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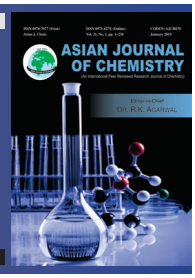

Studies and Synthesis of Substituted 4-Biphenyl Acetamide Derivatives

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A new series of substituted 4-biphenylamides have been synthesized by condensation of 4-biphenyl acetic acid with different primary amines (aromatic and aliphatic). 4-Biphenyl acetic acid was first treated with thionyl chloride in dry benzene to prepare substituted 4-biphenyl acetyl chloride, which is then treated with different aliphatic or aromatic amines to synthesize various substituted 4-biphenyl acid-amide derivatives. The structure of newly synthesized compounds has been established by analytical and spectral methods. These synthesized compounds have shown antifungal properties against *Fusarium udum* and *Curvularia lunata*.

Keywords: Synthesis, 4-Biphenyl acetic acid, Substituted 4-biphenyl acid amides, Antifungal properties.

INTRODUCTION

Biphenyls are the polynuclear aromatic hydrocarbons (PAHs) having more than one aromatic nucleus. Biphenyl acetic acid itself and its derivatives have been found to be effective against many therapeutic diseases. Literature findings have also been shown its various therapeutic uses, such as: anti-inflammatory agent [1], as analgesic [2], antipyretic [3], anti-arthritis [4], antirheumatoid [5], antihypertensive [2] and a binder to human blood plasma-prealbumen, *etc*. 4-Biphenyl acetic acid itself has been reported to possess many effective pharmacological activities, such as anti-inflammatory, analgesic, antibacterial [6] and topical steroidal anti-inflammatory activity [7]. The ointment containing 4-biphenyl acetic acid work very effectively as anti-inflammatory as well as analgesic agents [8]. 4-Biphenyl acetic acid cyclodextrin inclusion compounds are reported to show effective mono-nuclearro-genic anti-inflammatory properties and its phenyl alkanamide derivatives have shown agro horticultural and bactericidal activity [9]. Substituted biphenyls can also be used as anti-allergic drugs [10]. Biphenyl compounds have stronger analgesic activity along with antiallergic and anti-inflammatory activity [7]. Substituted biphenyl-4-acetamides have therapeutic use in the treatment of cancer [11] and is also used as an antitumor agent [12]. Felbinac (an active metabolite of 4-biphenyl acetic acid) patch shows anti-inflammatory and analgesic activities and also used in the treatment of adjuvant-arthritis [2]. Biphenyl-3-acetamide, 2-amino-thiazole shows antitumour activity also used in the treatment of cancer, alzeimer's disease, viral infection, auto-immune disease or neurodegenerative disorder [13]. 2-Biphenyl-acetic acid and 2-biphenyl acetamide used as agrochemical antifungal agent [14-18]. Biphenyl containing compounds possesses anti-psychotic and anxiolytic activity [19]. Some of the biphenyl hydrazide-hydrazone are known to exhibit good antimicrobial activity [20,21]. Some of the compounds having biphenyl moiety possess valuable medicinal properties like antihypertensive and calcium channel blockers [22,23]. Tetrazole are well known to possess antimicrobial properties [24]. Polychlorinated biphenyls (PCBs) are proved to cause reproductive, endocrine and neurological disorders, thyroid dysfunction, cognitive and motor deficits. Prenatal exposures are known to cause increased susceptibility to infectious diseases in early childhood [25]. PCB's influenced plants diffuse oxygen in soil promoting the growth of aerobic microbes. Soil aeration is also improved by formation of air channels when roots die and decay and by direct root oxygen release [26]. Molecules containing biaryl moieties are relatively common within natural products. For their preparation, nature has developed an ample array of biosynthetic strategies [27]. A number of these biaryl natural products belong to the biogenetic class of lignans [28]. During previous years, we have been investigated a range of analogs of natural products [29].

