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Synthesis of Flavones from Chalcones Mediated by Copper(I) Iodide and Azides: Mechanistic Insights

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Abstract

This article presents an alternative method for synthesizing flavones from chalcones using copper(I) iodide (CuI) and two azides-sodium azide and 4-azidobenzenesulfonamide-as key reagents. The azides most likely undergo a Michael addition with chalcones to form 1,2,3-triazoles, mediated by either stoichiometric or catalytic amounts of CuI. Interestingly, the expected 1,2,3-triazoles were not obtained; instead, the forming 1,2,3-triazoles intermediates appeared to undergo immediate intramolecular attack by the C2 hydroxy group of the A-ring of chalcones, followed by elimination of the azides, ultimately yielding flavones. The yields and reaction rates were influenced by the varying amounts of CuI and azides employed. The key role of azides was not only to act as nucleophiles and leaving groups, but also to accelerate the reaction rate in the presence of CuI, in contrast to reactions conducted with CuI alone as previously reported. The unique mechanistic pathway was examined in detail.

Keywords: azide; chalcone; copper iodide; flavone

Introduction

Flavones are a subfamily of flavonoids derived from natural sources and are known for their broad

spectrum of biological activities, including antioxidant, anti-inflammatory, and anticancer effects [1]. Chalcones also serve as versatile intermediates in the synthesis of various flavonoid derivatives, such as flavones [1,2], flavonols [3], flavanones [4], and aurones [1,5] (Figure 1). Several synthetic routes for flavones have been reported, including the Allan-Robinson reaction, Baker-Venkataraman reaction, Kostanecki reaction, and Mentzer reaction, among others [6]. In addition, a number of synthetic methods for flavones employ chalcones as starting materials, for example using $SiO_2/InBr_3$, $InCl_3$ [7], ICl/ultrasonic [8], CuI catalysts/ionic solvents [9], NBS/MeOH [10], RSSR/heat [11], $SiO_2/Ce(SO_4)_2 \cdot 4H_2O$ [12], hv/*N*-oxide [13], oxalic acid [14], $POCl_3$ [15], and I_2/DMSO [16]. Among these methods, the use of I_2 in DMSO has been the most frequently employed in our laboratory for the preparation of synthetic flavones [17].

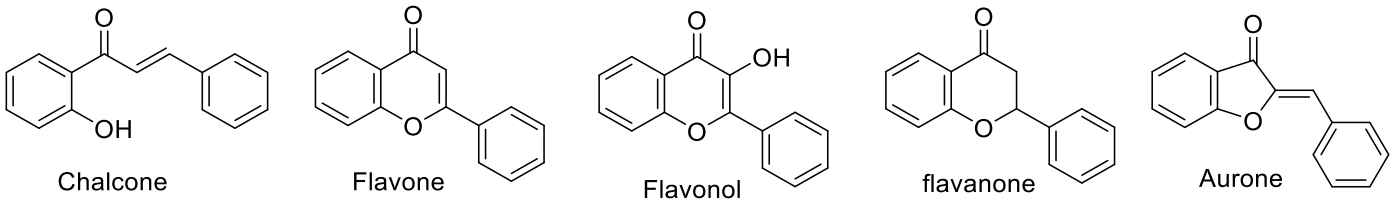


Figure 1. Representative structures of chalcone, flavone, flavonol, flavanone, and aurone

Nitrogen-containing heterocyclic compounds, whether derived from natural sources or synthesized artificially, are of significant pharmaceutical interest for the development of anticancer agents [18]. Among them, azoles are a prominent class of aromatic compounds characterized by a five-membered ring containing at least two heteroatoms, one of which is invariably a nitrogen atom [19-21] (Figure 2). Compounds with azole moiety were widely used as antifungal agents [21].

Figure 2. Selected azole derivatives

1,2,3-Triazoles possess three isomers [22] (Figure 3). There are several methods to approach the synthesis of them [23,24]. Among these strategies, the copper-catalyzed azide-alkyne cyclization (CuAAC reaction) is the most famous one [25,26].

