A STUDY OF NEWLY SYNTHESIZED BIS SCHIFF-BASE COMPOUNDS DERIVED FROM THE CONDENSATION OF 4,4'-(1-(9H-FLUOREN-2-YL)-2,2,2-TRIFLUOROETHANE-1,1-DIYL) BIS (2-METHYLANILINE) AND AROMATIC ALDEHYDE AND THEIR ANTIOXIDANTS

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Abstract: *New fluorene Schiff-base derivatives were synthesized from condensation of (1:2) diamine 4,4'-(1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl) bis (2-methylaniline) with different aromatic aldehydes such as (2,5-dihydroxybenzaldehyde, salicyldehyde, 5-nitrosalicyldehyde, 4-benzloxybenzaldehyde and indole-3-carboxyldehyde), glacial acetic acid was used in the reaction as a solvent and gave good yield in the range of 82-89%, the end of the reaction was indicated by Thin Layer Chromatography. The synthesized compounds were characterized using FTIR,* ^{1}*HNMR,* ^{13}*CNMR and Mass spectrometry. The physical properties of the synthesized compounds such as melting point and color were studied. The compounds [N1-N5] exhibited a strong scavenging ability to expel 2,2-diphenyl-1-picrylhydrazyl between 77.255 and 85.935 at high concentration of 1mg per ml, thus indicating that these compounds can be used as good antioxidant agents.*

Keywords*: Schiff-base, fluorene compounds, aromatic aldehydes, anti-oxidant.*
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Introduction

Fluorene derivatives have been in great use because of their different applications such as in optoelectronics [1] semiconductors [2], biological activities [3], solar cells [4] and fuel cells [5].

Schiff base compounds are produced from condensation reactions between aldehyde and ketone with primary amine. The use of an aromatic aldehyde and ketone results in the formation of more stable compounds. Further aldehyde is more stable than ketone in the synthesis of Schiff compound derivatives due to its steric hindered and electronic effect [6-9]. This reaction can be facilitated using acid or base catalysis without use any catalyzer. The azomethine group (R2C=N-R) is recognized the group for Schiff compounds [10-12]. This is considered a starting material and an intermediate for several reactions, such as those involving the Mannich base [13]. It is also used for complex reactions with metals [14]. Metal Schiff base complexes, especially that those containing iron (II), Ruthenium (II) and copper (II) are used in catalysis of oxidation reactions of alcohol compounds, hydrolysis of esters of amino acid compounds and cyclopropanation reactions to produce five, six seven-membered heterocyclic compounds [15-19] that have pharmacological properties [20]. These compounds have wide biological applications [21] such as antimalaria [22], antiprotozoal [23] antitumor [24] antifungal [25], and antioxidant properties [26, 27]. Their ability to stimulate biological activity is a result the hydrogen bonds formed between the nitrogen of Schiff base compounds and the active cell center [28, 29]. There are also used as photo-stabilizers of polymers [30], catalysts for organic reactions, and inhibitor of corrosion [31]. The antioxidant property is used to slow or prevent cell damage caused by free radicals [32]. Production of reactive oxygen species ROS increases with time causing several diseases affecting the human body. Schiff-base compounds play a role in the elimination of ROS because of their anti-oxidant properties [33].