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These products and many derivatives thereof, both of natural and synthetic origin, have been reported to to antioxidant, anti-inflammatory, antitumor, antidiabetic, antimicrobial, antineurodegenerative, antidepressant, pain control, gastrointestinal, cardiovascular and liver protective properties among others [30,31]. Until 1971, 61 % of PCBs were used in closed electrical systems, 13 % were used in nominally closed systems and 26 % were used in openend applications. After 1971, almost 100 % of all PCBs produced were used in closed electrical systems [32]. In present work, we describe the synthesis of the first representatives of previously unknown imidazoles, benzimidazoles containing a 4-biphenyl group at position 2 and different substituents at nitrogen atom. The presence of not one but two "privileged" fragments in the molecules of these compounds, namely, imidazoles, benzimidazoles and biphenyl should promote multitarget action. We also collected and analyzed the data on the activity of the synthesized biphenyl compounds with respect to a number of biological targets important for the therapy of diabetes mellitus and its vascular complications [33-36]. In view of these observations and in continuation of our research work on biologically active biphenyl derivatives, it is proposed to synthesize substituted biphenyl acid-amide derivatives by the condensation of 4-biphenyl acetic acid precursor first with thionyl chloride (to make its acid chloride) and then with different primary amines (aliphatic or aromatic).

EXPERIMENTAL

Synthesis of N-substituted 4-biphenyl acid amide from 4-biphenyl acetic acid: 4-Biphenyl acetic acid (1 g) dissolved in dry benzene (25 mL), added thionyl chloride (1 mL) dropwise along with constant stirring in a round bottom flask of 50 mL. then refluxed the reaction mixture for 2.5 h on water bath at 78-80 °C. The recovered benzene and thionyl chloride from reaction mixture through distillation traces of solvent through vacuum pump. 4-Biphenyl acetyl chloride (**1**) obtained as a viscous-oil (1.085 g, 100 % yield). It was used to form amides in next step without further purification by condensed it with different suitable primary amines (aromatic as well as aliphatic) to form different types of substituted acid amides.

Synthesis of N-substituted 4-biphenyl acid amides (2-16) from compound 1: Dissolved aliphatic or aromatic primary amine in calculated amount of pyridine or 4 N NaOH (25 mL) and added this mixture in to round bottom flask containing compound **1**. Dry benzene was added slowly drop wise at room temperature under constant stirring. The TLC of reaction-mixture showed that the reaction becomes complete, stirring continue up to the completion of the reaction and then work up the reaction mixture with suitable solvent after approximate 20 h. Reaction mixture was taken in separatory funnel along with distilled water. Compound dissolved in the solvent, washed the solvent with water about 3-4 times to remove the basic nature of the solvent layer. The solvent layer becomes neutral, solvent layer was taken in conical flask and added $MgSO_4$ (to absorb the moisture of solvent layer), waited for 5-10 min. Filtered the solution in round bottom flask and recovered the solvent from reaction mixture by distillation and traces of solvent with the help of vacuum pump. Concentrated residue was treated with *n*-hexane to complete the precipitation. The obtained white or coloured crystalline solid was filtered through Whattman filter paper (No. 42) and washed the precipitation with *n*-hexane about 3-4 times to remove the impurities, dried and weighed (**Scheme-I**).

Characterization data

Phenyl-4-biphenyl N-acetamide (2): Pale yellow crystalline solid; m.f. $C_{20}H_{17}NO$, m.p.: 195-97 °C; yield: 875 mg (64 %); TLC: R_f 0.44 (25 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 81.11 (81.11), H: 6.16 (6.92), N: 6.14 (6.17); O: 6.60 (6.17); ^{1}H NMR (DMSO-d_6) δ: 6.14-8.51 (18H-C_6H_5), 4.66 (1H-NH), 4.31 (2H-alicyclic-CH_2); IR (cm^{-1}): 1519, 1650, 3232, 1219, 317.

