Synthesis, Characterization, and Evaluation of Antioxidant and Physical Properties of New Bifunctional Aromatic Monomers Containing Imine and Heterocyclic Rings in The Main Chain.

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Abstract

New various monomers with di amine functional groups have been synthesized, containing di azomethine or di seven-membered heterocyclic rings in the backbone. A condensation reaction of benzidine with 4-amino benzaldehyde with some drops of glacial acetic acid in ethanol absolute prepared bi-functional amino groups monomer with azomethine core P1(4-[(1E)-[4[4-(E)-(4-aminophenyl)methylidene]amino]phenyl}phenylimino methyl aniline). The next step was the Cycloaddition reaction of P1 with succinic, malic, and phthalic anhydrides used to obtain aromatic heterocyclic monomers with oxazepane and oxazepine rings containing lactone and lactam groups. All the synthesized monomers were characterized spectrally with many techniques (FT.IR,1 H1.NMR, C13-NMR) that were used to investigate the functional groups in the structure of monomers. Monitoring the progress and completion of the reaction was performed using thin-layer chromatography (TLC). Physical characterizations, including detecting the melting point, purity, colour, retardation factor, molecular formula, and molecular weight, the polarized optical microscope (POM) used for Image-based microscopic interpretation of prepared compounds. All the prepared monomers are considered effective antioxidant agents as they give free radicals scavenging over 60%, reaching 80 %; this activity makes them useful in large numbers of industries as they can scavenge the free radicals that the industrial product may be exposed, which increases its shelf life and enhance its effectiveness.

Kye words

monomer, bi-functional, diamine, imine, amide, seven-membered ring, heterocyclic, antioxidant, oxazepane

Introduction

synthesis of new heterocyclic monomers has had good attention in recent years as they serve as repetitive units linked together to form the larger molecule of polymer[1]. Their industrial importance and application depend on their structure[2]. Aromatic–heterocyclic monomers are Organic compounds with versatile applications; polyamides are an essential type of polymer known for their stiffness, thermal and mechanical stability[3]. The aromatic diamines are used as monomers in polyamide prepearation[4]. Schiff bases are a chemical compound category that contains an imine group (–CH═N–) in their structure. These materials are the initial components for creating a wide range of heterocyclic compounds[6,7.] They are widely used with broad applications such as [(8-12)] Anticancer, Antimicrobial, Anti-corrosion, antibacterial, Antioxidant, Antifungal…etc. Monomers are distinguished from other organic compounds in their containing bi-functional groups instead of one. This leads to the formation of linear polymers[13,14], while if they include more than two functional groups, it will lead to the formation of a branched polymer, and each of the polymers has its applications and uses[15,16]. Oxazepanes are Heterocyclic compounds with a seven-ring ring[17] that contain lactone(O-C=O) and lactam (HN-C=O)[18]. Diamine monomers containing imine groups and heterocyclic rings have comprehensive and valuable applications in different fields [19]. The antioxidant property is a very important characteristic for scavenging free radicals that can enter the body and to which the industrial product may be exposed, whether it is a pharmaceutical, a food, or an industrial product[20].

Materials and Methods:

Materials

Fluka and Merck's company supplied all chemicals with the highest purity. The melting point measurements were measured by electro-thermal 9300 melting point engineering- LTD, U.K. FT-IR spectra were measured by Fourier transform infrared Shimadzu (8400) using potassium bromide pellets (whereby the values are expressed in units of cm-1), Silica gel was used to perform Thin Layer Chromatography (TLC). .H^1-NMR & C^{13}-NMR spectra were obtained using DMSO-d6 as the solvent, and the units of measurement used were ppm using (Bruker-Ultra Shield 300 MHz Switzerland)-Tehran university-Iran, the physical and using polarized optical microscope (POM) was at chemistry department- faculty of education-university of Kufa- (Iraq). The antioxidant study was made by microplate reader ELISA, from the

paramedical company, made in Italy, and presented at Al-Amin Center for Advanced Biotechnology and Research- Najaf – Iraq.

Methods

1- Synthesis of Schiff base monomer compound (p_1) [21,22]

4-[(1E)-[4[4-(E)-(4-aminophenyl)methylidene]amino]phenyl}phenylimino methyl aniline

Weighting (0.002mol, 0.242 g) from 4-amino benzaldehyde was solved in 20 ml of absolute ethanol. Then, you need to add 2-3 drops of glacial acetic acid. (0.001mol, 0.184 g) from (benzidine) 4-(4-aminophenyl) aniline that was solute in ethanol; this was added to the first solution. The mixture was stirred thoroughly and then subjected to reflux for 15 hours. The progress and completion of the reaction were monitored using the (TLC) by using dry benzene: Ethanol absolute (2:3). As shown in Figure 1, The solid particles were gathered and filtered. The coloring solid crystals were obtained and then purified using a recrystallization process using ethanol absolute.

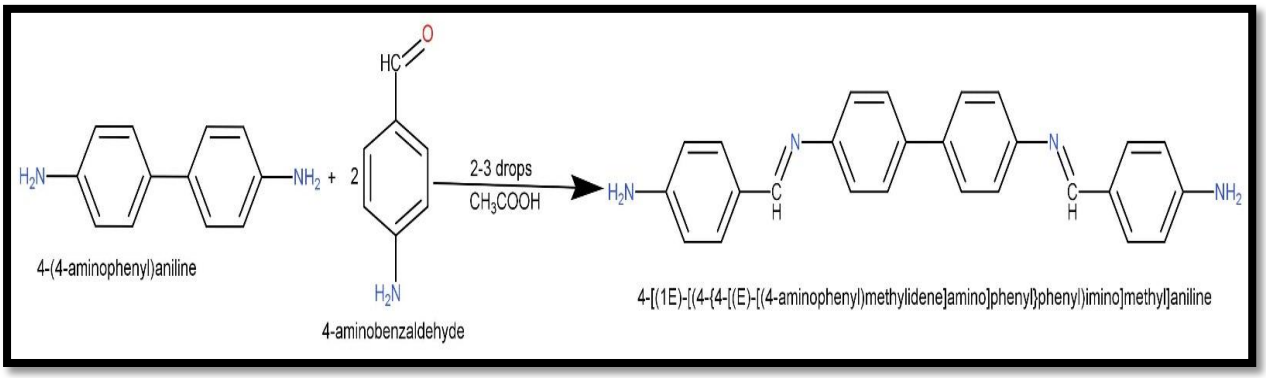


Figure 1: Synthesis reaction of Schiff base monomer P1

2- Synthesis of 2-(4-aminophenyl)-3-(4-(4(2-(4-aminoohenyl)-4,7-dioxo-1,3-oxazepan-3-yl)-1,3-oxazepepane-4,7dione heterocyclic monomer (P1C1)[23,24]

P1C1 was synthesized by mixing (0.001mole, 0.39g) of Schiff base compound P1 with (0.002mole, 0.201 g) of succinic anhydride after good solubility of each one in (30ml) dry benzene. The mixture of reactants was vigorously stirred together.at (55°C) for (10-13 hr.). The coloring solid crystals were recrystallized using dry 1,4-dioxin. The TLC experiment demonstrated the completion of the chemical reaction using (dry benzene: absolute Ethanol) on the ratio of (1:4). As noted in Figure 2.

