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SYNTHESIS AND CHARACTERIZATION OF SCHIFF'S BASES LINKED TO NEW SUCCINIMIDES THROUGH PHENYL RING MOIETY

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ABSTRACT

Some Schiff bases linked to new Succinimides have been synthesized via multistep synthesis. The first step involved reaction of succinic anhydride with P-Amino Acetophenone producing N-(4-acetophenyl) succinamic acid which was subsequently dehydrated to the corresponding N-(4-aceto phenyl)succinimide via treatment with acetic anhydride and anhydrous sodium acetate; and this in turn when introduced in condensation reaction with various aromatic amines afforded the target new Succinimides Linked to Schiff's bases through phenyl ring moiety. Structures of the prepared compounds were elucidated on the basis of FTIR, ^{1}HNMR and ^{13}CNMR spectral data which agreed with the proposed structures. The newly synthesized compounds are expected to have biological activity since they are built from biologically active components including succinimide and Schiff base.

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INTRODUCTION

Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis, and as polymer stabilizers (Dhar, 1982). Schiff bases have also been shown to exhibit a broad range of biological activities, including antifungal, antibacterial, antimalarial, antiproliferative, anti-inflammatory, antiviral, and antipyretic properties (Dhar, 1982; Przybylski *et al.,* 2009). Imine or azomethine groups are present in various natural, natural-derived, and non-natural compounds. The imine group present in such compounds has been shown to be critical to their biological activities (Cleiton et al., 2011; de Souza *et al.,* 2007; Guo *et al.,* 2007). On the other hand Cyclic imides are considered as an important functionality which have been found to maintain significant biological activities and pharmaceutical uses (Bagdahi *et al.,* 2007) such as anticancer (Huang *et al.,* 2004; Al-Zoubi, 2013; Rossi *et al.,* 2006; Sondhi *et al.,* 2009), antitumor (Al-Azzawi and Hassn, 2014; Andricopulo *et al.,* 2000; Al-Azzawi and Mehdi, 2010), analgesic (Andricopulo *et al.,* 2000), anti-inflammatory (Mahapatra *et al.,* 2010), anticonvulsant (Khan *et al.,* 2009; Bhat *et al.,* 2010; Vameca *et al.,* 2000), antimicrobial (Yeo et al., 2005; Shen *et al.,* 2013) and antiviral (Samee *et al.,* 2004). Succinimides and their N-substituted derivatives are key structural units in many important compounds including plant growth stimulator, additives for lubricating oils, corrosion inhibitors, drugs for memory enhancement and antitumor agents (Hargreaves *et al.,* 1970; Toja *et al.,* 1991).

MATERIALS AND METHODS

Melting points were determined on Thomas Hoover apparatus and were uncorrected. FTIR spectra were recorded on SHIMADZU FTIR-8400 Fourier Transform Infrared Spcetrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on Bruker 300 MHz instrument in Al-Albate University in Jordan using tetramethylsilane (TMS) as an internal standard and DMSO-d6 as solvent. 2-3-1- Preparation of N-(4-aceto phenyl)succinamic acid [15] To a solution of (0.01 mol, 1g) of succinic anhydride in (25 mL) of acetone, (0.01 mol, 1.35g) of 4-amino acetophenone dissolved in (10 mL) of acetone was added dropwise with stirring and cooling

(Al-Azzawi and Ali, 2008; Brana and Ramos, 2001). Stirring was continued for two hours at room temperature and the resulted solid was filtered, dried and recrystallized from ethanol. The Physical properties of compound [15] are listed in Table (1).

2-3-2- Preparation of N-(4-aceto phenyl)succinimide [16]