4-Biphenyl acetamide (3): White crystalline solid; m.p.: 190-92 ºC; m.f. $C_{14}H_{13}NO$, yield: 1.210 g (84 %); TLC: R_f 0.4337 (20 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 79.31 (79.62), H: 6.91 (6.16), N: 6.58 (6.63), O: 8.51 (8.58); ^{1}H NMR (DMSO-d_6) δ: 6.10-8.18 (9H-C_6H_5), 4.99 (2H-NH_2), 4.36 (2H-alicyclic CH_2); IR (cm^{-1}): 1518, 1652, 3243, 1217, 316.

***p*-Methylphenyl-4-biphenyl N-acetamide (4):** Yellow crystalline solid; ; m.f. $C_{21}H_{19}NO$, m.p.: 199-200 °C; Yield: 1.140 g (80.0 %); TLC: R_f 0.3139 (20 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 83.16 (83.72), H: 4.63 (4.31), N: 6.10 (4.65); O: 6.60 (6.31); ^{1}H NMR (DMSO-d_6) δ: 6.14-8.36 (13H-C_6H_5), 4.02 (1H-NH), 4.10 (2H-alicyclic-CH_2), 4.33 (3H-alicyclic-CH_3). IR (cm^{-1}): 1517, 1639, 3352, 1215, 318.

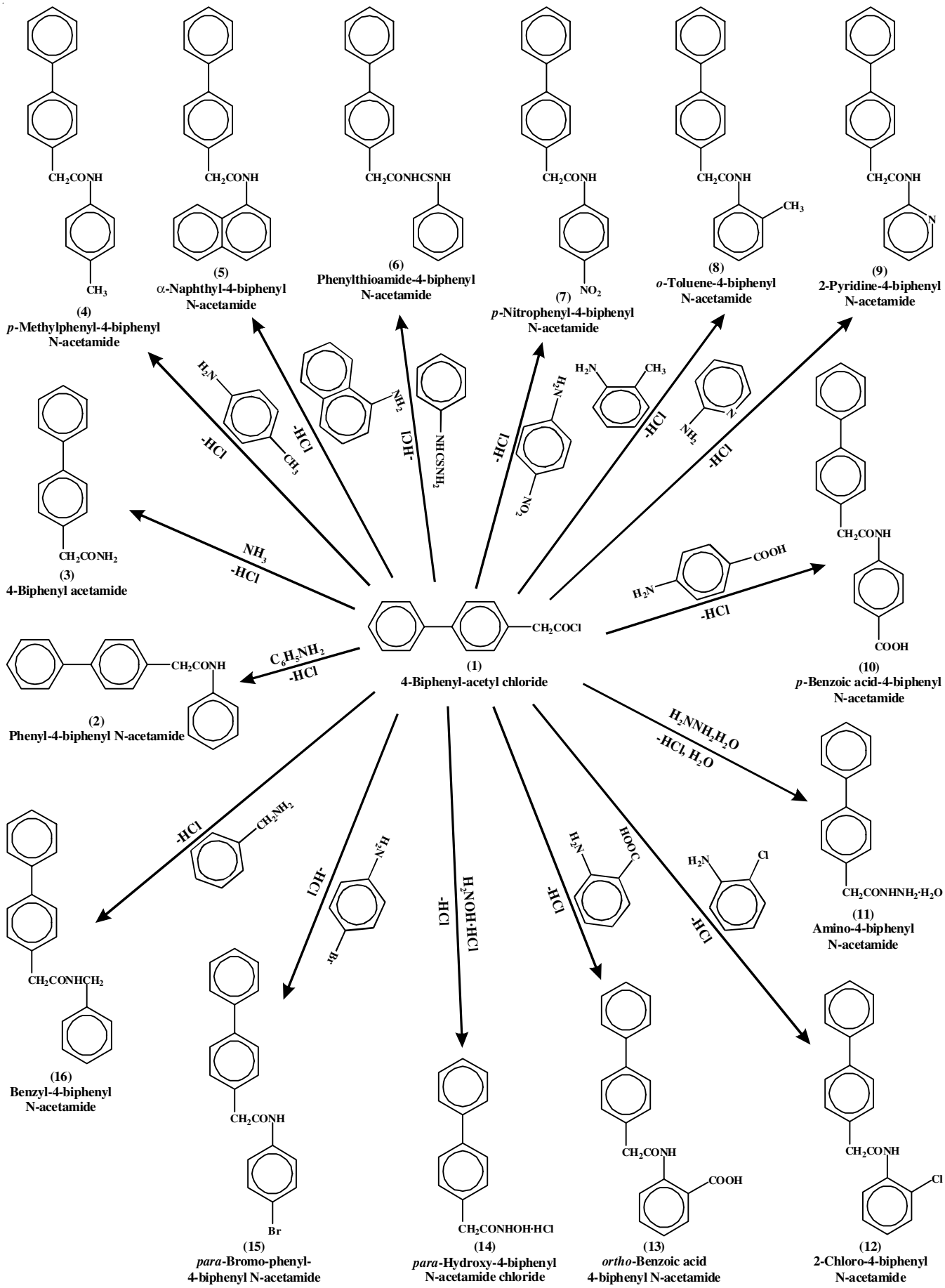
α-Naphthyl-4-biphenyl N-acetamide (5): Purple crystalline solid; ; m.f. $C_{24}H_{19}NO$, m.p.: 188-190 °C; Yield: 610 mg (45 %); TLC: R_f 0.418 (25 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 85.60 (87.45), H: 5.27 (5.63), N: 4.61 (4.10), O: 4.80 (4.74); ^{1}H NMR (DMSO-d_6) δ: 6.17-8.48 (16H-C_6H_5), 4.04 (1H-NH), 4.35 (2H-alicyclic-CH_2); IR (cm^{-1}): 1515, 1658, 3232, 1234, 316.

