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Synthesis of Novel 1,2,3-Triazoles-linked Indoles Through Copper(I)-catalyzed Click Reactions

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A series of novel 1,2,3-triazoles-linked indole derivatives were prepared by the reaction of 2-aryl-1-(prop-2-ynyl)-1*H*-indole-3-carbaldehydes with aryl azides *via* copper-catalyzed azide-alkyne cycloaddition reactions in the presence of sodium ascorbate, as a reducing agent. The effects of catalysts, solvents, and reaction temperature were investigated. Under the optimal condition, 1,2,3-triazoles linked-indoles were obtained in 63-92% yields.

Keywords: Indole, Triazole, Aryl azide, Propargyl bromide

INTRODUCTION

Indoles are one of the most important groups of N-containing heterocycles which are frequently found in cruciferous plants and marine sources [1-4], and also identified to possess a wide range of applications in biochemistry, pharmacology, and medicinal chemistry [5,6]. These compounds have shown some different pharmaceutical activities such as anticancer [7-9], anti-inflammatory [10], anti-estrogen, cytotoxic, and antimitotic [11-13].

1,2,3-Triazoles have exhibited wide biological activities including anti-helminthic, antitumor, and pharmacological properties [14-17]. In recent years, copper-catalyzed azide-alkyne cycloaddition (CuAAC) reaction (click reaction), has become one of the most important reactions used for the preparation of 1,2,3-triazoles [18]. It is well known that the CuAAC reaction takes place only in the presence of a Cu(I) species but not in the presence of Cu (II) or Cu(0) species [19-22]. However, Cu(I) salts are not susceptible to redox processes, and thus it is beneficial to protect and stabilize the active copper catalysts during a CuAAC reaction, and this has led to the discovery of various modified methods such as the use of Cu(II)/Cu(0) salts with different additives or ligands [23-25]. Various homogeneous copper catalysts such as $CuSO_4$ [26], CuBr [27], $Cu(OAc)_2$ [28], $CuCl_2$ [29] and heterogeneous catalysts including nano silica [30], nano Alumina [31], Cellulose [32], montmorillonite K10 clay [33], cuttlebone [34] and graphene oxide [35-37], have been reported for this purpose.

Synthesis of 1,2,3-triazoles linked-other pharmacophore through CuAAC reaction is an important method for the synthesis of new biologically active compounds [38-40]. Recently, we have reported the synthesis of 1,2,3-triazoles-linked heterocyclic moieties [41-44]. Our current research presents a highly effective method for synthesizing novel 1,2,3-triazoles-linked indoles.

EXPERIMENTAL

Material and Equipment

All chemicals were used reagent grade, and as received without further purification. ^{1}H NMR spectra were recorded using Bruker Advance DPX-250 NMR FT-300 MHz

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spectrometers instrument in DMSO-d_6. Coupling constants (J) are reported in hertz (Hz), and multiplicities are indicated as follows: s (singlet), d (doublet), dd (doublet of doublet), t (triplet), q (quartet), m (multiplet). IR spectra were recorded on a Shimadzu IR-435 grating spectrophotometer.

General procedure for the synthesis of 1,2,3-triazoles-linked indole derivatives (3a-h). To a 10-ml round-bottomed flask equipped with a magnetic stirrer bar and containing ethanol (3 ml) were added 2-aryl-1-(prop-2-ynyl)-1*H*-indole-3-carbaldehyde 1 (1.0 mmol), an aryl azide 2 (1.0 mmol), sodium ascorbate (0.4 mmol) and $Cu(OAc)_2$ (0.2 mmol). The reaction mixture was stirred at 70 ºC, and the reaction progress was monitored by TLC. The resulting crude product was purified by column chromatography on silica gel (eluent: hexane/ethyl acetate = 10/6) to give the corresponding product.

2-(4-Chloro-phenyl)-1-((1-(3-nitro-phenyle)-1H-1,2,3-triazol-4-yl)methyl)-1H-indole-3-carbaldehyde (3a). Yellow solid (90%); m. p. 178-180 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.52 (s, 2H, CH_2), 7.33-7.38 (m, 3H, ArH), 7.70-7.75 (m, 4H, ArH), 8.24-8.27 (m, 1H, ArH), 8.32-8.38 (m, 3H, ArH), 8.68 (t, J = 2.1 Hz, 1H, ArH), 8.99 (s, 1H, CH of triazole), 9.65 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 112.0, 115.3, 115.8, 121.5, 122.7, 123.6, 123.7, 124.6, 125.2, 126.6, 127.3, 129.3, 132, 133.4, 135.6, 136.8, 137.4, 144.3, 148.9, 150.3, 185.9 ppm; IR (KBr); 3431, 1649, 1601, 1520, 1456, 1338 cm^{-1}.

