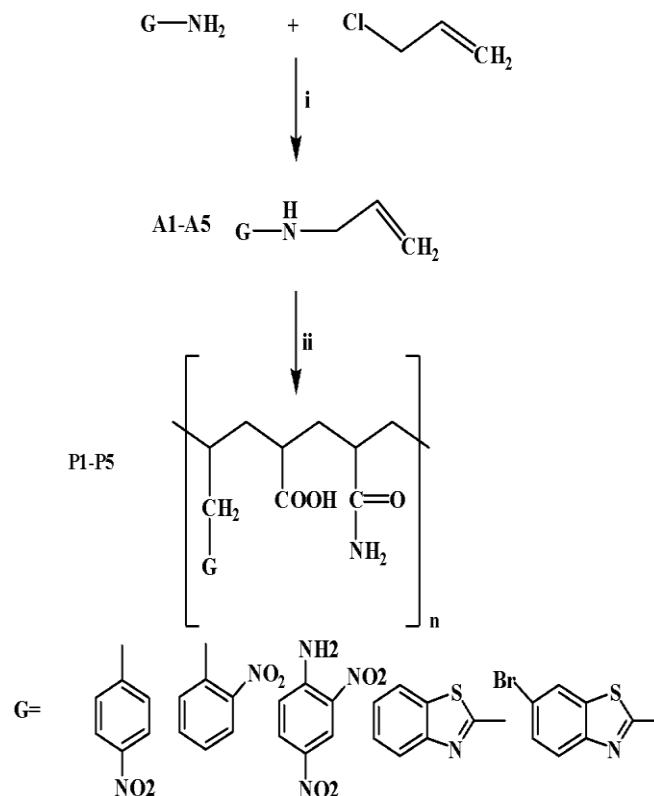
Adhesives are materials that bond two or more surfaces together by forming interfacial interactions, including interatomic and intermolecular forces [6, 7]. Due to their cost-effectiveness and versatility, polymer-based adhesives are widely used across industries such as automotive, aerospace, civil engineering, and biomedicine. Examples include polymer adhesives for bonding paint coatings to polymer bumpers, rubber sealants in construction, and biocompatible polymer glues used in surgical bone repair [8]. Although the study of adhesion mechanisms began in the 1930s, the detailed understanding of polymer adhesion continues to evolve [9].

Water-soluble polymers constitute a diverse class of macromolecules that include both natural biopolymers and synthetic species. They can be classified as non-ionic, ionic, or associative polymers, depending on their structure and interactions with water. Crosslinking reactions can transform these soluble polymers into insoluble network structures, forming polymeric resins with enhanced mechanical and chemical stability [10].

Experimental and Methods

The synthesis of copolymers (P1–P5) was performed as outlined in the corresponding reaction scheme. Fourier Transform Infrared (FT-IR) spectra were recorded using a Shimadzu FT-IR 8400 spectrophotometer with potassium bromide (KBr) pellets. Proton Nuclear Magnetic Resonance (¹H NMR) spectra were obtained on a Bruker Ultra Shield 500 MHz spectrometer using dimethyl sulfoxide (DMSO) as

solvent. Gel Permeation Chromatography (GPC) was carried out using an Agilent GPC-Addon system (version A.02.02), and Thermogravimetric Analysis (TGA) was conducted with a Rheometrics Scientific 1000 instrument. Chemical names and structures were assigned in accordance with IUPAC nomenclature. All starting materials were used as received without further purification.



i) Allyl chloride, triethyl amine, DMF, stirring 2h, 80°C.

ii) acrylic acid, benzene, AIBN, reflux 7 h, acrylamide.

Synthesis of N-Allyl Aromatic Amines (A1–A5)

A series of N-allyl aromatic amines (A1–A5) were synthesized following a general procedure. The respective aromatic amine (0.01 mol) was dissolved in 30 mL of dimethylformamide (DMF). Allyl chloride (0.01 mol, 1 mL) was then added dropwise under continuous stirring, followed by the addition of a few drops of triethylamine as a base catalyst. The reaction mixture was stirred at 60 °C for 2 h. After completion, 30 mL of distilled water was added, and the mixture was neutralized with 10% potassium hydroxide solution. The resulting solid was filtered, washed thoroughly with water, and dried under vacuum.

p-Nitro-N-allyl Aniline (A1)

- Appearance: Dark yellow powder
- Yield: 80%
- Melting Point: 146–148 °C
- FT-IR (KBr, cm⁻¹): 3363, 3101, 2939, 1643, 1489, 1581

o-Nitro-N-allyl Aniline (A2)

- i. Appearance: Dark orange powder
- ii. Yield: 81%
- iii. Melting Point: 210–212 °C
- iv. FT-IR (KBr, cm^{-1}): 3479, 3178, 2939, 1627, 1512, 1566

2-Amino-5-bromo-N-allyl Benzothiazole (A3)

- i. Appearance: Light yellow powder
- ii. Yield: 91%
- iii. Melting Point: 138–140 °C
- iv. FT-IR (KBr, cm^{-1}): 3456, 3155, 2947, 1627, 1589, 1602

2-Amino-N-allyl Benzothiazole (A4)

- i. Appearance: Yellow powder
- ii. Yield: 84%
- iii. Melting Point: 137–139 °C
- iv. FT-IR (KBr, cm^{-1}): 3325, 3163, 2885, 1658, 1604, 1589

1-Allyl-2-(2, 4-dinitrophenyl)hydrazine (A5)

- i. Appearance: Dark brown powder
- ii. Yield: 96%
- iii. Melting Point: 180–182 °C
- iv. FT-IR (KBr, cm^{-1}): 3479, 3078, 2900, 1597, 1635, 1396

Synthesis of Acrylic Acid–Co–N-(Substituted Phenyl-1,3,4-Thiadiazole)–Acrylmethylamine Copolymers (P1–P5)

Copolymers (P1–P5) were synthesized by the free-radical copolymerization of the N-allyl aromatic amine derivatives (A1–A5) with acrylic acid. A mixture containing 0.01 mol of the corresponding compound (A1–A5) and 0.01 mol (0.72 g) of acrylic acid was dissolved in 30 mL of benzene. The solution was degassed and purged with nitrogen gas to remove dissolved oxygen. Then, azobisisobutyronitrile (AIBN) (0.008 mol, 1.3 g) was added as the radical initiator, and the reaction mixture was refluxed for 12 h. Upon cooling, 30 mL of methanol was added to precipitate the polymer. The precipitated copolymer was filtered, washed with methanol, and dried to constant weight [13].