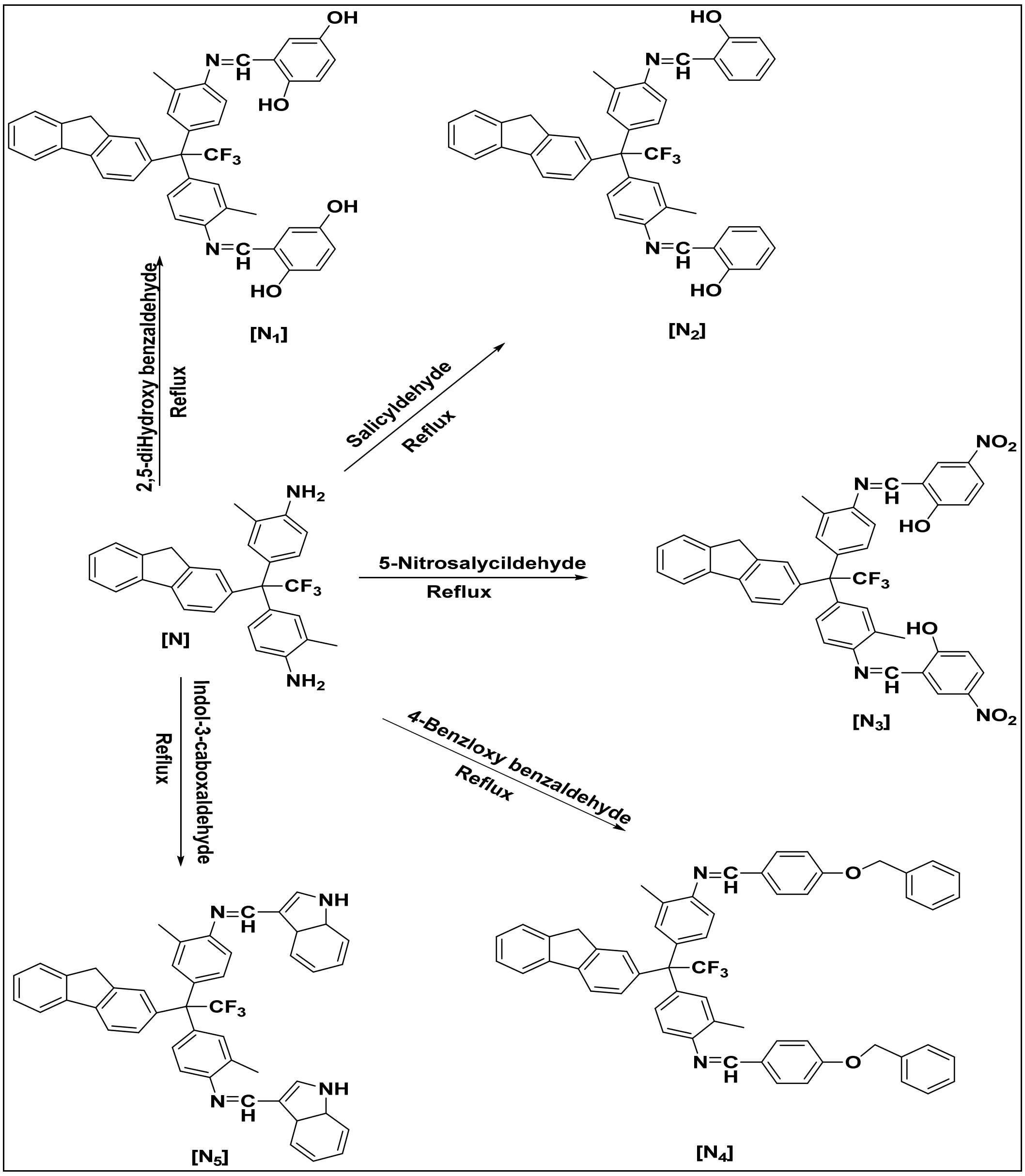
In this paper, novel Schiff base derivatives were synthesized through condensation of diamine derivatives with different aldehydes. These compounds were structurally validated using FTIR, Mass spectroscopy, ^{1}HNMR and ^{13}CNMR.

Experimental part

Two companies BDH and Merck supplied all the chemicals used in this study.

Shimadzu's (FT-IR 8400Series Japan) with a range of 4000-400 cm^{-1}; the ^{1}H and ^{13}C-NMR data were recorded using Bruker's (AC 400) NMR using a 400 MHz spectrometer; Mass spectroscopy 3200 Qtrap, at Ferdowsi University of Mashhad.

General procedure. 0.5 g of 0.001 mole 4,4'-(1-(9H-fluorene-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methylaniline) [N] was dissolved in absolute ethanol, followed by the addition aromatic aldehydes (0.002 mole, 0.301g, 0.266 g, 0.364 g, 0.462 g and 0.316g). Then a few drops of glacial CH_3COOH were added and reflux was observed for 8-12 hours. After the solvent evaporated, the final product was recrystallized from ethanol (Scheme 1).