2-(4-Methoxy-phenyl)-1-((1-(3-nitro-phenyle)-1H-1,2,3-triazol-4-yl)methyl)-1H-indole-3-carbaldehyde (3b). Yellow solid (83%); m. p. 180-182 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 3.36 (s, 3H, CH_3), 5.52 (s, 2H, CH_2), 7.18 (d, J = 8.7 Hz, 2H, ArH), 7.29-7.37 (m, 2H, ArH), 7.67-7.70 (m, 1H, ArH), 7.74 (d, J = 8.7 Hz, 2H, ArH), 7.89 (t, J = 8.2 Hz, 1H, ArH), 8.23-8.26 (m, 1H, ArH), 8.31-8.39 (m, 2H, ArH), 8.69 (t, J = 2.1 Hz, 1H, ArH), 8.98 (s, 1H, CH of triazole), 9.66 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 55.8, 60.2, 111.8, 114.7, 115.3, 115.5, 120.2, 121.4, 122.6, 123.4, 123.5, 123.7, 124.3, 125.3, 126.6, 131.9, 133, 136.7, 137.4, 144.5, 148.9, 152.0, 161.0, 186.0 ppm; IR (KBr); 3437, 1643, 1531, 1496, 1344, 1252, 1036 cm^{-1}.

2-(4-Bromo-phenyl)-1-((1-(3-nitro-phenyle)-1H-1,2,3-triazol-4-yl)methyl)-1H-indole-3-carbaldehyde (3c). Yellow solid (79%); m. p. 194-196 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.52 (s, 2H, CH_2), 7.31-7.39 (m, 2H, ArH), 7.77-7.92 (m, 6H, ArH), 8.24-8.27 (m, 1H, ArH), 8.31-8.38 (m, 2H, ArH), 8.68 (t, J = 2.1 Hz, 1H, ArH), 8.99 (s, 1H, CH of triazole), 9.65 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 112.0, 115.3, 115.7, 121.5, 122.7, 123.6, 123.7, 124.4, 124.6, 125.2, 126.6, 127.6, 132.0, 132.2, 133.6, 136.8, 137.4, 144.3, 148.9, 150.3, 185.9 ppm; IR (KBr); 3430, 1640, 1538, 1491, 1341 cm^{-1}.

2-(4-Bromo-phenyl)-1-((1-(4-nitro-phenyle)-1H-1,2,3-triazol-4-yl)methyl)-1H-indole-3-carbaldehyde (3d). Yellow solid (69%); m. p. 214-216 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.53 (s, 2H, CH_2), 7.32-7.38 (m, 2H, ArH), 7.73-7.77 (m, 3H, ArH), 7.85 (d, J = 8.4 Hz, 2H, ArH), 8.18 (d, J = 9 Hz, 2H, ArH), 8.26 (dd, J = 6.3, 2.5 Hz, 1H, ArH), 8.44 (d, J = 9 Hz, 2H, ArH), 8.95 (s, 1H, CH of triazole), 9.65 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 112.0, 115.7, 121.2, 121.5, 122.6, 123.7, 124.4, 124.6, 125.2, 126.0, 127.6, 132.2, 133.6, 136.8, 141.0, 144.5, 147.2, 150.3, 185.9 ppm; IR (KBr); 3433, 1648, 1520, 1339 cm^{-1}.

2-(4-Methoxy-phenyl)-1-((1-(4-nitro-phenyle)-1H-1,2,3-triazol-4-yl)methyl)-1H-indole-3-carbaldehyde (3e). Yellow solid (86%); m. p. 188-190 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 3.87 (s, 3H, CH_3), 5.52 (s, 2H, CH_2), 7.19 (d, J = 9 Hz, 2H, ArH), 7.31-7.35 (m, 2H, ArH), 7.67-7.71 (m, 1H, ArH), 7.74 (d, J = 9 Hz, 2H, ArH), 8.18 (d, J = 9 Hz, 2H, ArH), 8.23-8.26 (m, 1H, ArH), 8.44 (d, J = 9 Hz, 2H, ArH), 8.94 (s, 1H, CH of triazole), 9.65 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 55.8, 111.8, 114.7, 115.5, 120.2, 121.1, 121.4, 122.6, 123.5, 124.3, 125.3, 126.0, 133.0, 136.7, 141.1, 144.7, 147.2, 152.0, 161.0, 186.0 ppm; IR (KBr); 3431, 1646, 1534, 1343, 1250, 1032 cm^{-1}.