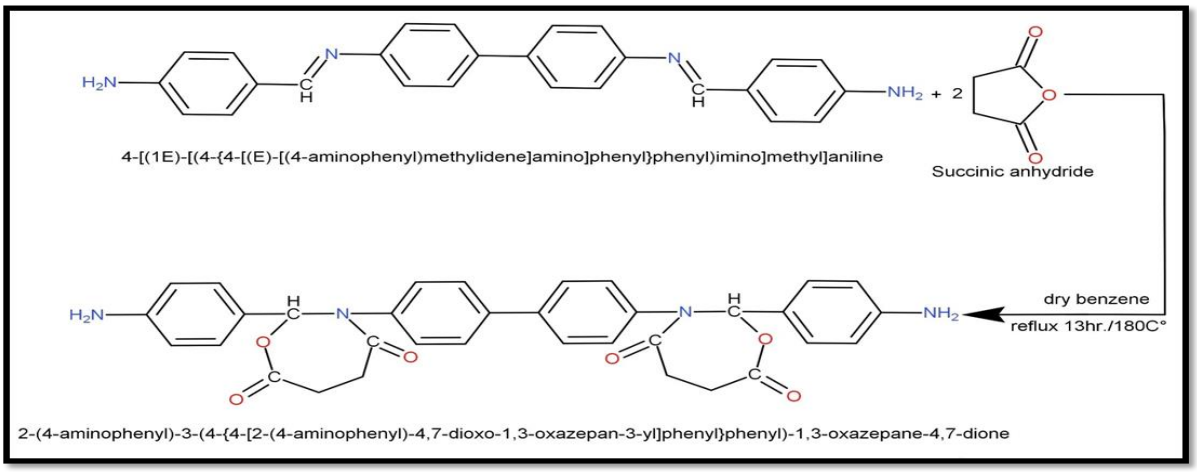


Figure 2: Synthesis reaction of P1C1 heterocyclic monomer

3- Synthesis of 3-(aminophenyl-4)-4(4-(3-(4-aminophnyl)-1-5- dioxo-1,3,4,5tetrahydro-2,4-benzoxazpin-4-ylphenyl)-1,3,4,5tetrahydro-2-4-benzoxazepine-1,5-dione heterocyclic monomer (P1C2)

The same procedure above was followed by using (0.002mole,0.3682 g) of phthalic anhydride, as shown in Figure 3 below.

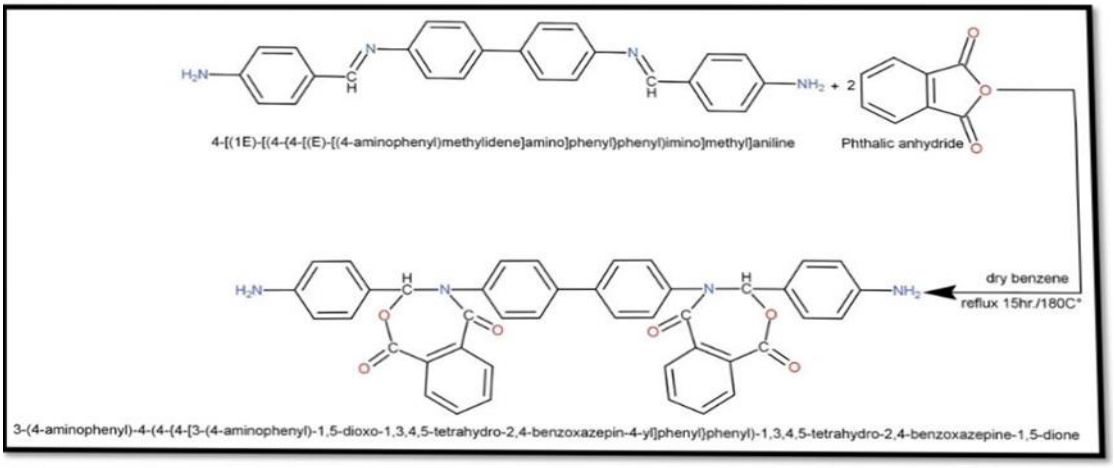


Figure 3: Synthesis reaction of P1C2 heterocyclic monomer

4- Synthesis of 2-(4-aminophebyl)-3-(4-(4-(2-(4-aminopenyl)-4,7- dioxi-2,3,4,7-tetrahydro-1,3-oxazepin-3-yl)phenyl)phenyl-2,3,4,7-tetrahdro-4,7-dione heterocyclic monomer (P1C3)

The fourth heterocyclic monomer was prepared using (0.002mole,0.1691 g) of Maleic anhydride to produce P1C3, as shown in Figure 4 below.

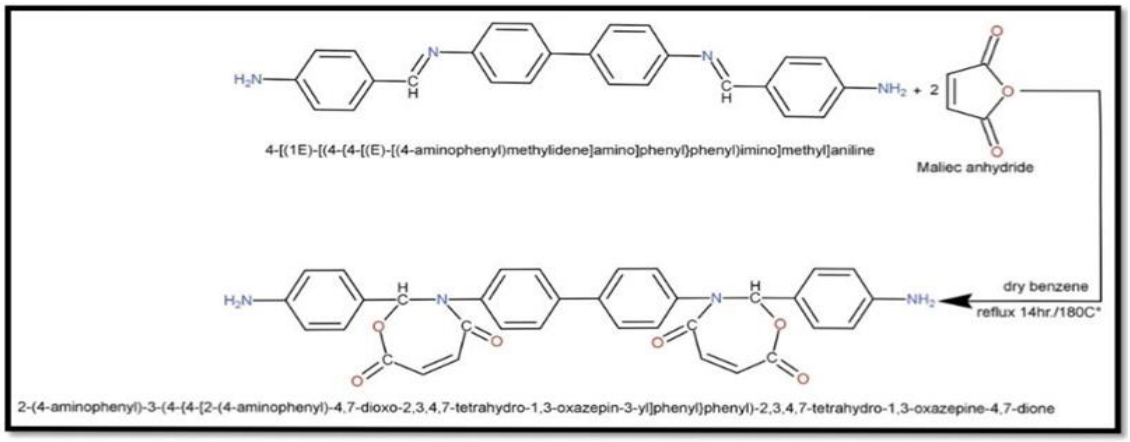


Figure 4: Synthesis reaction of P1C3 heterocyclic monomer

Antioxidant Evaluation study

To evaluate the free radical-scavenging effect, the modified DPPH radical cation method was utilized. A solution of 8 mg of this reagent dissolved in 100 mL of MeOH was prepared to obtain a concentration of 80 μL/mL. In a 96-well microplate, 100 μL of DPPH was mixed with 100 μL of the sample and then incubated at room temperature for 30 minutes. Afterwards, the absorbance was measured at 514 nm using an ELISA reader with pure methanol as a control. The DPPH scavenging effect was calculated using the following formula:

$$\textbf{Radical scavenging (\%)} = [(A)\textbf{control} - (A)\textbf{sample}] / (A)\textbf{control} \times 100$$

Extrapolated IC50 values were obtained from regression analysis of DPPH inhibition. Antioxidant activity was evaluated based on the IC50[26].