Scheme 1. Steps of synthesis of [N1-N5] compounds

2,2'-((((1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneyl ylidene))bis(methaneylylidene))bis(benzene-1,4-diol) [N1]: (0.001 mole, 0.5 g) [N] with (0.002 mole, 0.301 g) 2,5-dihydroxy benzaldehyde was reacted using the general method.

FT-IR (KBr, cm^{-1}): 3178(O-H), 3057(C-H$_{ar.}$), 2912 (C-H$_{alph.}$), 1645 (C=N), 1581 (C=C), (C-O), (C-F); ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 163.73 (C, CH=N), 6.83-7.36 (m,13H, CH$_{arm.}$), 2.27 (s, H, CH$_{aliph.}$); ^{13}C-NMR (100 MHz, DMSO, δ, ppm): 163.73 (C, CH=N), 112.75-150.04 (m, 22C,C$_{arm}$), 18.17 (2C, C$_{aliph.}$). MS (EI, m/z): calculated, 698.74; found [M+H], 699.72; [M+K] 737.28.

2,2'-((((1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaneyl ylidene))bis(methaneylylidene))diphenol [N2]: (0.001 mole, 0.5 g) [N] with (0.002 mole, 0.266 g) Salicylaldehyde was reacted using the general method.

FT-IR (KBr, cm^{-1}): 3367 (O-H), 3063(C-H$_{ar.}$), 2980 (C-H$_{alph.}$), 1664(C=N), 1645(C=C), (C-O), (C-F); ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 8.92 (s,H, Ar-CH=N-) , 6.77-7.90 (m,H, Ar-H), 18.17 (S ,H, CH$_{aliph.}$); ^{13}C-NMR (100 MHz, DMSO, δ, ppm): 163.93 (C, Ar-C=N-), 116.66-160.43 (C,C$_{Ar}$), 18.23 (C,C$_{aliph.}$); MS (EI, m/z): calculated, 666.74; found 667.25[M+H].

2,2'-((((1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(azaney lylidene))bis(methaneylylidene))bis(4-nitrophenol) [N3]: (0.001 mole, 0.5 g) [N] with (0.002 mole, 0.364 g) 5-nitrosalicylaldehyde was reacted using the general method.

FTIR (KBr, cm^{-1}): 3404 (O-H), 3078(C-H$_{ar.}$), 2980 (C-H$_{alph.}$), 1620(C=N), 1600(C=C), (C-O), (C-F); ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 9.12 (s,H, Ar-CH=N-) , 6.77-7.90 (m,H, Ar-H), 2.33 (s, H, CH$_{aliph.}$); ^{13}C-NMR (100 MHz, DMSO, δ, ppm): 166.88 (C, Ar-C=N-), 118.53-146.18 (C,C$_{Ar}$), 18.16 (C, C$_{aliph.}$) ; MS (EI, m/z): calculated, 756.74; 755.24 [M-H].

N,N'-((1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(4-(benzyloxy)phenyl)methanimine) [N4]: (0.001 mole, 0.5 g) [N] with (0.002 mole, 0.462 g) 4-Benzloxy benzaldehyde was reacted using the general method.

FTIR (KBr, cm^{-1}): 3034(C-H$_{ar.}$), 2943 (C-H$_{alph.}$), 1689(C=N), 1626(C=C), (C-O-C), (C-F) ; ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 8.49 (s,H, Ar-CH=N-) , 5.17-7.93 (m,H, Ar-H), 2.38 (S ,H, CH$_{aliph.}$); ^{13}C-NMR (100 MHz, DMSO, δ, ppm): 163.32 (C, Ar-C=N-), 115-143.57 (C,C$_{Ar}$), 17.71 (C,C$_{aliph.}$); MS (EI, m/z): calculated, 846.99; 847.17 [M+H].

N,N'-((1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methyl-4,1-phenylene))bis(1-(2,7a-dihydro-1H-indol-3-yl)methanimine) [N5]: (0.001 mole, 0.5 g) [N] with (0.002 mole, 0.316 g) Indol-3-carboxaldehyde was reacted using the general method.