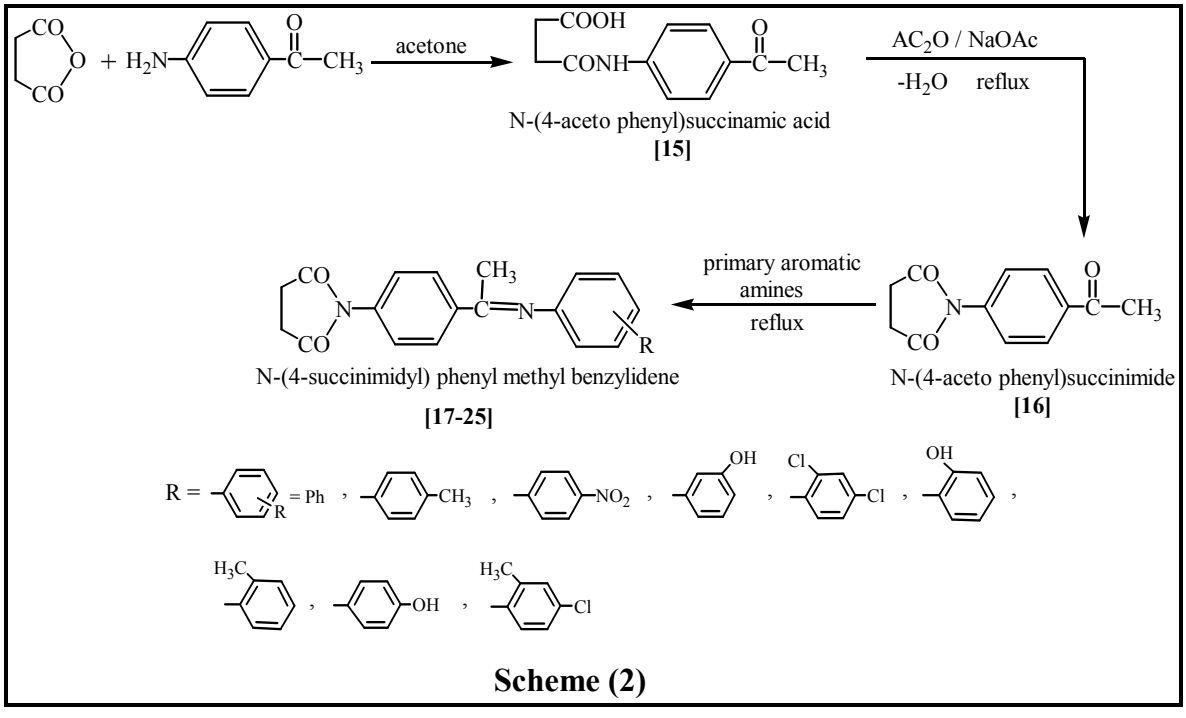
A mixture of (0.01 mol, 2.35 g) of N-(4-aceto phenyl) succinamic acid in (25 mL) of acetic anhydride and (5 %) by weight of anhydrous sodium acetate was refluxed for two hours with stirring (Al-Azzwai and Hassan, 2010; Al-Azzwai and Hassan, 2014). The resulted homogenous solution was cooled to room temperature and poured into crushed ice with stirring. The obtained solid was filtered, dried and recrystallized from cyclohexane. The Physical properties of compound [16] are listed in Table (1).

2-3-3-Preparation of Schiffs bases: 4-(N-succinimidyl) phenyl methyl benzylidene) [17-25]

A mixture of N-(4-aceto phenyl) succinimide (0.01 mol, 2.17 g), primary aromatic amine (0.01 mol) and (2-3) drops of glacial acetic acid in absolute ethanol (20 mL) was refluxed for six hours (Konstantinova and Miladinova, 2009). After cooling the obtained precipitate was filtered, washed with cold ethanol, dried and recrystallized from a suitable solvent. The Physical properties of schiffs Bases [17-25] are listed in Table (2).

RESULTS AND DISSCUSION

This involved synthesis of new succinimides linked to Schiff's bases through phenyl ring moiety. Performing this target involved three steps which are summarized in scheme (2).



Scheme (2)

3-2-1- N-(4-aceto phenyl)succinamic acid [15]

The first step involved synthesis of N-(4-acetophenyl) succinamic acid via reaction of succinic anhydride and 4-amino acetophenone.

FTIR spectrum of compound [15] showed two clear characteristic absorption bands at 3338 cm^{-1} and 3224 cm^{-1} which are due to ν(NH) amide and ν(OH) carboxylic. ν(C=O) carboxyl and ν(C=O) amide absorption bands appeared at 1716 cm^{-1} and 1693 cm^{-1} while absorption bands due to ν(C=C) aromatic and ν(C=O) ketone appeared at 1593 cm^{-1} and 1645 cm^{-1} respectively. ^{1}H-NMR spectrum of compound [15] showed signal at (δ=2.37) ppm belong to (CH_3) protons (Silverstein and Bassler, 1981), multiplet signal at (δ=2.42-2.6)ppm belong to (CH_2CH_2) protons and signals at (δ=6.01-8.39) ppm belong to aromatic protons. The spectrum showed also singlet signal at (δ=10.3) ppm belong to (NH) proton and singlet signal at (δ=12.1) ppm belong to (OH) carboxyl proton. ^{13}C-NMR spectrum of compound [15] showed signals at (δ=25.7-26.3) ppm belong to(CH_3) carbon, signals at (28.6-31.1) ppm belong to (CH_2CH_2) carbons and signals at (δ=112.4-153.5) ppm belong to aromatic carbons. Other signals appeared at (δ=170.7, 173.7 and 196.39) ppm which belong to(C=O) amide, (C=O) carboxyl and (C=O) ketone carbons respectively (Silverstein and Bassler, 1981). The FTIR spectral data of compound (15) are listed in table (3).