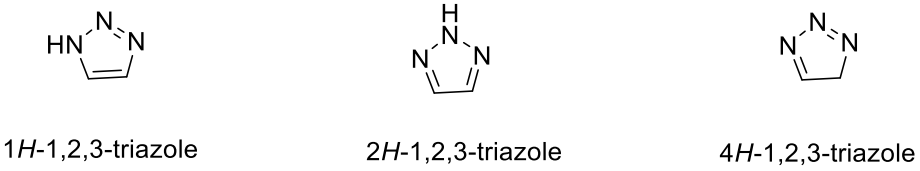


Figure 3. Isomers of 1,2,3-triazoles

It has been reported that chalcones **1** was treated with sodium azide catalyzed by CuO to yield the 1,2,3-triazole derivatives **2** (Figure 4) [27].

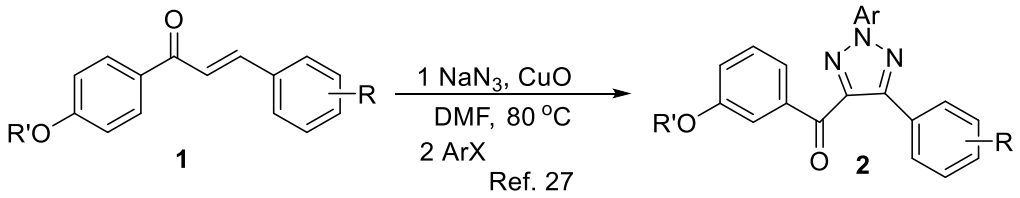


Figure 4. Literature report [27] in synthesis of 1,2,3-triazoles from chalcones using NaN_3 and CuO

In our recent study, we attempted to synthesize 1,2,3-triazole derivatives by reacting chalcones bearing a hydroxy group at the C2 position of the A-ring (Figure 5, compounds **3a**-**j**) with sodium azide or 4-azidobenzenesulfonamide in the presence of CuI, either in catalytic or stoichiometric amounts. Unexpectedly, flavones **4a**-**j** were obtained instead of the anticipated triazole products (compounds **5a**-**j** or **6a**-**j**). Preliminary trials using CuO [27] in place of CuI gave inconsistent and messy results, as indicated by

TLC. These findings suggest that an alternative reaction pathway is favored under the given conditions. Without the addition of CuI, the expected flavones were obtained only in low yields or no reaction occurred, depending on the azides employed (*vide infra*, Table 1).