Result and Discussion

In the preparation of monomers, p-amino benzaldehyde was used twice as much as benzidine, as benzidine has two functional groups. In contrast, the other compound has only one, so we double its molarity to obtain monomers with bi-functional groups. The FT-IR spectroscopy was recorded as KBr pellets in the 650- 4000 cm^{-1} range and was used to confirm the chemical structures of produced monomers. The FT-IR spectrum of monomer P1 showed two absorption bands at 3423 cm^{-1} and 3323 cm^{-1} belonging to stretching frequencies of (N-H), a symmetrical and symmetrical, respectively, with a absorption band at 3315-3310 cm^{-1} belonging to aromatic (C-H) vibration. Also, a strong absorption band at 1639 cm^{-1} attributed to (C=NH) confirms the azomethine formation of the Schiff base monomer[27]. The (C=C) aromatic

appears at 1602 cm^{-1} while the (C-N) vibration absorption band appears at 1350 cm^{-1}, as noted in Figure 5.

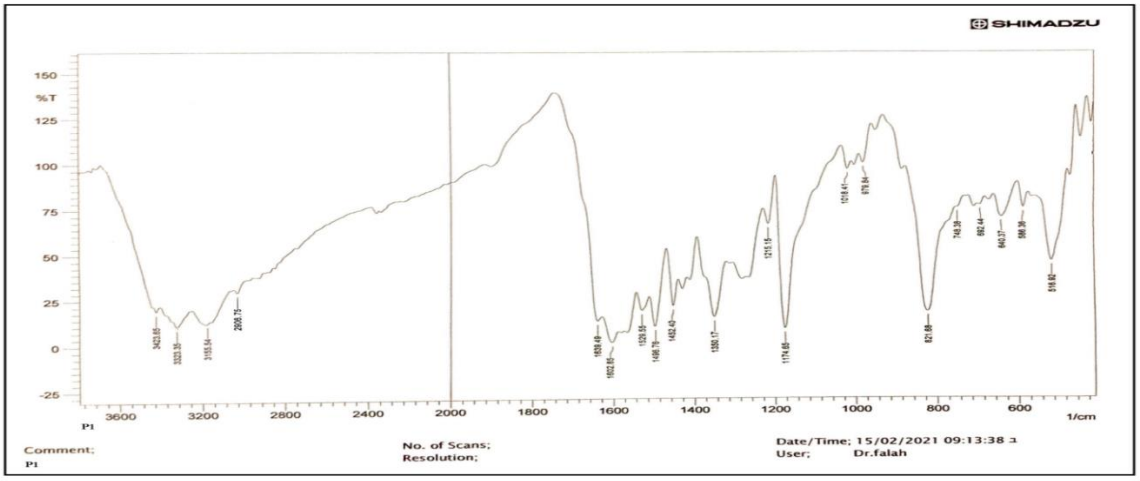


Figure 5: FT-IR of Schiff base monomer P1.

The FT-IR of P1C1 showed the disappearance of the imine absorption band at 1639 cm^{-1} and the appearance of new absorption bands at 1701 cm^{-1} and another at 1670 cm^{-1} referring to lactone(O-C=O) and lactam (N-C=O)formation of oxazepane monomer[28]. As oxazepane is a seven-membered heterocyclic compound with an aliphatic part, we notice an absorption band at 2933 $cm^{-}1$ belongs to aliphatic (C-H) in addition to an aromatic (=C-H) at 3040 cm^{-1} with a (C=C) vibration at 1325 cm^{-1} and remaining of two (N-H) vibrations as seen in Figure 6.

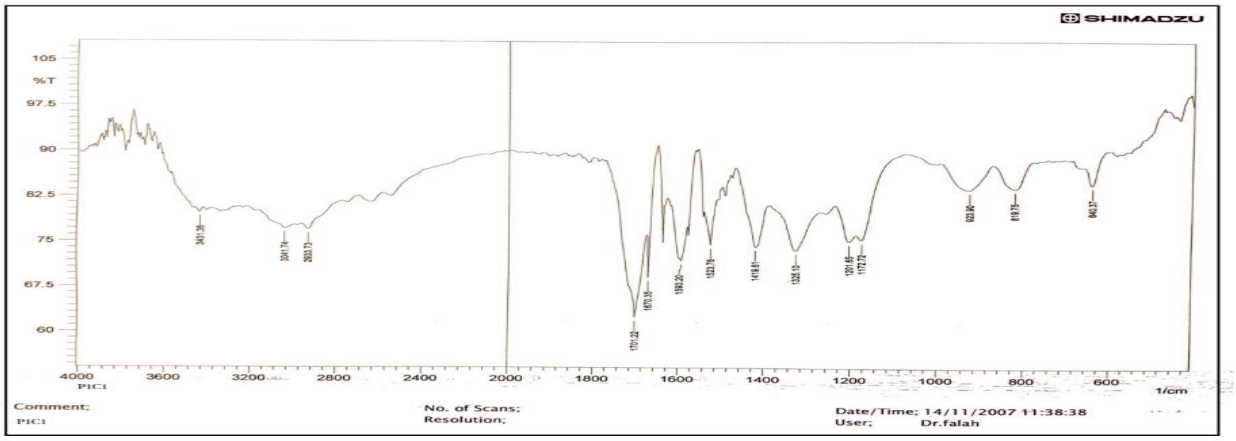


Figure 6: FT.IR of monomer P1C1.

The P1C2 spectrum showed the following absorption bands: (3437-3403) cm^{-1} for (N-H) vibrations, 3041cm^{-1} for the aromatic (=C-H) absorption band, and 2906 cm^{-1} for the aliphatic (C-H) absorption band. Like P1C1, we noticed the disappearance of the (H-N=C) absorption band of azomethine and the appearance of lactone and lactam absorption bands in 1701 and 1670 $cm^{-1,}$ respectively, (C=C) at 1593cm^{-1} and 1352cm^{-1} for (C-N). as noticed in Figure 7.