1-((1-(3-Nitro-phenyl)-1H-1,2,3-triazol-4-yl) methyl)-2-phenyl-1H-indole-3-carbaldehyde (3f). Orange solid (92%); m. p. 227-229 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.52 (s, 2H, CH_2), 7.33-7.37 (m, 2H, ArH), 7.64-7.66 (m, 3H, ArH), 7.71 (dd, J = 6, 3 Hz, 1H, ArH), 7.79-7.83 (m, 2H, ArH), 7.89 (t, J = 8.1 Hz, 1H, ArH), 8.24-8.27 (m, 1H, ArH), 8.31-8.38 (m, 2H, ArH), 8.68 (t, J = 2.1 Hz, 1H, ArH), 8.97 (s, 1H, CH of triazole), 9.65 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ

= 111.9, 115.3, 115.6, 121.4, 122.7, 123.6, 123.7, 124.4, 125.2, 126.6, 128.3, 129.2, 130.5, 131.5, 132.0, 136.7, 137.4, 144.4, 148.9, 151.9, 186.0 ppm; IR (KBr); 3437, 3091, 1649, 1603, 1531, 1433, 1342 cm^{-1}.

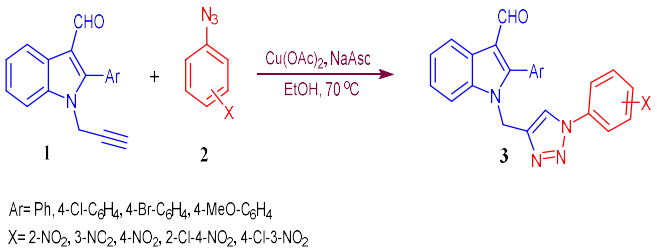
1-((1-(2-Nitro-phenyl)-1H-1,2,3-triazol-4-yl) methyl)-2-phenyl-1H-indole-3-carbaldehyde (3g). Orange solid (63%); m. p. 150-152 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.52 (s, 2H, CH_2), 7.32-7.41 (m, 2H, ArH), 7.64-7.66 (m, 3H, ArH), 7.74-7.88 (m, 5H, ArH), 7.92-7.98 (m, 1H, ArH), 8.20-8.27 (m, 2H, ArH), 8.61 (s, 1H, CH of triazole), 9.63 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 112.0, 121.4, 123.5, 124.4, 125.4, 126, 128.2, 128.3, 128.4, 129.2, 129.4, 130.5, 130.6, 130.7, 131.5, 131.8, 134.9, 136.9, 143.6, 144.4, 185.9 ppm; IR (KBr); 3438, 3090, 1646, 1602, 1530, 1343 cm^{-1}.

1-((1-(2-Chloro-4-nitro-phenyl)-1H-1,2,3-triazol-4-yl)methyl)-2-(4-chloro-phenyl)-1H-indole-3-carbaldehyde (3h). Brown solid (64%); m. p. 101-103 ºC; ^{1}H NMR (DMSO-*d6*, 300 MHz) δ = 5.56 (s, 2H, CH_2), 6.87-6.93 (m, 1H, ArH), 7.82-7.98 (m, 4H, ArH), 8.02-8.12 (m, 1H, ArH), 8.24-8.32 (m, 2H, ArH), 8.37 (s, 1H, ArH), 8.40-8.62 (m, 2H, ArH), 8.66 (s, 1H, CH of triazole), 9.64 (s, 1H, CHO) ppm; ^{13}C NMR (DMSO-d_6, 75 MHz) δ = 60.2, 112.0, 115.8, 121.4, 123.6, 124, 124.5, 125.1, 126.3, 127.3, 129.3, 129.6, 133.1, 133.4, 135.6, 136.8, 139.3, 143.2, 148.7, 149.3, 150.2, 185.8 ppm; IR (KBr); 3437, 2956, 2924, 1643, 1529, 1491, 1456, 1342 cm^{-1}.