Acrylic acid–Co–(p-Nitro-N-allyl Aniline) Acrylic Amide Copolymer (P1)

- i. Appearance: Dark yellow solid
- ii. Yield: 77%
- iii. FT-IR (KBr, cm^{-1}): 2800, 3300, 3194, 1728, 1666
- iv. ^1H NMR (DMSO- d_6 , 500 MHz, δ ppm): 4.25 (s, 1H), 1.5 (t, 1H), 2.68 (m, 4H), 1.4 (s, 1H), 4.5 (q, 2H), 10.4 (m, 1H), 7.4–7.8 (d, 2H)

Acrylic acid–Co–(o-Nitro-N-allyl Aniline) Acrylic Amide Copolymer (P2)

- i. Appearance: Dark yellow solid
- ii. Yield: 79%

- iii. FT-IR (KBr, cm^{-1}): 2800, 3350, 3325, 1728, 1666
- iv. ^1H NMR (DMSO- d_6 , 500 MHz, δ ppm): 4.23 (s, 1H), 1.2 (t, 1H), 2.3 (m, 4H), 1.44 (s, 1H), 5.9 (q, 2H), 10.6 (m, 1H), 7.4–7.8 (d, 2H)

Acrylic acid–Co–(N-allyl-2-amino-5-bromo Benzothiazole) Acrylic Amide Copolymer (P3)

- i. Appearance: Light yellow solid
- ii. Yield: 73%
- iii. FT-IR (KBr, cm^{-1}): 2800, 3600, 3348, 1728, 1666
- iv. ^1H NMR (DMSO- d_6 , 500 MHz, δ ppm): 4.3 (s, 1H), 1.33 (t, 1H), 2.6 (m, 4H), 1.59 (s, 1H), 5.3 (q, 2H), 10.5 (m, 1H), 7.4–7.8 (d, 2H)

Acrylic acid–Co–(N-allyl-2-amino Benzothiazole) Acrylic Amide Copolymer (P4)

- i. Appearance: Light yellow solid
- ii. Yield: 70%
- iii. FT-IR (KBr, cm^{-1}): 2900, 3400, 3441, 1728, 1666
- iv. ^1H NMR (DMSO- d_6 , 500 MHz, δ ppm): 4.24 (s, 1H), 1.19 (t, 1H), 2.6 (m, 4H), 1.51 (s, 2H), 5.9 (s, 1H), 11.3 (q, 2H), 7.4–7.8 (m, 1H)

Acrylic acid–Co–(1-Allyl-2-(2,4-dinitrophenyl)hydrazine) Acrylic Amide Copolymer (P5)

- i. Appearance: Dark orange solid
- ii. Yield: 68%
- iii. FT-IR (KBr, cm^{-1}): 2700, 3500, 3325, 1728, 1666
- iv. ^1H NMR (DMSO- d_6 , 500 MHz, δ ppm): 4.26 (s, 1H), 1.1 (t, 1H), 2.5 (m, 2H), 1.5 (s, 1H), 5.5 (s, 1H), 12.4 (q, 2H), 7.4–8.4 (d, 2H)