FTIR (KBr, cm^{-1}): 3394(N-H), (C-H$_{ar.}$), 2974 (C-H$_{alph.}$), (C=N), (C=C), (C-F); ^{1}H-NMR (400 MHz, DMSO, δ, ppm): 9.93 (s,H, Ar-CH=N-) , 7.00-8.40 (m,H, Ar-H), 2.34 (S ,H, CH$_{aliph.}$); ^{13}C-NMR (100 MHz, DMSO, δ, ppm): 159.01 (C, Ar-C=N-), 112.74-143.50 (C,C$_{Ar}$), 17.70 (C,C$_{aliph.}$); MS (EI, m/z): calculated, 712.85; found 713.21 [M+H]; 735.47 [M+Na].

At room temperature, all the synthesized compounds dissolved in dimethylsulfoxide and acetone, partially, were soluble in ethanol, and soluble in solvents like hexane benzene and water (Table 1 and 2).

Table 1. Solubility of synthesized compounds [N1-N5]

Comp. no.	Solvents					
	DMSO	acetone	water	Hexane	Ethanol	benzene
[N1]	+	+	-	-	δ	-
[N2]	+	+	-	-	δ	-
[N3]	+	+	-	-	δ	-
[N4]	+	+	-	-	δ	-
[N5]	+	+	-	-	δ	-

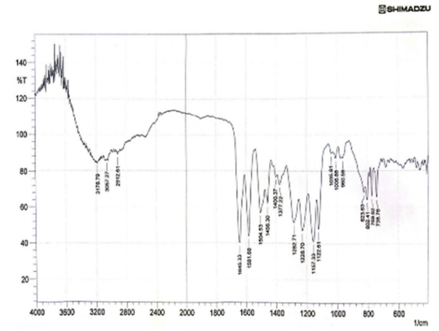
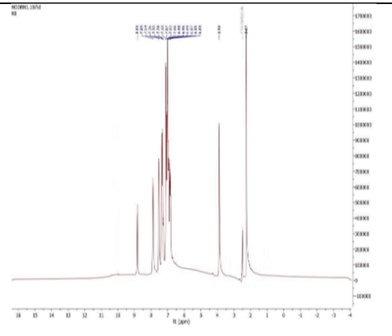
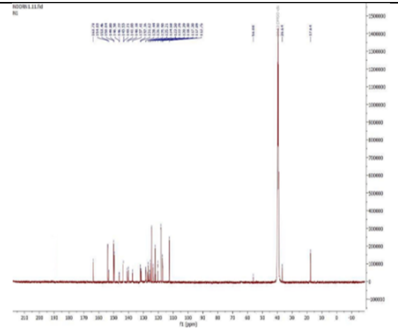
*soluble (+), partially soluble (δ), insoluble (-)

Table 2. Physical properties for synthesized compounds [N1-N5]

Comp. No.	**Chemical structure**	**color**	**Melting point**	**yield**	**TLC**		
					solvent	**Ratio**	**R_f**
[N1]	$C_{43}H_{33}F_3N_2O_4$	Orange	118-120	84	Ethylacetate:hexane	3:2	0.7
[N2]	$C_{43}H_{33}F_3N_2O_2$	Yellow	115-117	82	Ethylacetate:hexane	3:2	0.5
[N3]	$C_{43}H_{31}F_3N_4O_6$	Red	163-165	87	Ethylacetate:hexane	3:2	0.8
[N4]	$C_{57}H_{45}F_3N_2O_2$	Pale red	160-162	89	Ethylacetate:hexane	3:2	0.6
[N5]	$C_{47}H_{39}F_3N_4$	Dark yellow	185-187	84	Ethylacetate:hexane	3:2	0.7