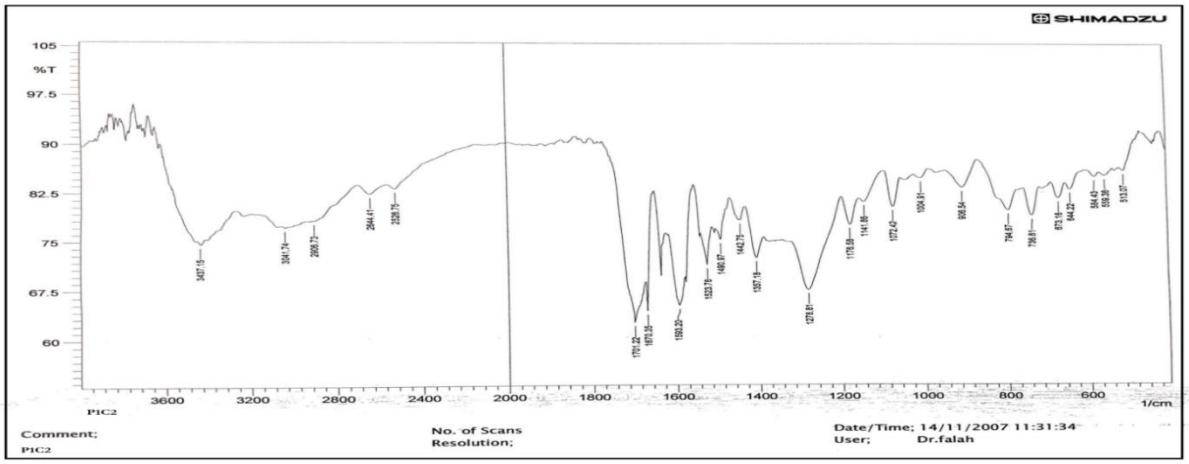


Figure 7: FT.IR of monomer P1C2.

The P1C3 also shows lactone formation at 1707 cm and lactam bonds at 1670 cm, with the Schiff base absorption band disappearing as evidence of oxazepane cycle formation. With 3107cm^{-1} 2906 cm^{-1} for aromatic and aliphatic(C-H) absorption band. 1577 cm^{-1} and 1176 cm^{-1} for (C=C) and (C-N). as below.

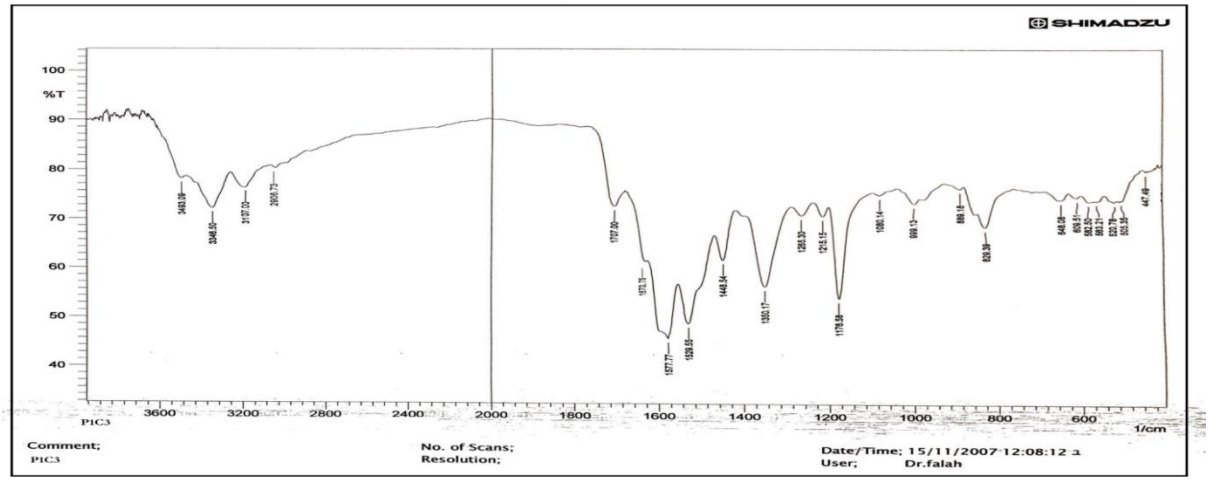


Figure 8: FT.IR of monomer P1C3.

H^1-NMR spectroscopy is used to identify the structure of molecules. As we see in Figure 9, the chemical shifts (8.5 ppm) were caused by the azomethine proton (CH=N) of Schiff base monomer P1, and the protons of aromatic rings were assigned at (7-7.9) ppm. Amine signal showed at 6.4 ppm and 2.5 ppm related to the solvent[29]. Figures 10-12 show the H^1-NMR Spectra of P1C1, P1C2, and P1C3 and give the signals 1.2 ppm and 1.7 ppm related to methylene groups in heterocyclic rings—a single signal at 8.9 ppm related to(C-H) between lactone and lactam in oxazepane. Phenyl rings give multiple signals (7-7.9) ppm[30].

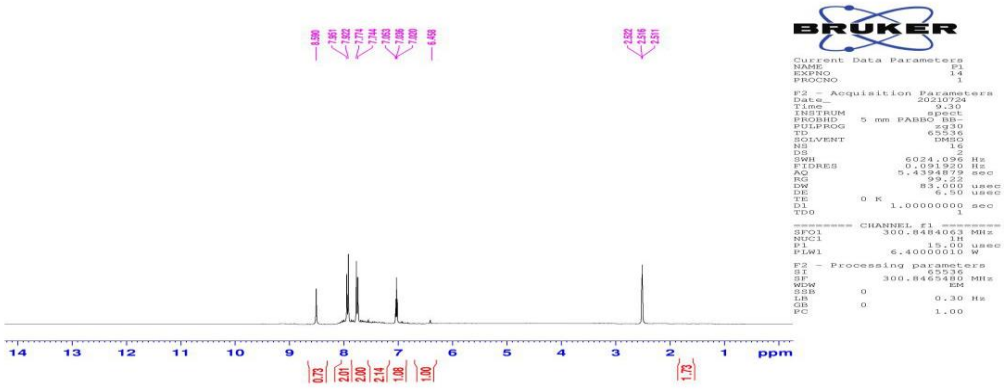


Figure 9: H^1-NMR of Schiff base monomer P1.

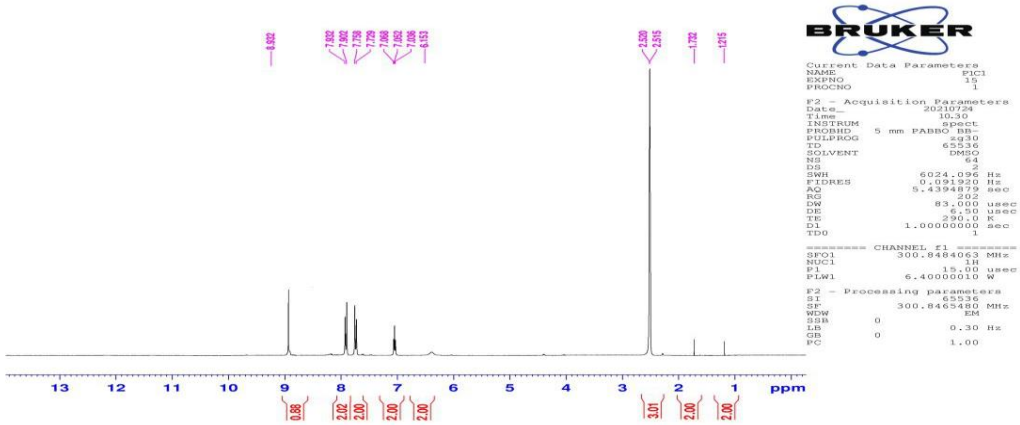


Figure 10: H^1-NMR of P1C1 monomer.