3-2-2- N-(4-aceto phenyl)succinimide[16]

The titled compound [16] was prepared in the second step of this part via dehydration of amic acid [15], In preparation of compound [16] acetic anhydride and anhydrous sodium acetate are used as dehydrating agent. FTIR spectrum of compound [16] showed disappearance of ν(OH) carboxyl and ν(NH) amide absorption bands and appearance of absorption bands at 1772 cm^{-1} and 1712 cm^{-1} (Silverstein and Bassler, 1981) due to asym. and sym. ν(C=O) imide. These two points are excellent proofs for success of dehydration reaction. Other absorption bands appeared at 1602 cm^{-1}, 1394 cm^{-1} and 1683 cm^{-1} which belong to ν(C=C) aromatic, ν(C-N) imide and ν(C=O) ketone respectively. ^{1}HNMR spectrum of compound [16] showed disappearance of signals belong to (OH) carboxyl and (NH) amide protons and this is a very important proof for success of imide formation. The spectrum showed signal at

(δ=2.52) ppm belong to (CH_3) protons, signals at (δ=2.61-2.81) ppm belong to (CH_2CH_2) protons and signals at (δ=7.45-8.4) ppm belong to aromatic protons. ^{13}CNMR spectrum of compound [16] showed signals at (δ=24.11) ppm belong to (CH_3) carbon, signals at (δ=26.32-29.15) ppm belong to (CH_2CH_2) carbons and signals at (δ=114.2-136.71) ppm belong to aromatic carbons. Other signals appeared at (δ=176.54-197.24) ppm which belong to (C=O) imide and (C=O) ketone carbons. The FTIR spectral data of compound (16) are listed in table (3).

3-2-3- Schiffs Bases:N-(4-succinimidyl) phenyl methyl benzylidene [17-25]

The third step involved preparation of new succinimides linked to Schiff's bases via introducing of compound [16] in condensation reaction with different primary aromatic amines. FTIR spectra of compounds [17-25] showed disappearance of ν(C=O) ketone absorption band at 1683 cm^{-1} and appearance of absorption bands at (1596-1676) cm^{-1} due to □(C=N) imine

Table (1): Physical properties of compounds [15,16]

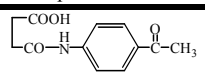
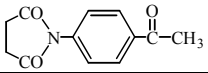
Comp.No.	Compound structure	Color	Melting Points °C	Yield%	Recrystallization solvent
15		Yellow	203-205	84	Ethanol
16		White	162-163	72	Cyclohexane

Table (2): Physical properties of compounds [17-25]

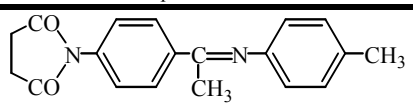
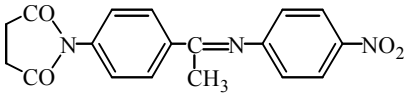
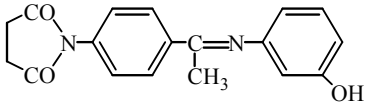
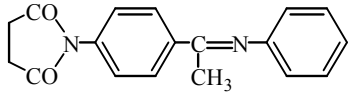
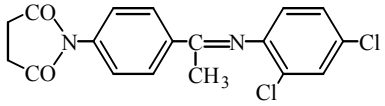
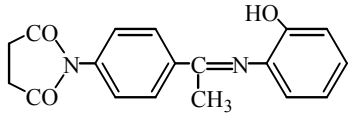
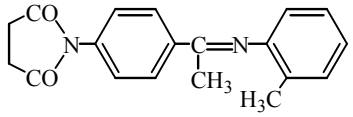
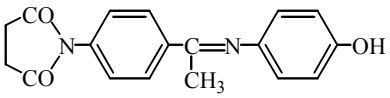
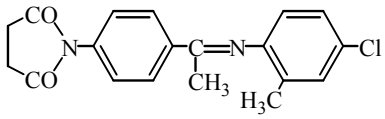
Comp. No.	Compound structure	Color	Melting Points °C	Yield %	Recrystallization solvent
17		White	192-194	86	Acetone
18		Yellow	190-191	73	Acetone
19		Brown	174-176	76	Ethanol
20		White	186-188	77	Dioxane
21		White	178-180	84	Acetone
22		White	182-183	85	Ethanol
23		White	184-186	82	Acetone
24		Brown	171-173	80	Ethanol
25		White	197-198	81	Acetone