Results and discussion

Novel Schiff base compounds were synthesized through a condensation reaction of one mole of di amine derivatives (4,4'-(1-(9H-fluoren-2-yl)-2,2,2-trifluoroethane-1,1-diyl)bis(2-methylaniline) [N] with two mole of different aromatic aldehydes (Scheme 1). The aldehyde used has less steric effect that eases the path for the nucleophile to approach to the carbonyl group. Further these aldehydes have electrons with donating group that accelerate the reaction. Glacial acetic was used as a catalyst to activate amine and make it more reactive toward the carbonyl groups in the aldehydes by protonating it. This reaction results in formation of an imine intermediate, which forms Schiff base compounds. The reaction completion time was monitored using Thin Layer Chromatography (TLC), which shows new spots for target compounds. The purity of the final products was checked using different solvents, using factors such as polarity, with the retention factor begin 0.5-0.8, and the yield of synthesis compounds begin 82-89, depending on the groups attached to aromatic aldehydes. All absorption peaks for the synthesized compounds were in agreement with the literature. The IR spectra of compounds display peaks at 3392-3178, 3078-3034, 2985-2912, 1689-1620 1645-1573, indicating the presence of (s, OH), (s, CH_{arm}), (m, $CH_{aliph.}$), (m, CH_{imine}), (m, $C{=}N_{imine}$), (m, $C{=}C_{arm.}$); and absence of the amine group of compound [N] at (3441, 3362) (Fig. 1, Table 3). ^{1}H-NMR display signals at 9.93 -8.83 (H, CH=N), 8.68-7.89 for (H, OH), 8.40-5.17 (H, $CH_{arm.}$), and 2.38-2.27 (H, $CH_{aliph.}$). ^{13}C-NMR show signals at 166.88-159.01 (C, CH=N), 165.71-153.46 (C, C-OH), 160.43-112.75 (C, $C_{arm.}$), and 18.23 -17.70 (C, C_{aliph}) (Fig. 1, Table 3). Mass Spectroscopy structurally validated the synthesized compounds through molecular ions (m/z) and the other peaks indicated pieces of ions, as shown in Table (4).

Co. no.	**FTIR**	**^{1}H-NMR**	**^{13}C-NMR**
[N1]			