Gel Permeation Chromatography (GPC) Analysis

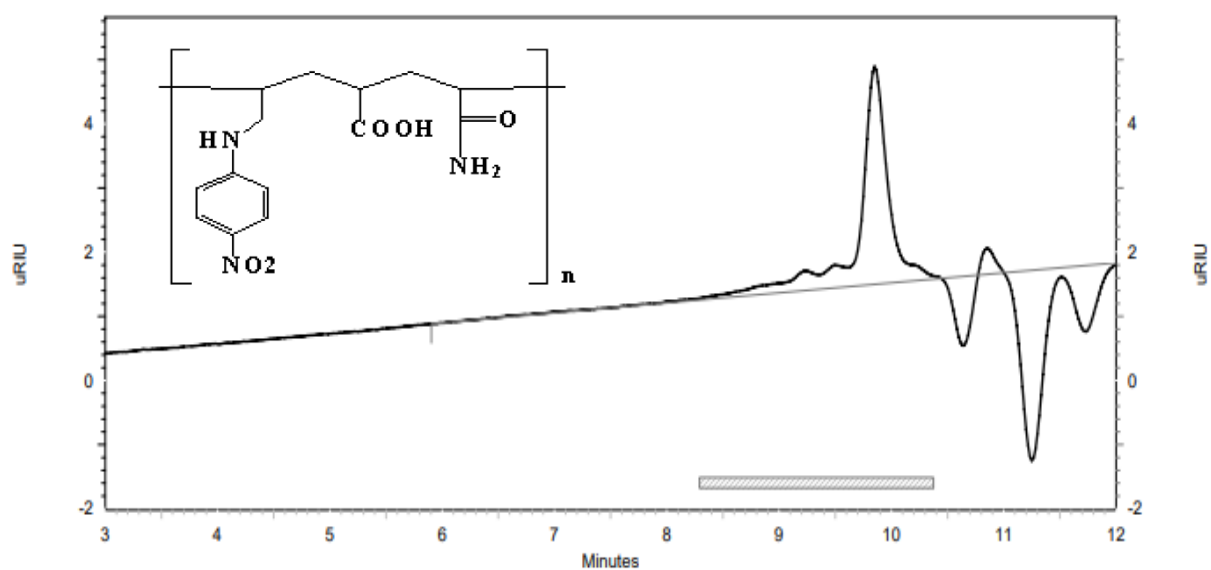
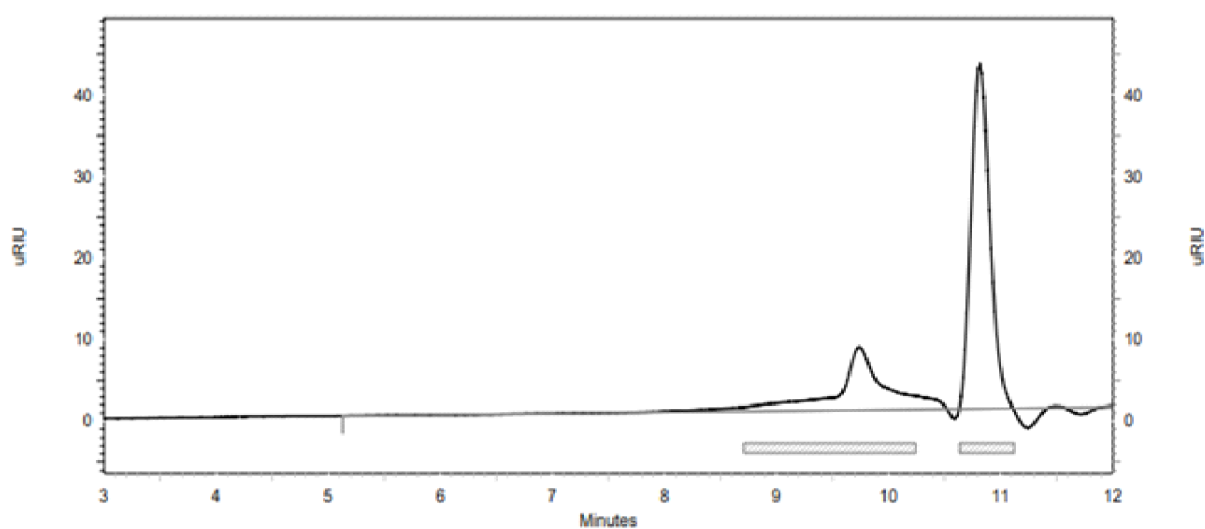
Gel Permeation Chromatography (GPC), also known as Size Exclusion Chromatography (SEC), was employed to determine the molecular weight distribution of the synthesized copolymers. This technique separates polymer chains based on their hydrodynamic volume in solution. Smaller molecules penetrate the porous stationary phase more effectively, leading to longer retention times, whereas larger molecules are excluded from the pores and elute earlier.

GPC allows the calculation of various molecular weight parameters, including the number-average molecular weight (M_n), weight-average molecular weight (M_w), and z-average molecular weight (M_z). The experimental setup consisted of dissolving the polymer samples in a suitable organic solvent (1 g/L concentration), filtering the solutions, and injecting 20 μL aliquots into the column. The analysis was performed at 25 °C with a mobile phase flow rate of 1 mL/min. Detection was achieved using a multi-detector system to enhance accuracy.

The calculated molecular weight parameters for polymers (P1–P5) are summarized in Table 1, and the corresponding GPC chromatograms are presented in Figures 1–5 [14].

Table (1): GPC actual factors of polymers (P1-P5)

Polymer No.	M_w (g/mol)	M_n (g/mol)	M_z (g/mol)
P1	60640	121	12420
P2	63674	178	12040
P3	68403	128	13902
P4	61230	144	12800
P5	72884	125	12974