RESULTS AND DISCUSSION

In this study, we reported an efficient method for the synthesis of novel 1,2,3-triazole-linked indole framework 3 from the reaction of 2-aryl-1-(prop-2-yn-1-yl)-1*H*-indole-3-carbaldehyde 1 with aryl azides 2 catalyzed by copper(II) acetate in ethanol at 70 ºC (Scheme 1).

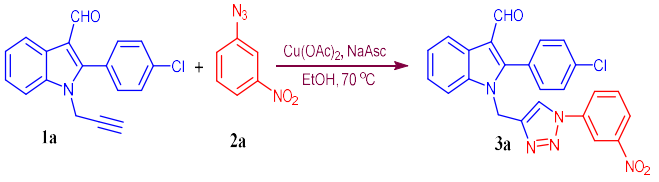
The starting materials 2-aryl-1-(prop-2-yn-1-yl)-1*H*-indole-3-carbaldehyde derivatives 1 were prepared according to our previously reported [34]. In the



Scheme 1. Synthesis of 1,2,3-triazole-linked indole 3

preliminary experiments, the reaction of compound 1 with 1-azido-3-nitrobenzene 2a was examined to find out the best reaction conditions. To find the optimum reaction conditions for the synthesis of compound 3a, the effects of various solvents, copper catalysts, and reaction temperatures were studied, and the results are shown in Table 1. As shown in Table 1, when the model reaction was performed in the presence of various copper catalysts such as $Cu(OAc)_2$, $CuSO_4$, and CuI in ethanol at 50 ºC, the best result was observed with $Cu(OAc)_2$ as a catalyst (Table 1, entry 1). Secondly, the effects of different solvents including MeOH, H_2O, DMF, THF, and CH_3CN on the synthesis of compound 3a were also investigated, and the results were summarized in Table 1. In the above study, the best result was observed with EtOH (Table 1, entry 1). Next, the effect of the dosage of $Cu(OAc)_2$ and reaction temperature on the synthesis of compound 3a was further investigated. When 10 mol% of $Cu(OAc)_2$ was added to the reaction system, 3a was obtained with only a 65% yield (Table 1, entry 10). However, the larger amount of catalyst (30 mol%) was not necessary and helpful for the reaction (Table 1, entry 11). When the reaction was performed at 70 °C, 3a could be obtained with an excellent yield (90%) (Table 1, entry 12). However, higher temperatures did not improve the product yield (Table 1, entry 13). The optimal reaction condition for yield generation requires 1 mmol of indole 1a, 1 mmol of aryl azide 2a, 20 mol% of $Cu(OAc)_2$, 40 mol% of sodium ascorbate, 3 ml of ethanol at 70 °C.

Table 1. Effect of Various Solvents, Catalysts, and Temperatures on the Synthesis of 2-(4-Chlorophenyl)-1-((1-(3-nitrophenyl)-1*H*-1,2,3-triazole-4-yl)methyl)-1*H*-indole-3-carbaldehyde 3a[a]

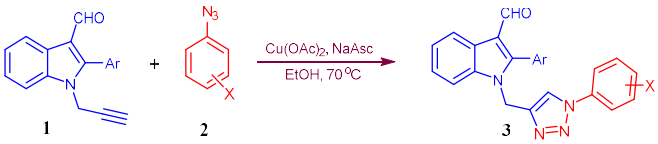


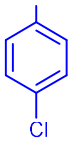
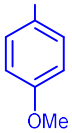
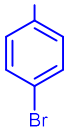
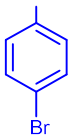
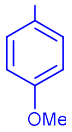
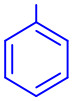
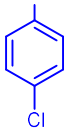
Entry	Catalyst (mol%)	Solvent	Temp. (°C)	Yield (%)[b]
1	$Cu(OAc)_2$ (20)	EtOH	50	80
2	$CuSO_4$ (20)	EtOH	50	56
3	CuI (20)	EtOH	50	65
4[c]	$Cu(OAc)_2$ (20)	MeOH	50	71
5[c]	$Cu(OAc)_2$ (20)	H_2O	50	64
6	$Cu(OAc)_2$ (20)	DMF	50	41
7	$Cu(OAc)_2$ (20)	THF	50	15
8	$Cu(OAc)_2$ (20)	CH_3CN	50	72
9	$Cu(OAc)_2$ (20)	CH_2Cl_2	38	25
10	$Cu(OAc)_2$ (10)	EtOH	50	65
11	$Cu(OAc)_2$ (30)	EtOH	50	81
12	$Cu(OAc)_2$ (20)	EtOH	70	90
13	$Cu(OAc)_2$ (20)	EtOH	90	88

[a]Reaction conditions: compound 1a (1 mmol), 1-azido-3-nitrobenzene 2a (1.0 mmol), copper salt, sodium ascorbate (twice the amount of copper salt), reaction time (3 h), solvent (3 ml). [b]Isolated yield.