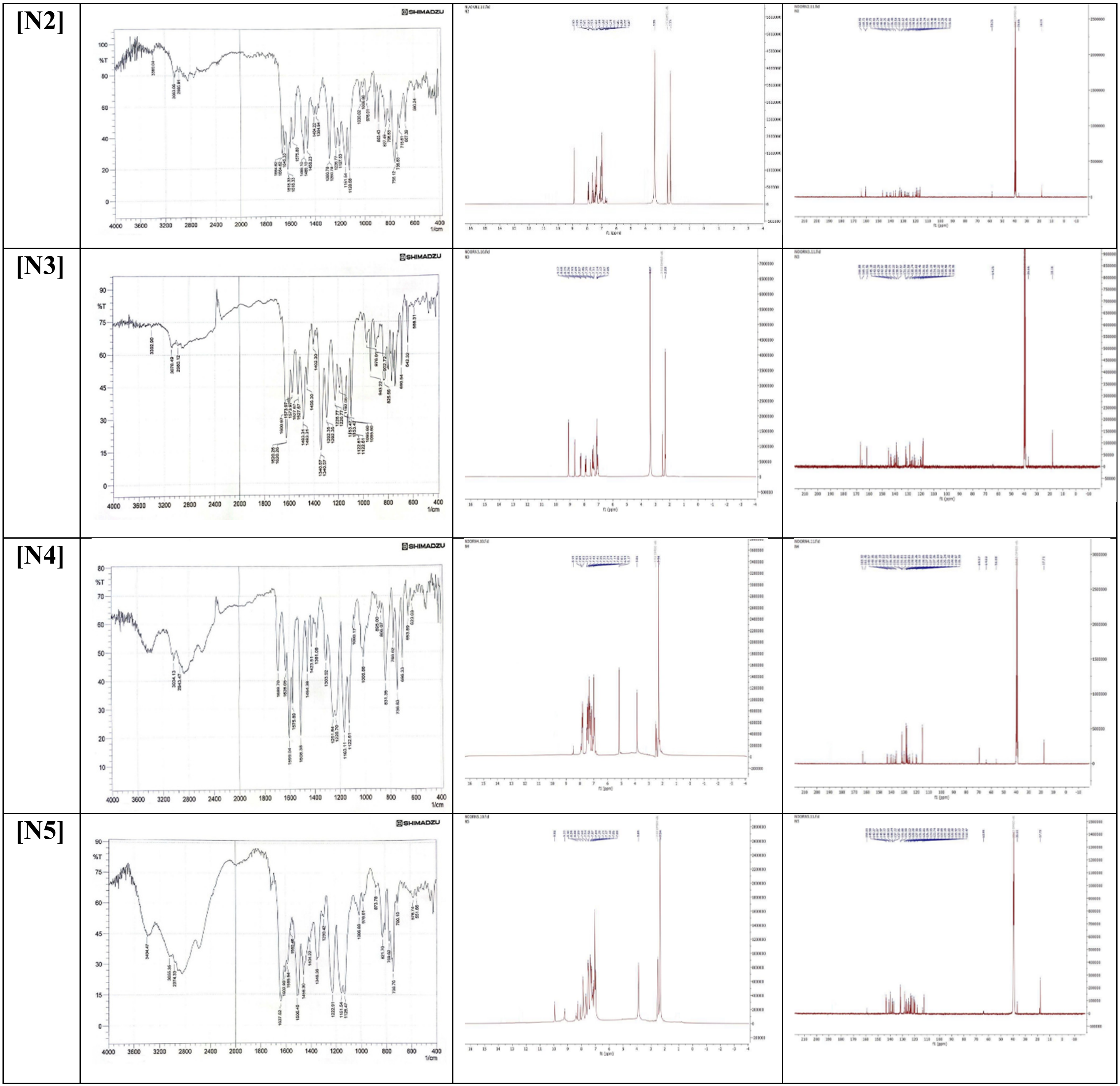


Fig. 1. FTIR, ^{1}HNMR and ^{13}CNMR spectrum for compounds [N1-N5]

Table 3. Fundamental bands of synthesis compounds [N1-N5] in FT-IR spectra

Comp. no.	**OH** ν **(cm^{-1})**	**CH$_{arm.}$** ν **(cm^{-1})**	**CH$_{aliph.}$** ν **(cm^{-1})**	**C=N$_{imine}$** ν **(cm^{-1})**	**C=C$_{arm.}$** ν **(cm^{-1})**	**C-F** ν **(cm^{-1})**	**Other bands** ν **(cm^{-1})**
[N1]	3178	3057	2912	1645	1581	1122	-
[N2]	3389	3063	2985	1664	1645	1120	-
[N3]	3392	3078	2980	1620	1573	1126	NO_2 1153,1340
[N4]	-	3034	2943	1689	1626	1122	C-O-C 1006
[N5]	-	3055	2974	1637	1602	1126	N-H 3404

Table 4. ^{1}HNMR , ^{13}CNMR and Mass spectrum of synthesis compounds [N1-N5]