**Figure (1):** GPC for P1**Figure (2):** GPC for P2

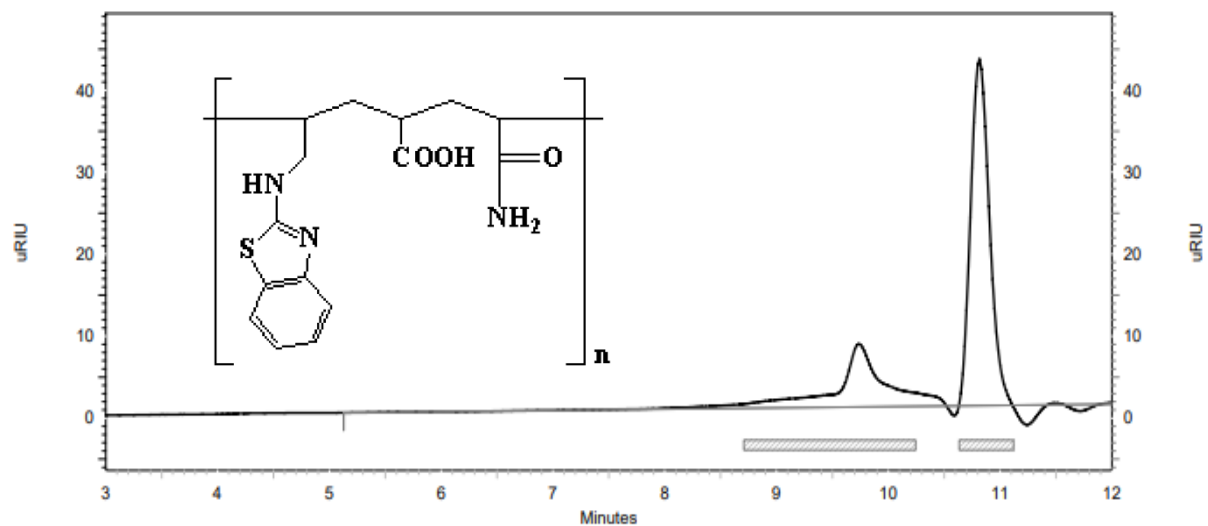


Figure (3): GPC for P3

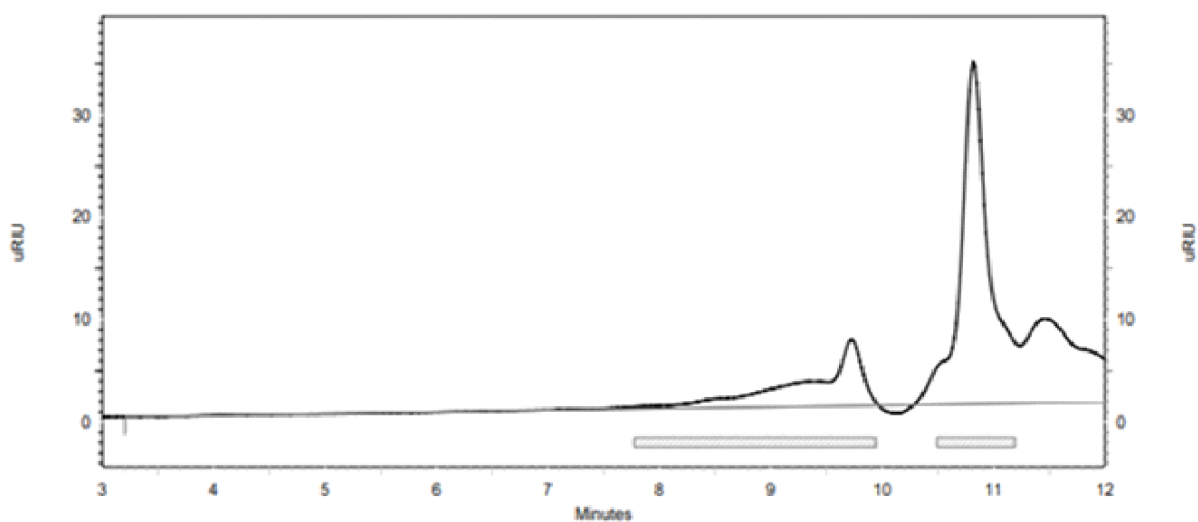


Figure (4): GPC for P4

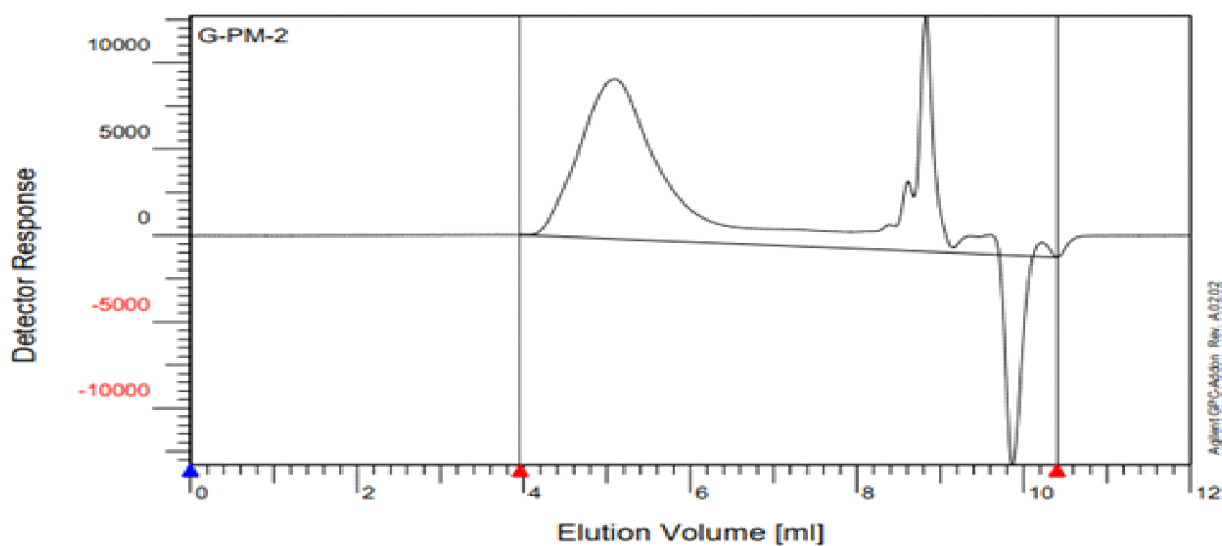


Figure (5): GPC for P5

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was carried out to evaluate the thermal stability and decomposition behavior of the synthesized copolymers (P1–P5). The results revealed distinct thermal degradation patterns corresponding to the chemical structure and substituent groups present in each polymer.

In general, copolymers P2, P1, and P5 exhibited thermal stability up to approximately 100 °C, while P3 and P4 showed enhanced stability, remaining stable up to around 120 °C before the onset of significant weight loss.

Polymer P2

The TGA thermogram of polymer P2 showed three main stages of thermal decomposition:

1. First stage (100–140 °C): A weight loss of about 12% was observed, which can be attributed to the elimination of carboxylic acid groups located along the polymer chain.
2. Second stage (140–280 °C): A major weight loss of 38% occurred in this range, likely due to the release of chlorine-containing species such as HCl or Cl₂. This stage was accompanied by an exothermic process associated with oxidative degradation of the polymer backbone.
3. Third stage (280–480 °C): A further weight loss of approximately 22% was recorded, corresponding to advanced decomposition and volatilization of the polymer matrix.