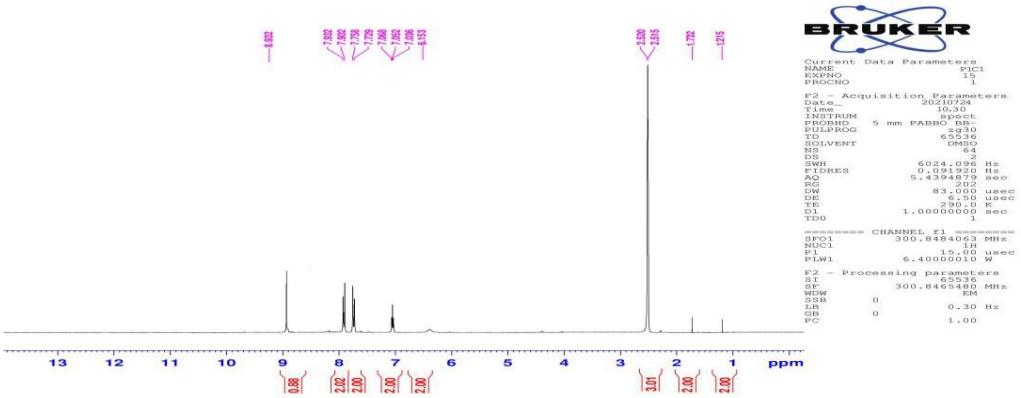


Figure 11: H^1-NMR of P1C2 monomer.

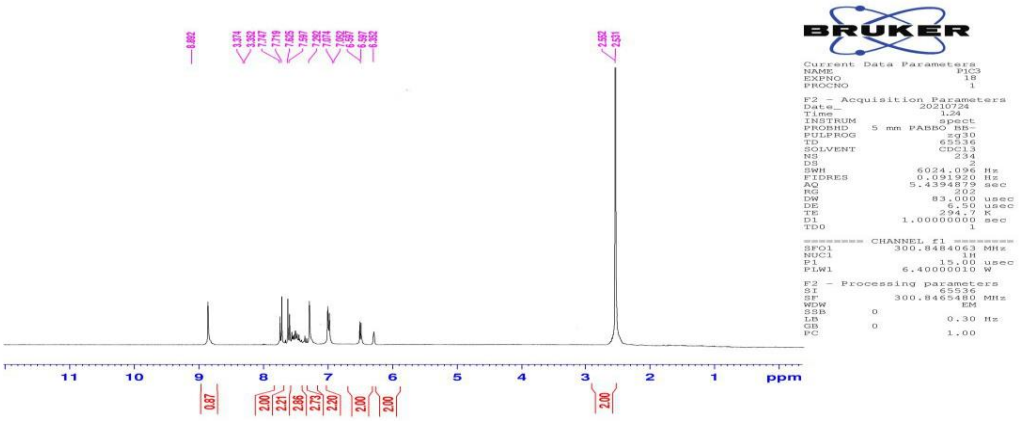


Figure 12: H^1-NMR of P1C3 monomer.

C^{13}-NMR Spectra, carbon-13 applies nuclear magnetic resonance (NMR) spectroscopy to carbon, essential for chemical structure determination[31]. C^{13}-NMR Spectra of the synthesized monomers were recorded. They provided chemical shifts in the range of (160 ppm) for the carbon of azomethine in Schiff base monomer, (117-131) ppm related to the phenyl ring, and 40 ppm for the solvent, as seen in Figure 13. For heterocyclic monomers, we notice signal at 160 ppm refer to the ring enclosing, with signals at 174ppm and 176 ppm related to lactam and lactone, respectively; the phenyl aromatic rings appear as multiple signals in the range of (112-130) ppm[32].

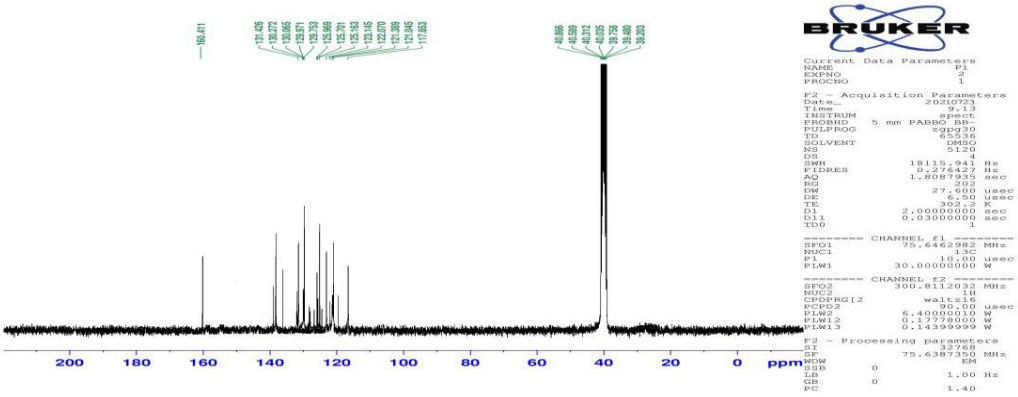


Figure 13: C^{13}-NMR of P1 monomer.

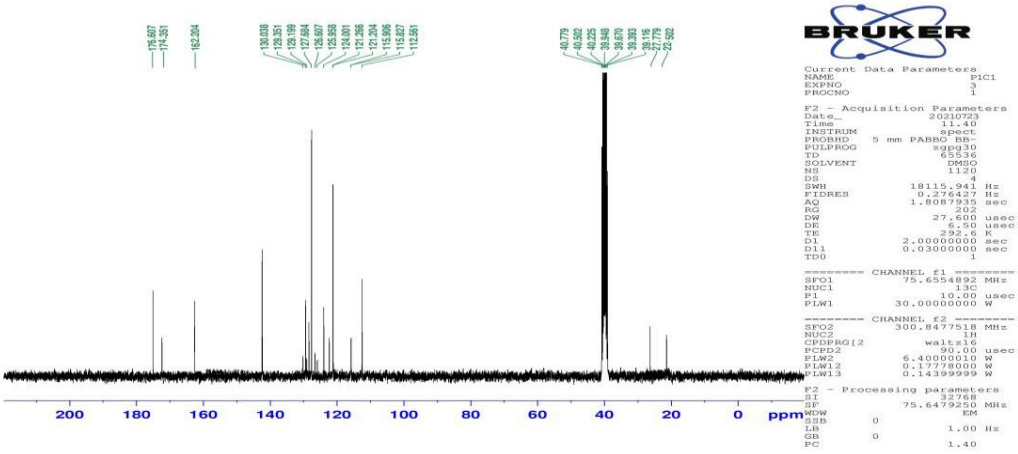


Figure 14: C^{13}-NMR of P1C1 monomer.