Phenylthioamide-4-biphenyl N-acetamide (6): Light purple crystalline solid; m.p.: 172-173 °C; m.f. $C_{21}H_{18}N_2OS$, Yield: 390 mg (30 %); TLC: R_f 0.45 (20 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 72.62 (72.83), H: 5.32 (5.20), N: 8.30 (8.09), O: 3.35 (3.62), S: 9.23 (9.24); ^{1}H NMR (DMSO-d_6) δ: 6.13-8.41 (14H-C_6H_5), 4.03 (2H-NH), 4.34 (2H-alicyclic-CH_2); IR (cm^{-1}): 1516, 1652, 3349, 1215, 319.

***p*-Nitrophenyl-4-biphenyl N-acetamide (7):** Pale yellow crystalline solid; m.p.: 170-172 °C; m.f. $C_{20}H_{18}N_2O_3$, Yield: 0.78 g (88.64 %); TLC: R_f 0.559 (50 % chloroform:benzene); Elemental analysis calcd. (found) (%): C: 72.92 (72.28), H: 4.43 (4.42), N: 9.61 (9.43), O: 13.01 (14.45); ^{1}H NMR (DMSO-d_6) δ: 6.18-8.54 (17H-C_6H_5), 4.03 (1H-NH), 4.32 (4H-alicyclic-CH_2); IR (cm^{-1}): 1518, 1675, 3310, 1216, 316.

***o*-Toluene-4-biphenyl N-acetamide (8):** Light brown crystalline solid; m.p.: 163-165 °C; m.f. $C_{21}H_{19}NO$, Yield: 930 mg (70 %); TLC: R_f 0.418 (70 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 80.04 (81.72), H: 2.73 (2.31), N: 7.37 (7.65), O: 9.21 (9.31); ^{1}H NMR (DMSO-d_6) δ: 6.11-8.28 (13H-C_6H_5), 4.08 (1H-NH); 4.33 (2H-alicyclic-CH_2) 4.38 (3H-alicyclic-CH_3); IR (cm^{-1}): 1518, 1688, 3230, 1212, 314.