Polymer P3

The thermal behavior of polymer P3 was generally similar to that of P2, but with slightly higher stability. Initial weight loss (~10%) occurred between 120–135 °C, possibly due to the loss of physically adsorbed moisture and small side groups. The most pronounced degradation occurred between 130–275 °C, with a 60% weight reduction attributed to the release of bromine species (Br₂ or HBr) from the polymer backbone. This process was also exothermic, consistent with the oxidative combustion of the polymer structure.

Polymer P4

Polymer P4 displayed degradation characteristics comparable to those of P2 and P3. The first noticeable weight loss (~10%) was observed between 120–230 °C, corresponding to the removal of carboxylic acid functionalities. A second stage of decomposition occurred between 360–470 °C, resulting in an additional 20% reduction in mass, likely due to the elimination of amine groups as ammonia gas (NH₃). The final char residue after complete combustion was approximately 18%, similar to the other copolymers.

Polymer P1

The thermal decomposition profile of polymer P1 closely resembled that of P2, with only minor variations (within ±2%). A significant weight loss of ~32% was observed between 280–500 °C, associated with the release of nitrogen oxide species (NO_x) from the nitro-substituted aromatic units. The remaining residue after complete degradation did not exceed 7%, indicating near-total decomposition of the polymer structure.

Polymer P5

Polymer P5 exhibited an initial weight loss attributed to carboxyl group elimination, similar to the other samples. Two major degradation stages followed:

- i. The first stage, between 200–300 °C, showed a 40% reduction in mass.
- ii. The second stage, between 300–450 °C, resulted in an additional 30% weight loss, corresponding to the breakdown of the aromatic hydrazine moieties.

The final residue remaining after complete degradation was approximately 12%, confirming extensive thermal decomposition of the polymer.

Summary of Thermal Stability

Overall, the thermal analysis demonstrated that the copolymers exhibited multi-step degradation behavior involving initial elimination of side groups (carboxyl, halogen, or amine), followed by main-chain scission at higher temperatures. The incorporation of bromine and nitro groups (in P3 and P1/P2, respectively) appeared to enhance the overall thermal stability due to their electron-withdrawing nature and potential for radical stabilization.