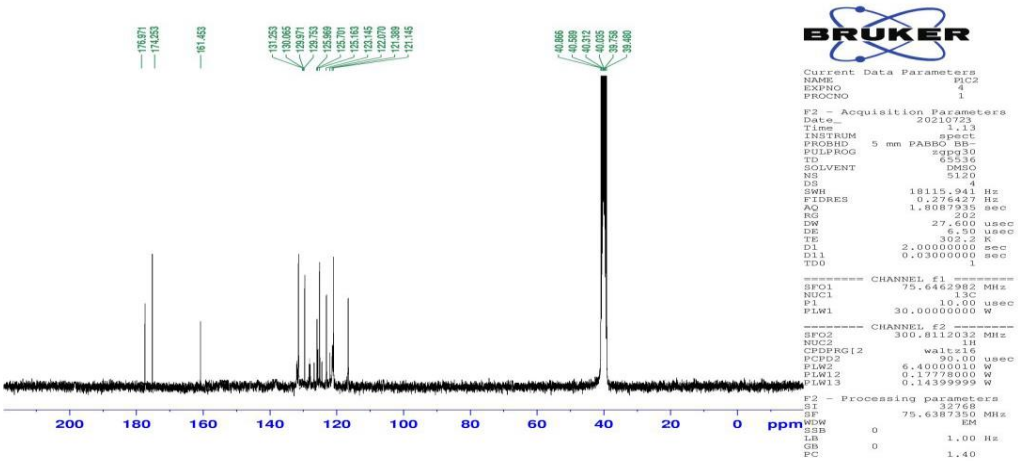


Figure 15: C^{13}-NMR of P1C2 monomer.

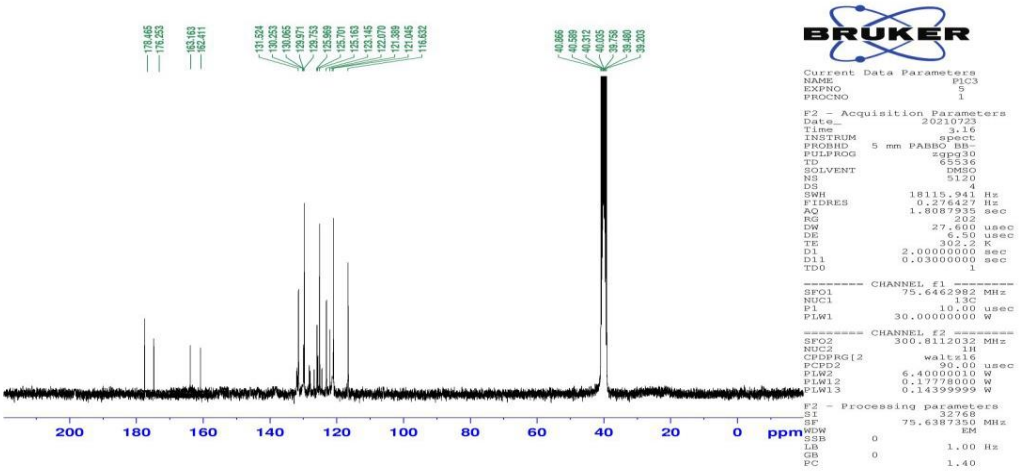
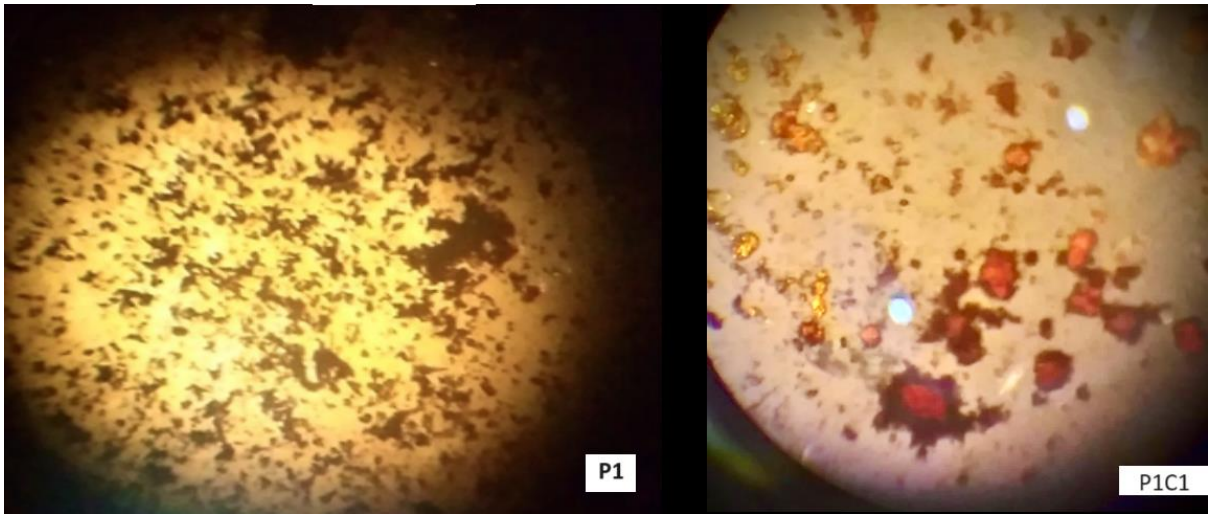
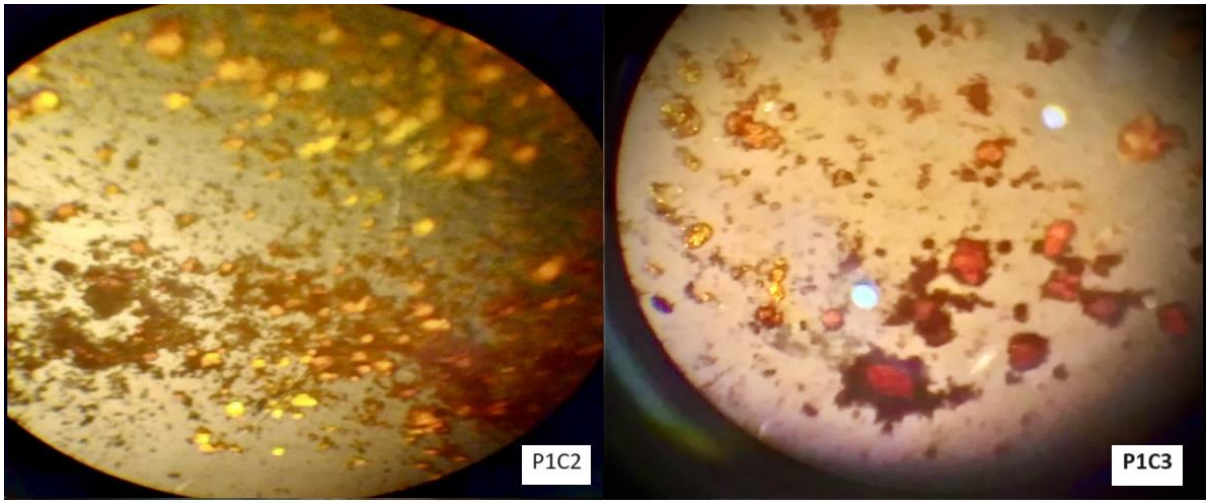


Figure 16: C^{13}-NMR of P1C3 monomer.