2-Pyridine-4-biphenyl N-acetamide (9): Light brown crystalline solid; m.p.: 208-210 °C; m.f. $C_{19}H_{16}N_2O$, Yield: 260 mg (42 %); TLC: R_f 0.705 (5 % methanol:chloroform); Elemental analysis calcd. (found) (%): C: 78.85 (78.16), H: 5.90 (5.55), N: 9.23 (9.72), O: 5.29 (5.55); ^{1}H NMR (DMSO-d_6) δ: 6.11-


(1)
4-Biphenyl-acetyl chloride
(2)
Phenyl-4-biphenyl N-acetamide
(3)
4-Biphenyl acetamide
(4)
p-Methylphenyl-4-biphenyl N-acetamide
(5)
α-Naphthyl-4-biphenyl N-acetamide
(6)
Phenylthioamide-4-biphenyl N-acetamide
(7)
p-Nitrophenyl-4-biphenyl N-acetamide
(8)
o-Toluene-4-biphenyl N-acetamide
(9)
2-Pyridine-4-biphenyl N-acetamide
(10)
p-Benzoic acid-4-biphenyl N-acetamide
(11)
Amino-4-biphenyl N-acetamide
(12)
2-Chloro-4-biphenyl N-acetamide
(13)
ortho-Benzoic acid 4-biphenyl N-acetamide
(14)
para-Hydroxy-4-biphenyl N-acetamide chloride
(15)
para-Bromo-phenyl-4-biphenyl N-acetamide
(16)
Benzyl-4-biphenyl N-acetamide
-HCl
NH3
C6H5NH2
H2NNH2H2O
-HCl, H2O
H2NOH·HCl

Scheme-I

8.39 (13H-C_6H_5), 4.02 (1H-NH), 4.52 (2H-NH_2), 4.36 (2H alicyclic -CH_2); IR (cm^{-1}): 1514, 1669, 3331, 1216, 315.

***p*-Benzoic acid-4-biphenyl N-acetamide (10):** Cream crystalline solid; m.p.: 243-245 °C; m.f. $C_{21}H_{17}NO_3$, Yield: 220 mg (44 %); TLC: R_f 0.341 (20 % methanol:chloroform); Elemental analysis calcd. (found) (%): C:72.86 (72.13), H: 6.40 (6.13), N: 5.56 (5.22), O: 15.01 (15.50); ^{1}H NMR (DMSO-d_6) δ: 6.05-8.31 (13H-C_6H_5), 4.02 (1H-NH); 4.35 (2H-alicyclic-CH_2); IR (cm^{-1}): 1513, 1666, 3305, 1207, 133.

Amino-4-biphenyl N-acetamide (11): Pale yellow crystalline solid; m.p.: 232-233 °C; m.f. $C_{14}H_{16}N_2O_2$, Yield: 490 mg (92 %); TLC: R_f 0.474 (5 % methanol:chloroform); Elemental analysis calcd. (found) (%): C:70.76 (70.85), H: 5.80 (5.55), N: 10.80 (10.47), O: 12.90 (12.11); ^{1}H NMR (DMSO-d_6) δ: 6.02-8.05 (9H-C_6H_5), 4.05 (1H-NH); 4.37 (2H-alicyclic-CH_2); IR (cm^{-1}): 1516, 1692, 3209, 1233, 312.

2-Chloro-4-biphenyl N-acetamide (12): White crystalline solid; m.p.: 136-38 °C; m.f. $C_{20}H_{16}NOCl$, Yield: 325 mg (92 %); TLC: R_f 0.392 (10 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C:76.76 (76.12), H: 5.20 (5.12), N: 4.49 (4.36), O: 4.16 (4.98), Cl: 10.09 (10.05); ^{1}H NMR (DMSO-d_6) δ: 6.12-8.29 (13H-C_6H_5), 4.05 (1H-NH); 4.27 (2H-alicyclic-CH_2); IR (cm^{-1}): 1524, 1692, 3248, 1223, 131.