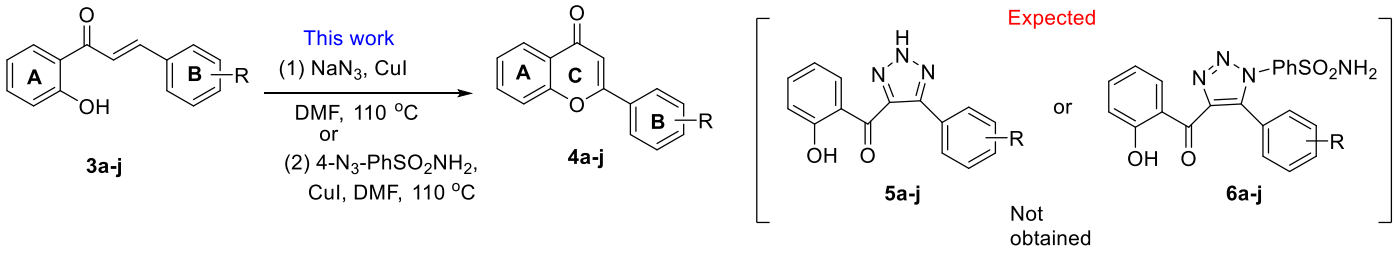


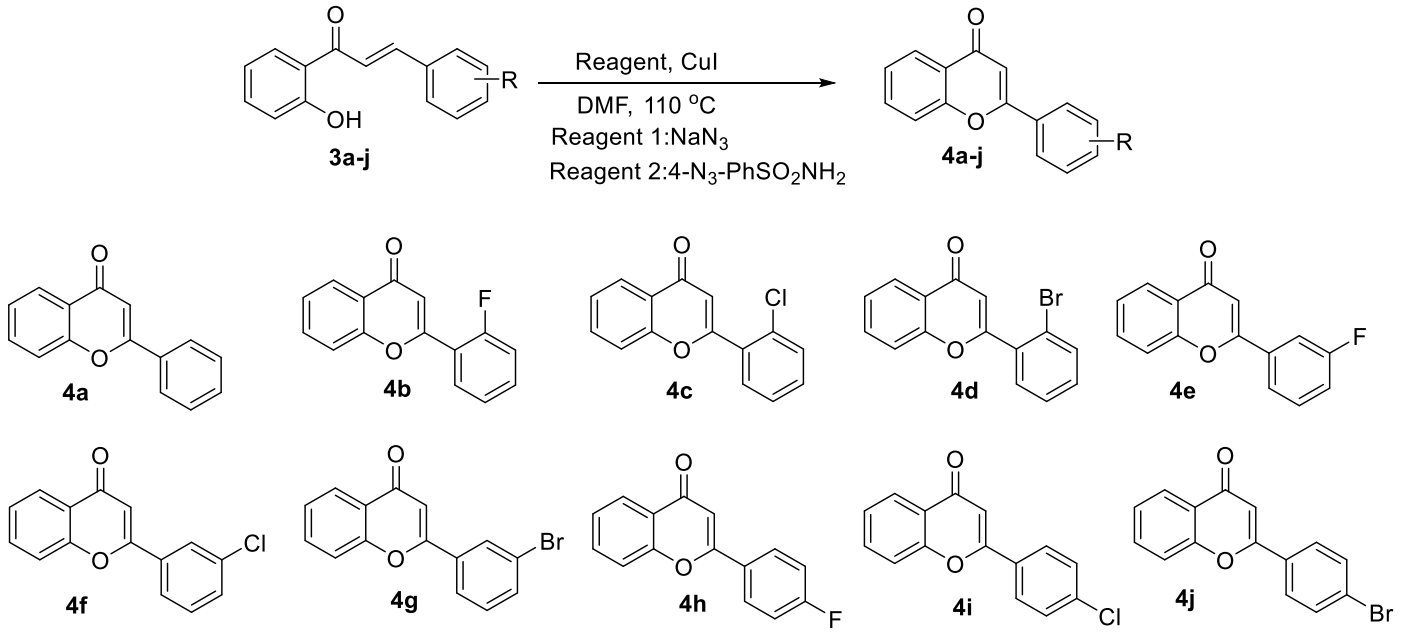
Figure 5. Synthesis of flavones (**4a-j**) from chalcones (**3a-j**) with NaN_3 or 4-azidobenzenesulfonamide via CuI-mediated reaction in this study.

Rather than the common method, such as I_2 in DMSO, we used to use in the synthesis of flavones from chalcones, CuI mediated azides method reported herein is unprecedent. To explore the potential an alternative method in synthesis flavones from chalcones mediated by CuI and azides, the detailed mechanisms are discussed.

Results and discussion

To evaluate the roles of CuI and azides in the synthesis of flavones from chalcones, chalcones **3a-j** were employed as starting materials [3] (Scheme 1). Therefore, six experimental conditions were investigated: (1) NaN_3 (1.0 equivalent) and without CuI; (2) NaN_3 (1.0 equivalent) with CuI (10 mol%); (3) NaN_3 (1.0 equivalent) with CuI (1.0 equivalent); (4) 4-azidobenzenesulfonamide (1.0 equivalent) and without CuI; (5) 4-azidobenzenesulfonamide with CuI (10 mol%); (6) 4-azidobenzenesulfonamide (1.0 equivalent) with CuI (1.0 equivalent). The concentration of reaction mixture is 0.20 M (0.20 mmol of chalcones **3a-j** in 1.0 mL DMF) and heated at 110 °C with the same reaction temperature as described in literature [9]. These results are

summarized in Table 1.