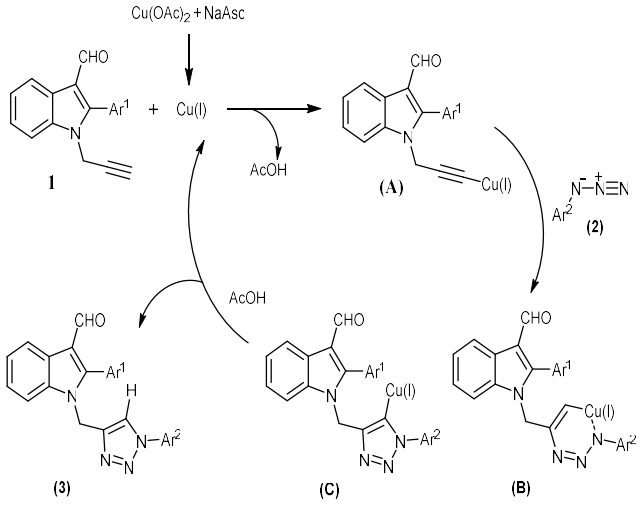
To examine the scope of this method, the optimized conditions were then applied to the synthesis of a variety of 1,2,3-triazoles-linked indole from 2-aryl-1-(prop-2-ynyl)-1*H*-indole-3-carbaldehyde derivatives with various aryl azides (Table 2). According to Table 2, the CuAAC reactions of various substrates, led to 1,2,3-triazoles-linked indole derivatives in good-to-excellent yields (63-92%). As shown in Table 2, 1-azido-3-nitrobenzene, and 1-azido-4-nitrobenzene undergo the CuAAC reaction to give the 1,2,3-

Table 2. Synthesis of 1,2,3-Triazoles-linked Indole *via* Click Reaction[a]



Entry	Ar	X	Time (h)	Product	Yield (%)[b]
1		3-NO_2	4	3a	90
2		3-NO_2	3	3b	83
3		3-NO_2	4	3c	79
4		4-NO_2	5	3d	69
5		4-NO_2	5	3e	86
6		3-NO_2	5	3f	92
7		2-NO_2	5	3g	63
8		2-Cl-4-NO_2	5	3h	64

[a]Reaction conditions: compound 1 (1 mmol), aryl azide 2 (1.0 mmol), $Cu(OAC)_2$ (0.2 mmol), sodium ascorbate (0.4 mmol), EtOH (3 ml), reaction temperature (70 °C). [b]Isolated yield.



Scheme 2. Proposed mechanism

triazoles-linked indole derivatives in high-to-excellent yields (Table 2, entries 1-6). However, the yield of the product was slightly decreased with 1-azido-2-nitrobenzene, and 1-azido-2-chloro-4-nitrobenzene (Table 2, entries 6, and 7 respectively). This may attribute to the steric hindrance of the ortho groups.

A proposed mechanistic route of 1,2,3-triazole-linked indoles synthesis in the presence of $Cu(OAc)_2$ catalyst is displayed in Scheme 2: a) Reduction of Cu(II) to Cu(I) by sodium ascorbate; b) 2-aryl-1-(prop-2-ynyl)-1*H*-indole-3-carbaldehyde derivatives 1 was changed to copper(I) acetylide intermediate A in the presence of Cu(I) catalyst; c) the formation of six- membered ring copper metallacycle B by the reaction of intermediate A with aryl azide 2; d) cyclization to produce of intermediate C; and e) formation of triazole ring and regenerates the catalyst.

CONCLUSION

In summary, we developed a simple and efficient protocol for the synthesis of 1,2,3-triazoles-linked indole derivatives from various indoles and aryl azides with $Cu(OAc)_2$ as a catalyst. This method has many advantages such as readily available catalysts, a wide range of substrates, and the ease and safety of the operation.

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