Physical characterization

Table 1 summarizes the investigated physical properties, including melting point (MP), purity, color, retardation factor (RF), molecular formula (MF), and molecular weight (M. WT). We observe an increase in melting point with increasing molecular weight. The monomers prepared exhibit high melting points, indicating good thermal stability and narrow range, which implies good purity[33,34]. The polarized optical microscope (POM)was used for Image-based microscopic interpretation for all the prepared monomers at room temperature[35], and the following images were obtained.





Pic. 1: POM of the prepared monomers.

Table 1: the physical properties of all the synthesized monomers

Sample no.	M.P.	M.F.	M. wt.	Color	Rf	Yield %
P_1	155-156	$N_4 H_{22} C_{2.}$	390	Red-brown	0.9	82.35
P1C1	163-164	$C_{33}H_{26}O_6$	574	light brown	0.93	68.66
P1C2	169-170	$C_{24}H_{30}N_4O_6$	686	umber	0.98	69.25
P1C3	165-166	$C_{34}H_{26}N_4O_6$	586	Yellow-brown	0.98	70.55

Antioxidant activity evolution

The compound has an antioxidant property that makes it important in many applications, especially medical and pharmaceutical applications and in the field of drug delivery[36,37]. When a compound gives more than 50% inhibition of free radicals, it is considered a good antioxidant agent[38]. The prepared monomers showed antioxidant activity between (62-80) %, even in low concentrations, as shown in the figures below.

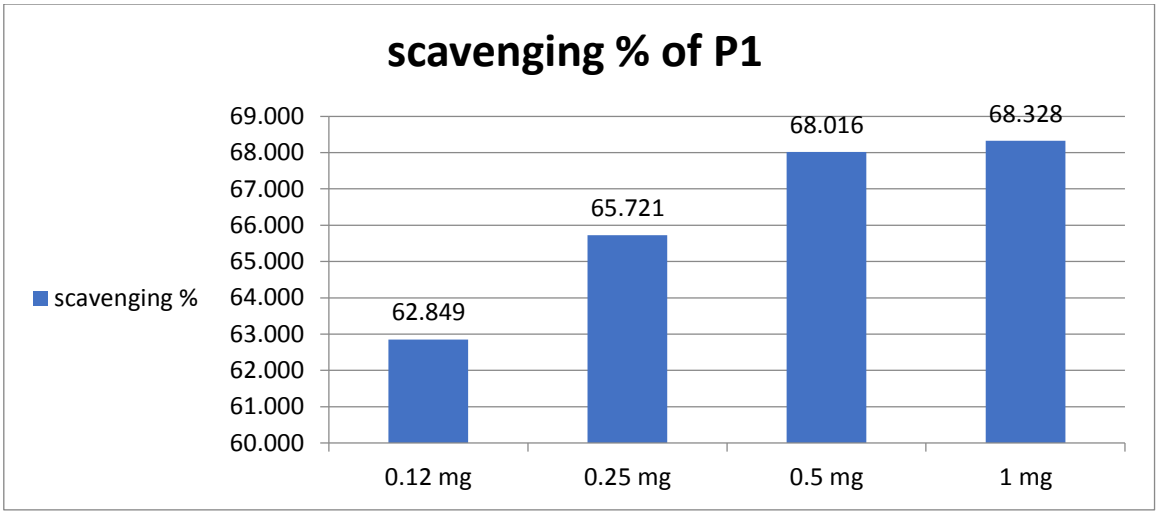


Figure 17: free radical scavenging of P1 monomer.

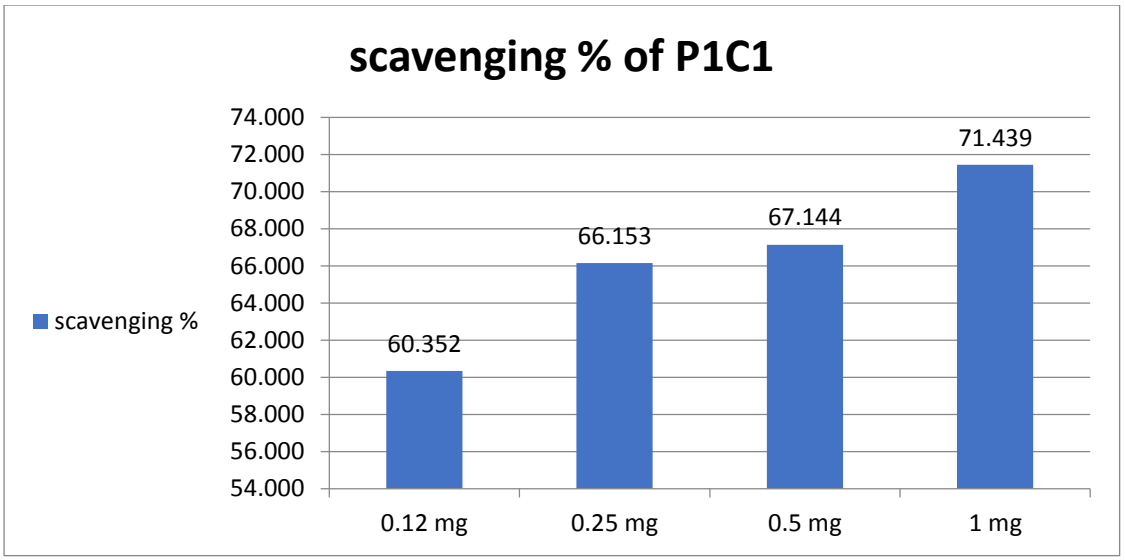


Figure 18: free radical scavenging of P1C1 monomer.

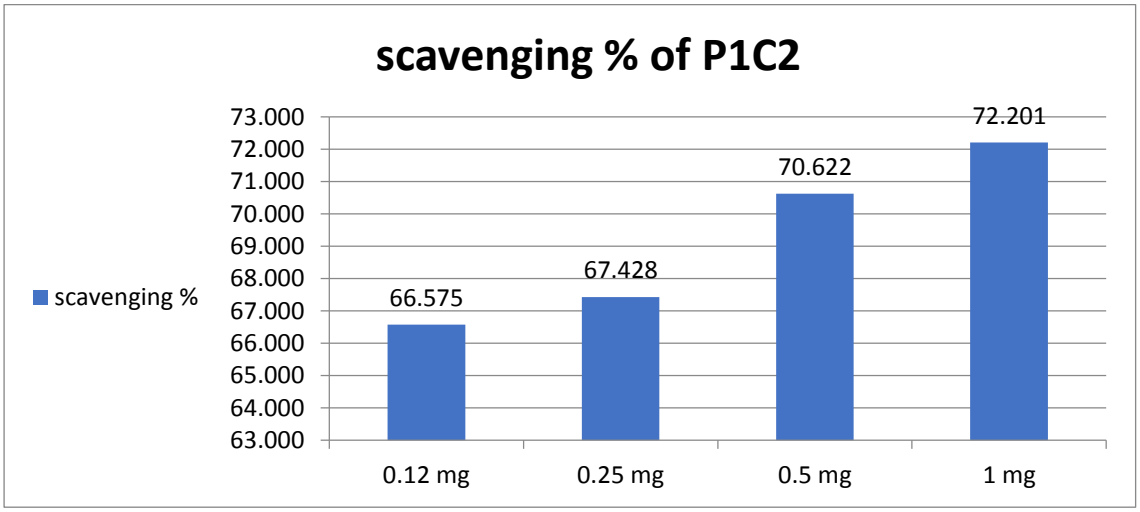


Figure 19: free radical scavenging of P1C2 monomer.