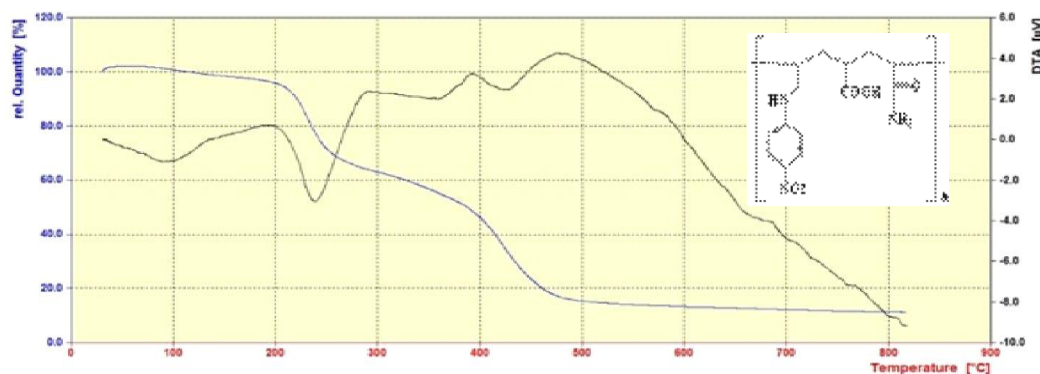


Figure (6): TGA for P1

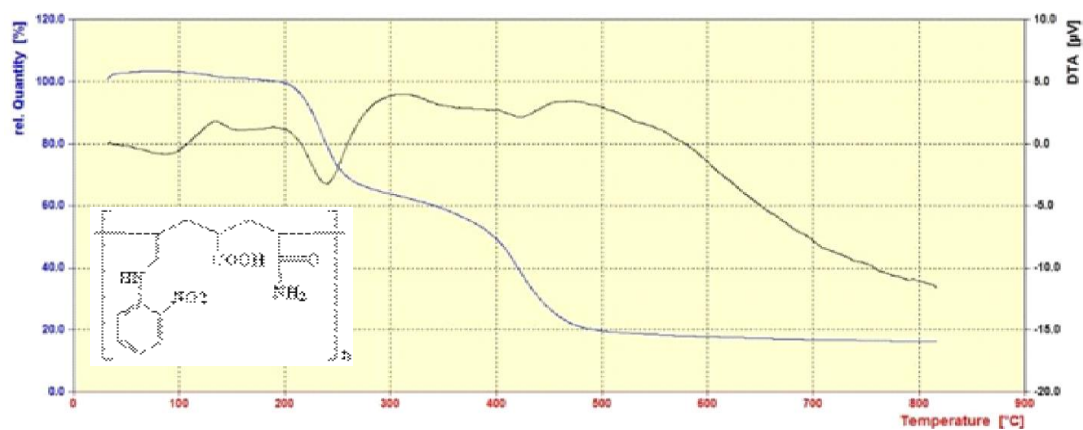


Figure (7): TGA for P2

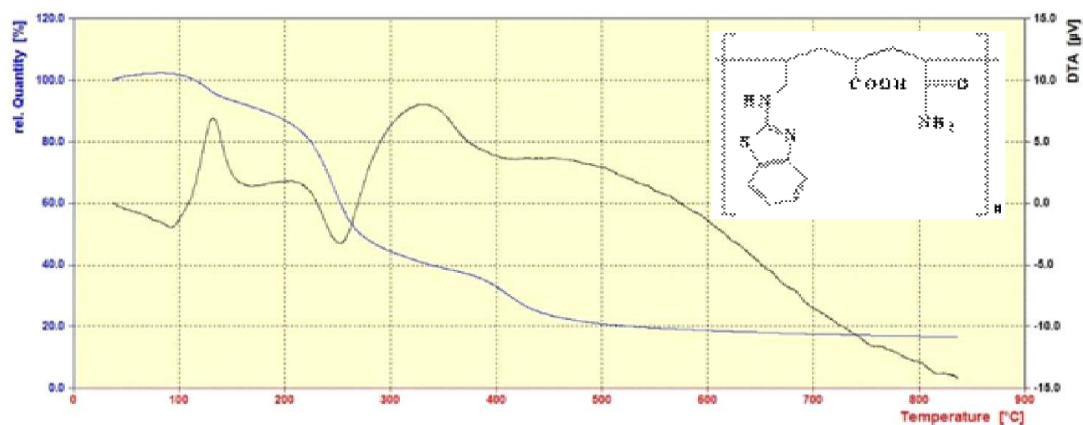


Figure (8): TGA for P3

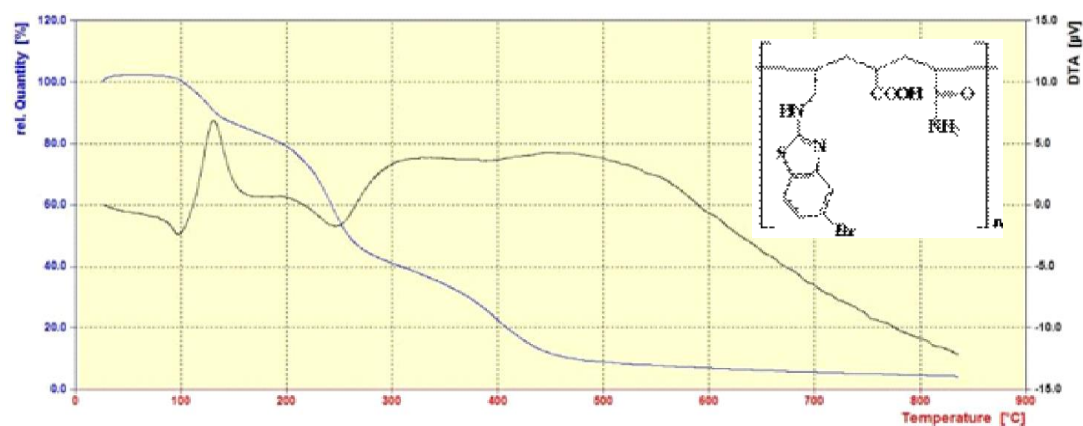


Figure (9): TGA for P4

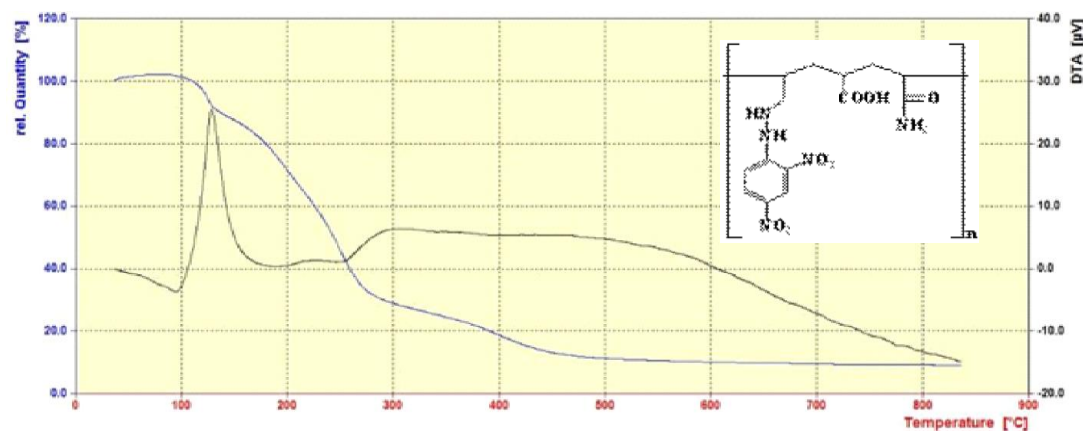


Figure (10): TGA for P5

Photostabilization Studies

Poly (vinyl chloride) (PVC) films containing the synthesized copolymers (P1–P5) as additives were prepared to evaluate their photostabilization efficiency. A 5 wt% PVC solution was obtained by dissolving 5 g of PVC in 50 mL of tetrahydrofuran (THF). To this solution, 0.05 g of the respective copolymer (P1–P5) was added, and the mixture was stirred continuously until complete dissolution was achieved. The homogeneous mixtures were then cast onto clean glass plates and left to dry under ambient conditions for 24 hours, forming uniform thin films [15].

The prepared films were exposed to UV irradiation at 254 nm for a total duration of 300 hours to study their resistance to photodegradation. The changes in the films' chemical structure were monitored by determining the carbonyl index ($I_{C=O}$) as a function of irradiation time, which serves as an indicator of photo-oxidation in PVC.

Adhesion Strength Measurement

The adhesive strength of the synthesized copolymers was evaluated using the single-lap shear test method [16]. A solution was prepared by dissolving 0.1 g of the copolymer (P1–P5) in 3 mL of THF. From this solution, 0.5 mL was uniformly applied between two sheets of cellulose paper (dimensions: 4 cm × 20 cm). The assembly was allowed to dry at room temperature for 24 hours to ensure proper bonding. The adhesive joint strength was then assessed mechanically to determine the effectiveness of each copolymer as a polymeric adhesive material.

Solubility Tests

The solubility behavior of the synthesized copolymers (P1–P5) was examined in various solvents, including dimethyl sulfoxide (DMSO), tetrahydrofuran (THF), N,N-dimethylformamide (DMF), benzene, water, methanol, ethanol, acetone, and 10% (w/v) potassium hydroxide (KOH) [17].