Scheme 1. CuI-mediated azide synthesis of flavones **4a-j** from chalcones **3a-j**

Table 1. Conditions and yields of azides mediated by CuI in synthesis of flavones **4a-j** from chalcones **3a-j**

Compound	Condition a		Condition b		Condition c		Condition d		Condition e		Condition f	
	Yield	Time (h)	Yield	Time (h)	Yield	Time (h)	Yield	Time (h)	Yield	Time (h)	Yield	Time (h)
4a	6%	72	58%	1	79%	1	ND	72	67%	20	79%	11
4b	9%	72	25%	1	44%	1	ND	72	67%	20	68%	10
4c	4%	72	32%	1.5	50%	1	ND	72	50%	22	57%	7
4d	9%	72	28%	1.3	35%	1	ND	72	32%	24	61%	10
4e	7%	72	17%	1	58%	1	ND	72	63%	20	69%	12
4f	9%	72	25%	1	55%	1	ND	72	61%	20	57%	7
4g	4%	72	26%	1	42%	1	ND	72	68%	22	63%	9
4h	6%	72	29%	1	59%	1.25	ND	72	63%	20	57%	13
4i	3%	72	39%	1	65%	1	ND	72	66%	20	65%	13
4j	6%	72	35%	3	67%	1.8	ND	72	60%	20	75%	10

Condition a: NaN_3 (1.0 equivalent), DMF, 110 °C.

Condition b: NaN_3 (1.0 equivalent), CuI (10 mol%), DMF, 110 °C.

Condition c: NaN_3 (1.0 equivalent), CuI (100 mol%), DMF, 110 °C.

Condition d: 4-N_3-PhSO_2NH_2 (1.0 equivalent), DMF, 110 °C.

Condition e: 4-N_3-PhSO_2NH_2 (1.0 equivalent), CuI (10 mol%), DMF, 110 °C.

Condition f: 4-N_3-PhSO_2NH_2 (1.0 equivalent), CuI (100 mol%), DMF, 110 °C

ND: not determined and starting material recovered

A similar work done by Du and co-workers reported a CuI-catalyzed synthesis of flavones from chalcones in ionic liquids or various solvents under air or O_2 (1 atm) in 2011 [9]. They proposed three possible mechanistic pathways (Figure 6). The optimized yield of compound **4a** (96%) was achieved in [bmim][NTf_2] using 20 mol% CuI after 24 h. When 10 mol% CuI was used in the same ionic liquid, compound **4a** was obtained in 92% yield after 48 h, indicating that increasing the CuI loading reduced the reaction time from 48 h to 24 h while maintaining nearly equivalent yields. In contrast, when the reaction was carried out with 100 mol% CuI in DMF under air at 130 °C for 3 h, compound **4a** was produced in 69% yield. Thus, although higher CuI loading and elevated temperature significantly shortened the reaction time, the yield decreased when the ionic liquid was not employed. Du's isotope-labeling experiments suggested that mechanistic pathways b and c were the most likely routes (Figure 6).