Table (3): FTIR spectral data (cm^{-1}) of compounds [15,16]

Comp. No.	ν(O-H) carboxylic	ν(N-H) amide	ν(C-H) aromatic and aliphatic	ν(C=O) carboxylic	ν(C=O) ketone	ν(C=O) amide	ν(C=C) aromatic
15	3338	3224	3090 2937	1716	1645	1693	1593
Comp. No.	ν(C-H) aromatic	ν(C-H) aliphatic	ν(C=O) imide	ν(C=C) aromatic	ν(C=O) ketone		ν(C-N) Imide
16	3060	2970	asym.1772 sym.1712	1602	1683		1394

Table (4): FTIR spectral data (cm^{-1}) of Schiff's bases [17-25]

Comp. No.	FTIR spectral data cm^{-1}						
	ν(C-H) aromatic	ν(C-H) aliphatic	ν(C=O) imide	V(C=N) imine	ν(C=C) aromatic	ν(C-N) Imide	Others
17	3045	2930	1778 asym. 1706 sym.	1629	1602	1390	-
18	3062	2923	1676 asym. 1699 sym.	1676 1645	1598	1392	V(NO2) 1500,1342
19	3080	2923	1774 asym. 1706 sym.	1604	1558	1390	V(OH) phenolic 3460
20	3070	2954	1740 asym. 1715 sym.	1664	1600	1375	-
21	3060	2923	1720 asym. 1689 sym.	1598	1560	1370	V(C-Cl) 1095
22	3080	2970	1730 asym. 1689 sym.	1596	1570	1357	V(OH) phenolic 3427
23	3199 3060	2972	1672	1650	1598	1396	-
24	3120	2920	1740 asym. 1706 sym.	1652	1548	1369	V(OH) phenolic 3400
25	3080	2943	1780 asym. 1703 sym.	1637	1600	1398	V(C-Cl) 1097

(Silverstein and Bassler, 1981). Other absorption bands appeared at (1720-1780) cm^{-1}, (1672-1715) cm^{-1}, (1548-1602) cm^{-1} and (1357-1398) cm^{-1} which belong to asym. ν(C=O) imide, sym. □(C=O) imide, ν(C=C) aromatic and ν(C-N) imide respectively. FTIR spectra of compounds [19], [22] and [24] showed absorption bands at (3400-3460) cm^{-1} due to ν(OH) phenolic while FTIR spectra of compounds [21] and [25] showed absorption band at (1095-1097) cm^{-1} due to ν(C-Cl) and FTIR spectrum of compound [18] showed absorption bands at 1500 cm^{-1} and 1342 cm^{-1} due to ν(NO_2). ^{1}H-NMR spectrum of compound [18] showed signal at δ=(2.51) ppm belong to (CH_3) protons, signals at (δ=2.61-2.8) ppm belong to (CH_2CH_2) protons and signals at (δ=7.09-8.21) ppm belong to aromatic protons. ^{13}C-NMR spectrum of compound [18] showed signal at (δ=17.38) ppm belong to (CH_3) carbon and signals at (δ= 26.32-28.52) ppm belong to (CH_2CH_2) carbons. Signals belong to aromatic carbons appeared at (δ=118.14-136.7) ppm while signals belong to (C=N) imine and (C=O) imide carbons appeared at (δ=168 and 176.55) ppm respectively. ^{1}H-NMR spectrum of compound [19] showed signal at (δ=2.36) ppm belong to (CH_3) protons and signals at (δ=2.5-2.6) ppm belong to (CH_2CH_2) protons. Signals for phenolic (OH) was appeared at (δ= 5.15) ppm while signals belong to aromatic protons appeared at (δ=6.45-8.45) ppm. ^{13}C-NMR spectrum of compound [19] showed signal at (δ=23.1) ppm belong to (CH_3) carbon (Silverstein and Bassler, 1981) and signal at (δ=29.4) ppm belong to (CH_2CH_2) carbons. Signals for aromatic carbons appeared at (δ=105-138.1) ppm and signals at (δ= 149.7) and (δ=169.2) ppm belong to (C=N) imine and (C=O) imide carbons respectively. The FTIR spectral data of compounds (17-25) are listed in table (4).

Conclusion

A series of new Schiff bases containing two biologically active components was synthesized successfully by application of multistep synthesis. The newly synthesized compounds were expected to possess high biological activity since they contain two known biologically active moieties.

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