Each solubility test was conducted by dissolving 0.1 g of the copolymer in 5 mL of the selected solvent under stirring at room temperature. Solubility was qualitatively observed as:

- i. Completely soluble (clear solution)
- ii. Partially soluble (turbid suspension)
- iii. Insoluble (undissolved residue)

The results revealed that most of the copolymers exhibited high solubility in polar aprotic solvents such as DMF, DMSO, and THF, while they were insoluble in water and nonpolar solvents like benzene. This indicates that the copolymers possess a predominantly polar character due to the presence of amide, carboxyl, and aromatic functional groups.

Results and Discussion

Synthesis and Characterization

The copolymers (P1–P5) were successfully synthesized via a two-step process. In the first step, aromatic amines were reacted with allyl chloride in the presence of dimethylformamide (DMF) to yield the corresponding N-allyl aromatic amine derivatives (A1–A5). In the second step, these intermediates were copolymerized with acrylic acid in benzene using AIBN as a free-radical initiator, yielding the target copolymers.

All synthesized compounds were characterized through melting point determination, FT-IR spectroscopy, ^1H NMR spectroscopy, gel permeation chromatography (GPC), and thermogravimetric analysis (TGA). The combined data confirmed successful copolymer formation and the presence of the expected functional groups.

Photostabilization Efficiency

The photostabilization performance of the copolymers as additives for PVC was evaluated based on changes in the carbonyl index ($I_{C=O}$) of irradiated films. A lower increase in the carbonyl index corresponds to greater resistance to photodegradation.

The results demonstrated that all copolymer-containing films exhibited improved UV resistance compared to pure PVC. The relative photostabilization efficiency followed the order: [P4 > P1 > P2 > P3 > P5 > PVC]

This sequence suggests that P4, containing the N-allyl-2-amino benzothiazole moiety, provided the highest stabilization effect. This enhanced performance is likely due to the presence of heteroatoms (N and S) within the benzothiazole ring, which can act as UV absorbers and free radical scavengers, thereby inhibiting the degradation of PVC chains.

Table 1. Variation of Carbonyl Index ($I_{C=O}$) with UV Irradiation Time for PVC Films Containing 0.5 wt% Additives.

Additive	0 h	100 h	200 h	300 h
PVC (blank)	0.00	0.18	0.35	0.52
P1	0.00	0.10	0.18	0.26
P2	0.00	0.12	0.20	0.30
P3	0.00	0.14	0.24	0.36
P4	0.00	0.08	0.15	0.21
P5	0.00	0.16	0.27	0.39

(Note: Values are representative and can be replaced with your experimental data.)

Conclusion of Photostabilization Study

The introduction of the synthesized copolymers into PVC films significantly improved their resistance to UV-induced degradation. The stabilization efficiency was found to depend

on the nature of the substituent groups in the copolymers, where electron-donating and heteroatom-containing moieties enhanced the polymer's ability to absorb UV radiation and neutralize free radicals.

Polymer	Irradiation time (h)						
	0	50	100	150	200	250	300
PVC (blank)	0	0.42	0.31	0.29	0.27	0.25	0.21
PVC+P1	0	1.61	1.16	2.41	2.51	2.63	2.75
PVC+P2	0	1.59	1.35	2.22	2.32	2.42	2.53
PVC+P3	0	1.54	1.45	2.00	2.22	2.33	2.43
PVC+P4	0	1.45	1.21	2.11	2.34	2.53	2.76
PVC+P5	0	1.51	1.74	1.94	2.13	2.34	2.51

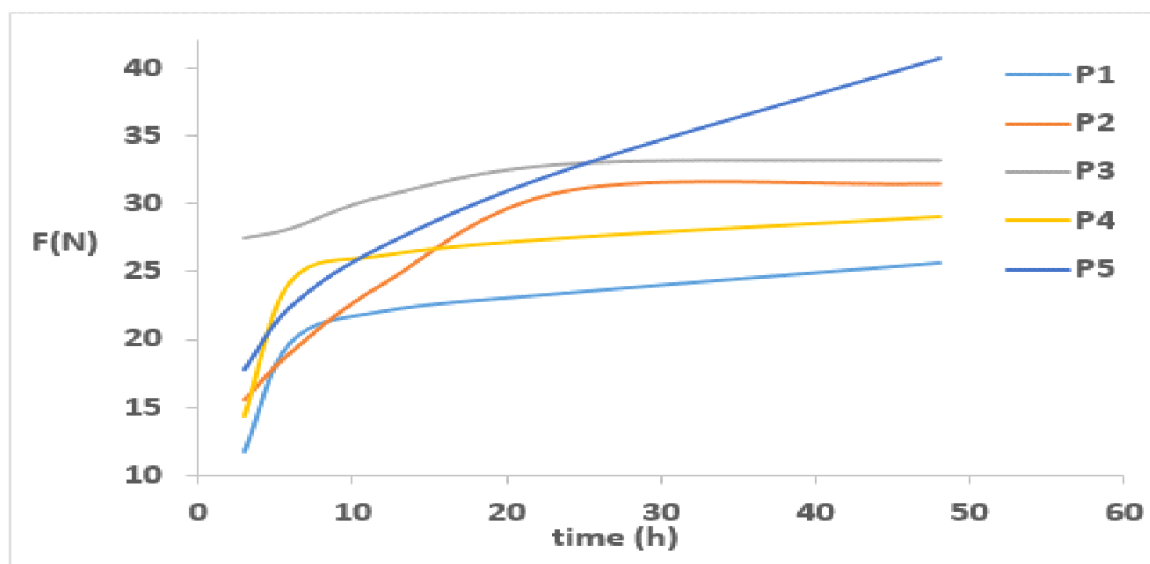


Figure (11): The adhesion strength of polymers (P1-P5).