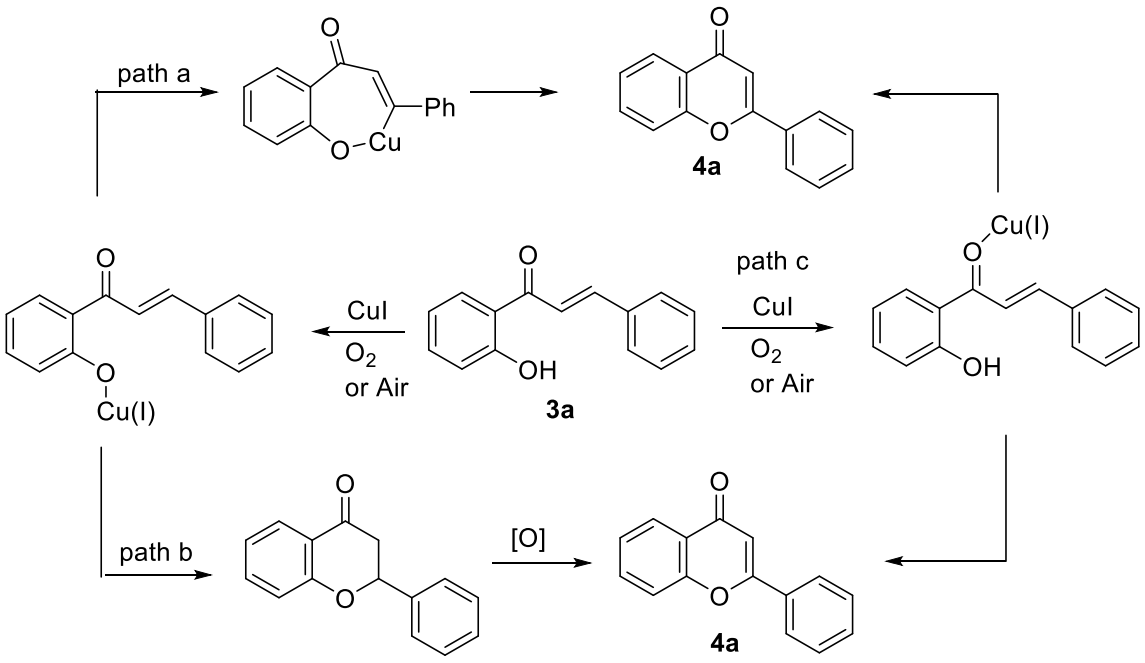


Figure 6. The proposed mechanisms by Du's coworkers in synthesis of flavone from flavone via CuI in air

or O_2 (1 atm) or ionic liquid

The proposed mechanisms in synthesis of compounds **4a-j** are illustrated in Figures 7 for sodium azide and Figure 8 for 4-azidobenzenesulfonamide, respectively. Each following similar pathways; however, the yields and reaction times varied depending on whether without CuI and either catalytic or stoichiometric amounts of CuI were employed. In the absence of CuI, with only sodium azide present, flavones were obtained in trace amounts (3%-9%) and most chalcones were recovered after 72 h (Table 1, condition a). The reaction proceeded very sluggishly, likely due to intramolecular hydrogen bonding in compounds **7a-j**, which retarded the formation of **8a-j** that led to intramolecularly attacking the β position to form **4a-j** (Figure 7, path a).