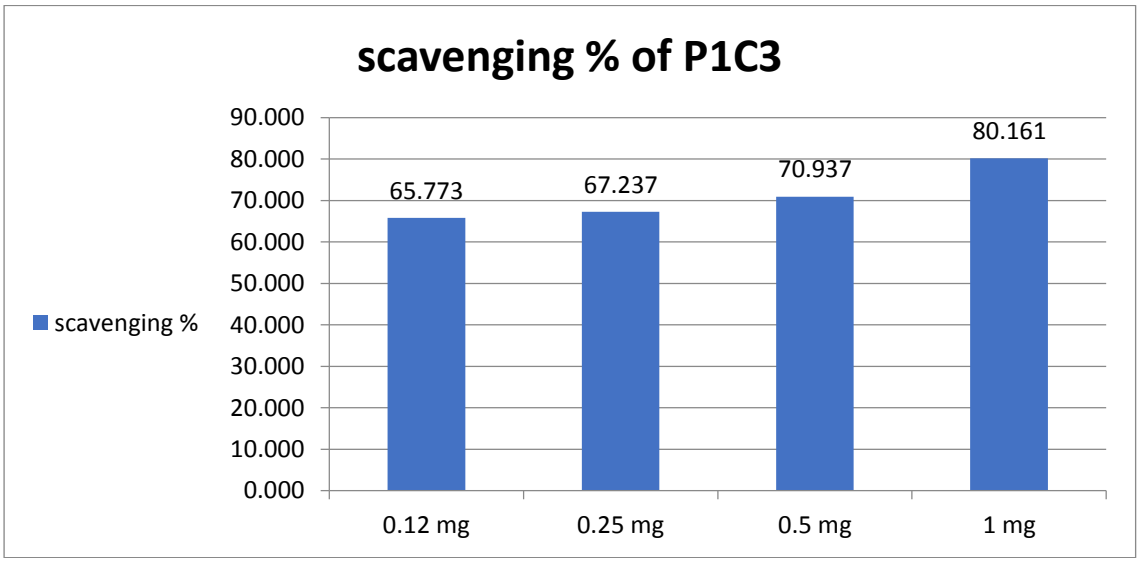
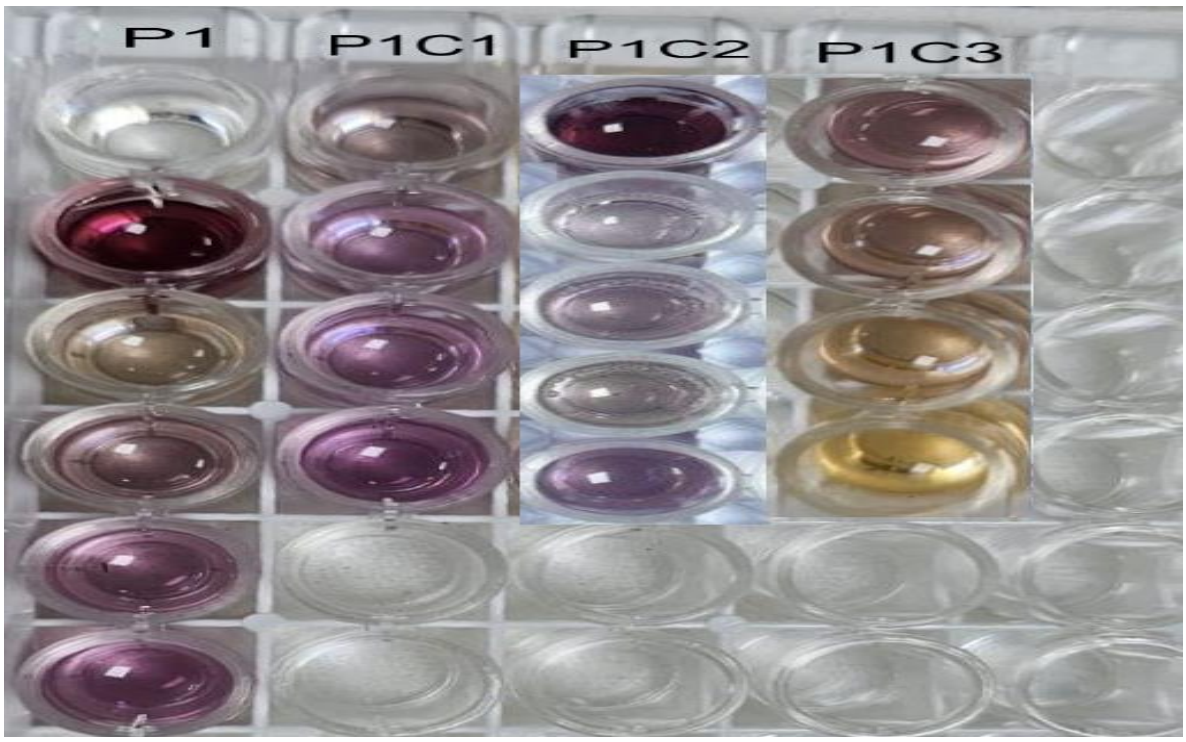


Figure 20: free radical scavenging of P1C3 monomer.

A compound with antioxidant properties increases its effectiveness if it loads and delivers drugs[38]. the antioxidant test is show below.



Pic. 2: antioxidant evaluation of the prepared monomers.

The first colorless circle refers to methanol blank; the dark red belongs to the dpph reagent before adding the compounds. The lighter circles refer to the free radicals' scavengers' effect of increasing concentrations of the prepared compounds; we notice the color becomes yellow and lighter as the concentration increase, the greater its effectiveness in scavenging free radicals and thus its greater effect as an antioxidant[39].

Conclusions

This research described the successful synthesis of various new monomers with imine and seven-membered heterocyclic rings in the leading chains in good yield and purity. FTIR, H1, and C13-NMR spectroscopy were used to detect the functional groups in the structure of the new monomers spectrally. All prepared monomers give good physical and good antioxidant activity, making them a good choice for preparing different polymers useful in various industries.

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