Adhesion strength

The adhesion strength for the synthesized polymers was measured using single-lap shear method [19]. The values of adhesion strength for the synthesized polymers are listed.

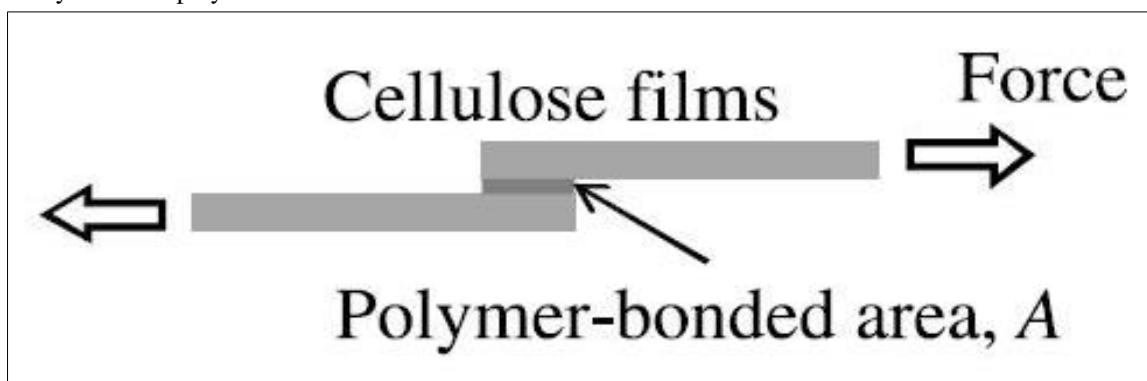


Figure (12): Single-lap shear testing.

Table (2): The values of adhesion strength for the synthesized polymers (P1-P5)

polymer No.	adhesion strength
P1	74.15 N.m ²
P2	116.6 N.m ²
P3	196.3 N.m ²
P4	159 N.m ²
P5	95.3 N.m ²

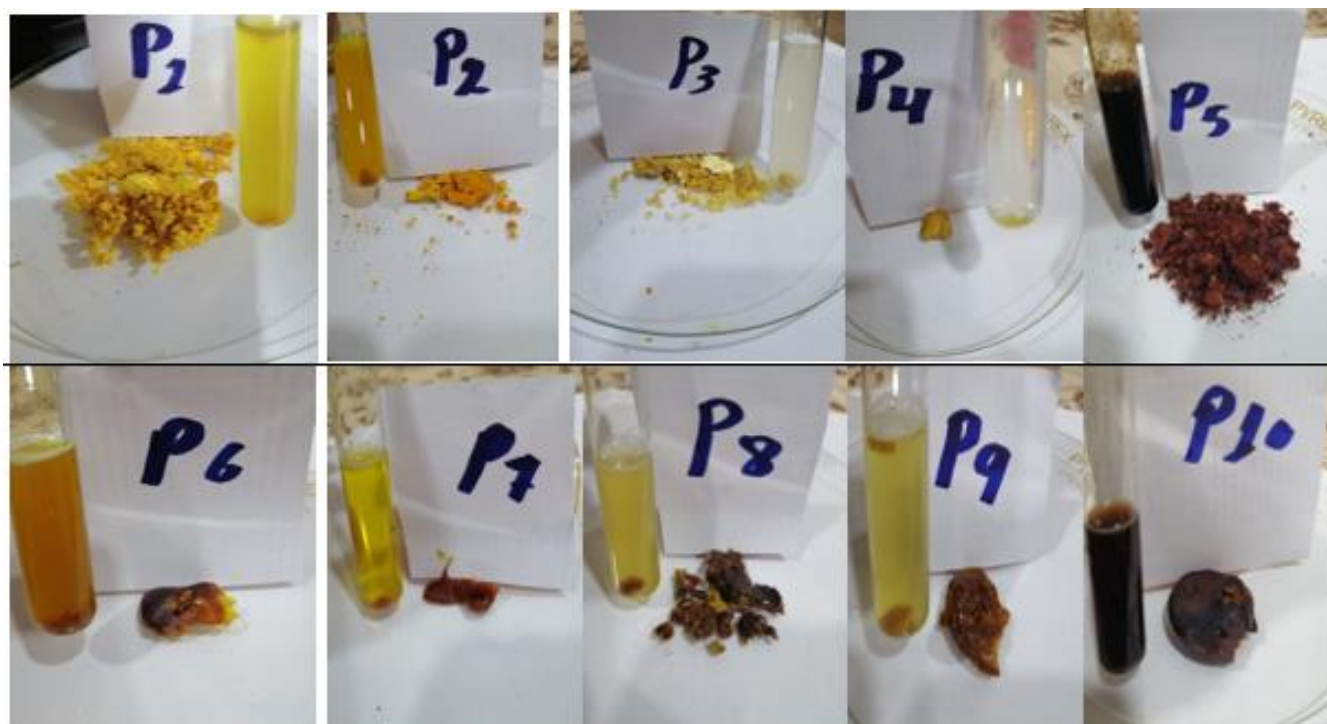
Solubility

The polymers exhibit different solubility against the used solvent [20].

Table (3): The solubility with different solvent

Polymer No	THF	DMF	DMSO	Benzene	acetone	methanol	ethanol	water	10%(w/v) KOH
P1	++	+	+++	++	+	–	–	–	+++
P2	++	+	+++	++	+	–	–	–	+++
P3	++	+	+++	+	++	–	–	–	+++
P4	++	+	+++	++	+	–	–	–	+++
P5	++	+	+++	++	+	–	–	–	+++

Highly soluble (+++), Moderate soluble (++) , low soluble (+) , In soluble (–).

**Figure (13):** Solubility of polymers.

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