***ortho*-Benzoic acid 4-biphenyl N-acetamide (13):** Dirty Yellow crystalline solid; m.p.: 162-163 °C; m.f. $C_{21}H_{17}NO_3$, Yield: 225 mg (32 %); TLC: R_f 0.2809 (30 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 72.86 (72.13), H: 6.40 (6.13), N: 5.56 (5.22), O: 15.01 (15.50); ^{1}H NMR (DMSO-d_6) δ: 6.17-8.28 (13H-C_6H_5), 4.01 (1H-NH); 4.34 (2H-alicyclic-CH_2) 11.2 (1H. CO-OH); IR (cm^{-1}): 1509, 1663, 3451, 1233, 130.

***para*-Hydroxy-4-biphenyl N-acetamide chloride (14):** Mustered crystalline solid; m.p.: 178-180 ºC; m.f. $C_{14}H_{14}NO_3Cl$, Yield: 175 mg (35 %); TLC: R_f 0.648 (70 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 68.08 (63.87), H: 5.17 (5.32), N: 9.48 (9.32), O: 5.24 (5.16), Cl: 11.78 (13.30); ^{1}H NMR (DMSO-d_6) δ: 6.01-8.07 (11H-C_6H_5), 4.04 (1H-NH); 12.01 (1H-COOH), 4.32 (2H-alicyclic-CH_2); IR (cm^{-1}): 1512, 1617, 3199, 1219, 135.

***para*-Bromo-phenyl-4-biphenyl N-acetamide (15):** Pink crystalline solid; m.p.: 133-135 °C; ; m.f. $C_{20}H_{16}NOBr$, Yield: 320 mg (48 %); TLC: R_f 0.563 (25 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 63.08 (65.57), H: 5.17 (5.37), N: 5.22 (5.82), O: 5.46 (5.16), Br: 20.78 (21.82); ^{1}H NMR (DMSO-d_6) δ: 6.13-8.26 (13H-C_6H_5), 4.02 (1H-NH); 11.91 (1H-COOH), 4.33 (2H-alicyclic-CH_2); IR (cm^{-1}): 1523, 1654, 3291, 1215, 136.

Benzyl-4-biphenyl N-acetamide (16): Pale yellow crystalline solid; m.p.: 196-198 °C; ; m.f. $C_{21}H_{19}NO$, Yield: 590 mg (90 %); TLC: R_f 0.310 (30 % EtOAc:hexane); Elemental analysis calcd. (found) (%): C: 84.66 (83.72), H: 5.57 (6.31), N: 4.97 (4.65), O: 5.10 (5.31); ^{1}H NMR (DMSO-d_6) δ: 6.62-8.40 (14H-C_6H_5), 4.02 (1H-NH); 4.31 (2H-alicyclic-CH_2); IR (cm^{-1}): 1545, 1635, 3287, 1215, 131.

RESULTS AND DISCUSSION

Commercially available 4-biphenyl acetic acid was used for the preparation of substituted 4-biphenyl acid amide derivatives. 4-Biphenyl acetic acid was treated with thionyl chloride in dry benzene on water bath for 2-3 h at 70-80 °C. After 1 h, the colour of reaction mixture becomes change from pale yellow to orange and then on completion of reaction, it changes from orange to brown. This oily mass of 4-biphenyl acetyl chloride is then treated with different types of aliphatic and aromatic amines in pyridine or 4 N-NaOH to synthesis a series of amides. These synthesized-4-biphenyl acid amides were treated with *n*-hexane for crystallization. The synthesized 4-biphenyl amides have been studied for their antifungal activity against *Fusarium udum* and *Curvularia lunata*. The culture of each species was incubated at 12 ± 3 °C and the zone of the inhibition was measured after 120 h. Most of these compounds were found to be active against these fungi.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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