Comp. no.	**^{1}HNMR δ (ppm)**	**^{13}CNMR δ (ppm)**	**Mass spectrum m/z (g/ mol)**
[N1]	8.83 (s, H, CH=N) 7.89 (s, H, OH) 7.45 (s, H, OH) 6.83-7.36 (m,13H, $CH_{arm.}$) 3.90 (s, H, ph-CH_2-ph) 2.27 (s, H, $CH_{aliph.}$)	163.73 (C, CH=N) 154.03 (C,C-OH) 153.46 (C,C-OH) 112.75-150.04 (m, 22C, C_{arm}) 132.16 (C, CF_3) 56.08 (C, ph-CH_2-ph) 36.64 (3ph-C-CF) 18.17 (2C, $C_{aliph.}$)	calculated 698.74 found 699.23 [M+H] base peak 579.21
[N2]	8.92 (s, H, CH=N) 7.93 (s, H, OH) 6.77-7.90 (m, 14H, $CH_{arm.}$) 3.38 (s, H, ph-CH_2-ph) 2.30 (s, H, $CH_{aliph.}$)	163.93 (C, CH=N) 160.75 (C,C-OH) 116.66-160.43 (24C, C_{arm}) 132.69 (C, CF_3) 58.26 (C, ph-CH_2-ph) 36.66 (3ph-C-CF) 18.23 (2C, $C_{aliph.}$)	calculated 666.74 found 667.25 [M+H] base peak 563.22
[N3]	9.12 (s, H, CH=N) 8.68 (s, H, OH) 7.05-8.29 (m, 13H, $CH_{arm.}$) 3.37 (s, H, ph-CH_2-ph) 2.33 (s, H, $CH_{aliph.}$)	166.88 (C, CH=N) 165.71 (C,C-OH) 161.96 (C, C-NO_2) 118.53-146.18 (22C, C_{arm}) 131.94 (C, CF_3) 64.25 (C, ph-CH_2-ph) 36.66 (3ph-C-CF) 18.16 (C, $C_{aliph.}$)	calculated 756.74 found 755.24 [M-H] base peak 606.15
[N4]	8.49 (s, H, CH=N) 5.17-7.93 (m, 15H, $CH_{arm.}$) 3.86 (s, H, ph-CH_2-ph) 2.38 (s,H, $CH_{aliph.}$)	163.32 (C, CH=N) 161.45 (C,C-O-C) 115-143.57 (26C, C_{arm}) 131.97 (C, CF_3) 69.57 (C, ph-CH_2-ph) 64.03 (C, ph-CH_2-O) 56.08 (3ph-C-CF) 17.71 (2C, $C_{aliph.}$)	calculated 846.9 found 847.17 [M+H] base peak 458.89
[N5]	9.93(s, H, CH=N) 9.22 (s, H,NH) 7.00-8.40 (m, 13H, $CH_{arm.}$) 3.89 (s, H, ph-CH_2-ph) 2.34 (s, H, $CH_{aliph.}$)	159.01(C, CH=N) 112.74-143.50 (26C, C_{arm}) 131.88 (C, CF_3) 63.99 (C, ph-CH_2-ph) 36.61 (3ph-C-CF) 17.70 (2C, $C_{aliph.}$)	calculated 712.85 found 713.21 [M+H] base peak 586.23

Anti-oxidant activity. All synthesized compounds exhibited high inhibition against the stable (2,2-diphenyl-1-bicrylhydrazyl) free radical. This can be used to measure the radical scavenging activity of antioxidants. The DPPH contains an odd electron on the nitrogen atom which is reduced by subtracting a hydrogen atom from the antioxidant; the scavenging percentage for compounds [N1-N5] (80, 52, 65, 78, 68) % for 0.12 mg/ml and (84, 77, 77, 85, 78) % for1 mg/ml means that the inhibition activity increases with as increase in the concentration of all the synthesized compounds as shown in Table 5 and Fig. (2-7).

Table 5. Anti-oxidant activity of compounds [N1-N5]

Comp no.	**0.12 mg/ml**		**0.25 mg/ml**		**0.5 mg/ml**		**1 mg/ml**	
	absorbance	**scavenging %**	**absorbance**	**scavenging %**	**absorbance**	**scavenging %**	**absorbance**	**scavenging %**
[N1]	0.2154	80.785	0.2031	81.882	0.1828	83.693	0.1696	84.871

[N2]	0.5227	52.748	0.4719	57.340	0.324	70.711	0.2516	77.255
[N3]	0.3642	65.654	0.3431	67.644	0.3416	67.786	0.2367	77.678
[N4]	0.2238	78.831	0.1742	83.523	0.1715	83.778	0.1487	85.935
[N5]	0.3266	68.411	0.2875	72.193	0.2626	74.601	0.2273	78.015

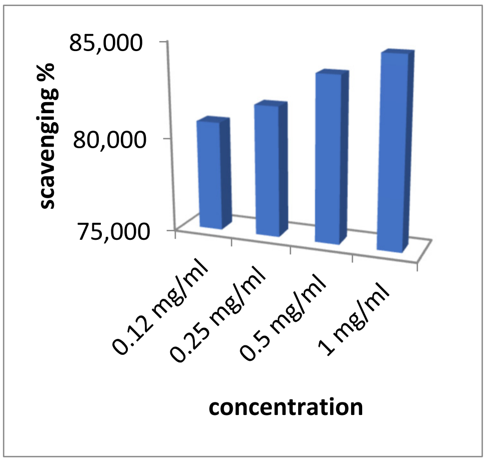


Fig. 2. Antioxidant activity of the [N41] compound

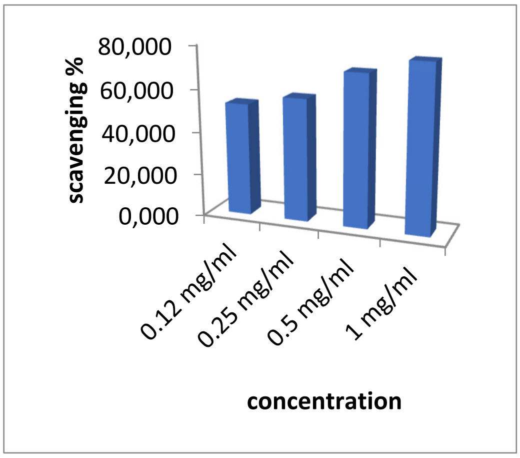


Fig. 3. Antioxidant activity of the [N2] compound

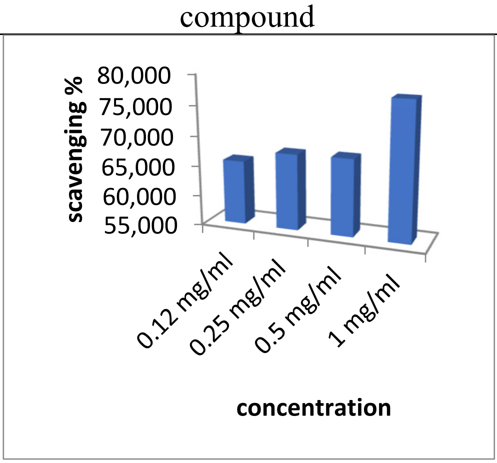


Fig. 4. Antioxidant activity of the [N3] compound

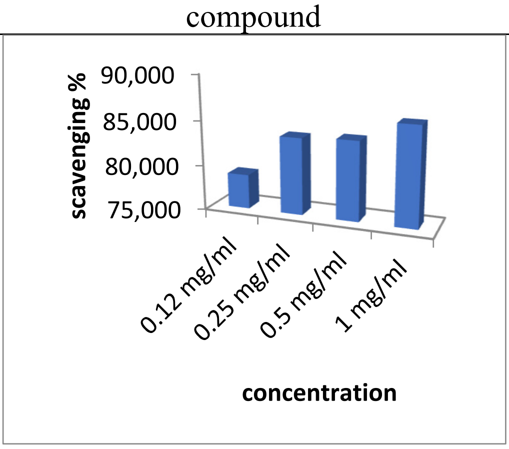


Fig. 5. Antioxidant activity of the [N4] compound

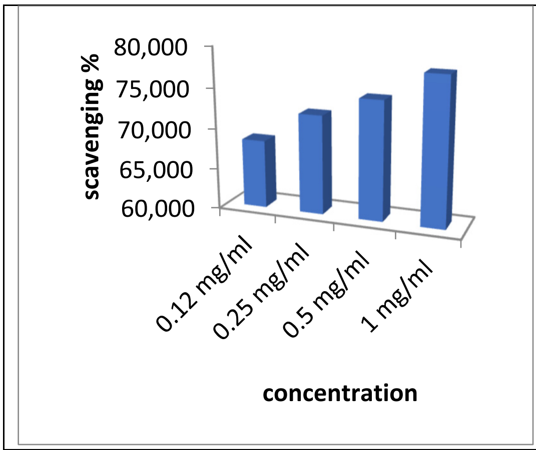


Fig. 6. Antioxidant activity of the [N5] compound

Fig. 7. Change in color of DPPH free radical with change in concentration of compounds [N1-N5]

Conclusions

All the synthesized compounds were colored, stable at room temperature and showed good antioxidant activity with good scavenging ability against 2,2-diphenyl-1-picrylhydrazyl between 77.255 and 85.935. The color can be attributed to the chromophore group which represented by imine. The Schiff base compounds showed good yield due to the use of aromatic aldehydes containing electrons in the donating group which react with aromatic diamine. Glacial acetic acid was used as catalyst, and it released water during the condensation reaction. The synthesized compounds were characterized by FT-IR, Mass, ^{1}H-, and ^{13}C-NMR spectroscopy.

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