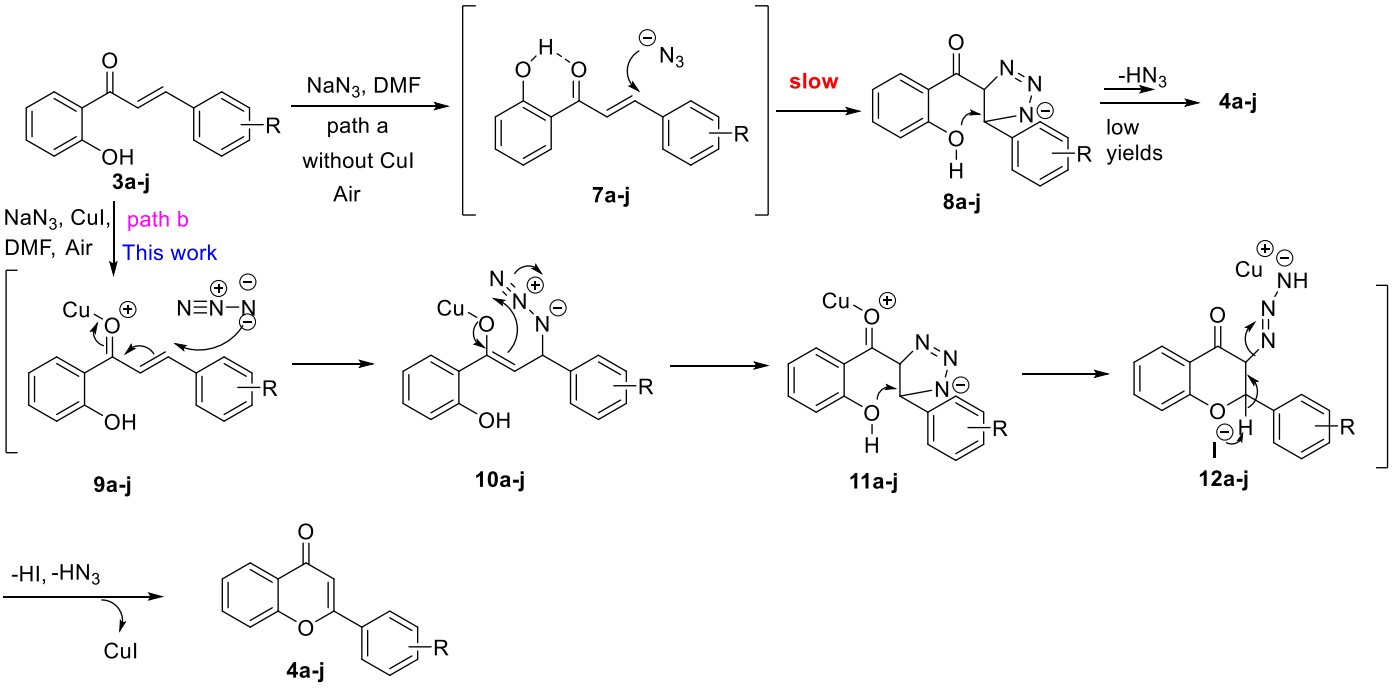


Figure 7. Plausible mechanisms of chalcones **3a-j** react with NaN_3 without (path a) or with (path b) CuI in formation of flavones **4a-j** at 110 °C.

When catalytic or stoichiometric amounts of CuI were present with sodium azide, Cu(I) first coordinated to the carbonyl group to form **9a-j**. The nucleophilic nitrogen of N_3^- then underwent Michael addition to afford **10a-j**. Subsequently, the enolate attacked the terminal nitrogen, generating intermediates **11a-j**. The hydroxy

group then attacked the *β*-position to form **12a-j**. Finally, *β*-elimination of **12a-j** yielded the corresponding flavones **4a-j** (Figure 7, path b). The role of CuI is crucial, as it facilitates the formation of 1,2,3-triazole intermediates **11a-j** from azide and promotes intramolecular attack by the hydroxy group to yield **4a-j**.

When 4-azidobenzenesulfonamide was used as a nucleophile to react with **3a-j**, the reaction did not proceed without CuI assistance, as intramolecular hydrogen bonding hindered the process due to the weak nucleophilicity of 4-azidobenzenesulfonamide (Figure 8, path a, **13a-j →6a-j**). In the presence of CuI, either in catalytic or stoichiometric amounts to interrupt the intramolecular hydrogen bonding, the reaction first underwent Michael addition to the chalcones while Cu(I) coordinated to the carbonyl group (**3a-j →14a-j**). This was followed by intramolecular attack at the terminal nitrogen (**15a-j**), generating a 1,2,3-triazole-like intermediate (**16a-j**). Subsequently, the hydroxy group attacked the *β*-position to form **17a-j**, and iodide-assisted elimination of Cu(I)/4-azidobenzenesulfonamide yielded the corresponding flavones **4a-j** along with recovery of 4-azidobenzenesulfonamide (Figure 8, path b). The recovery of 4-azidobenzenesulfonamide strongly supports the proposed mechanism.

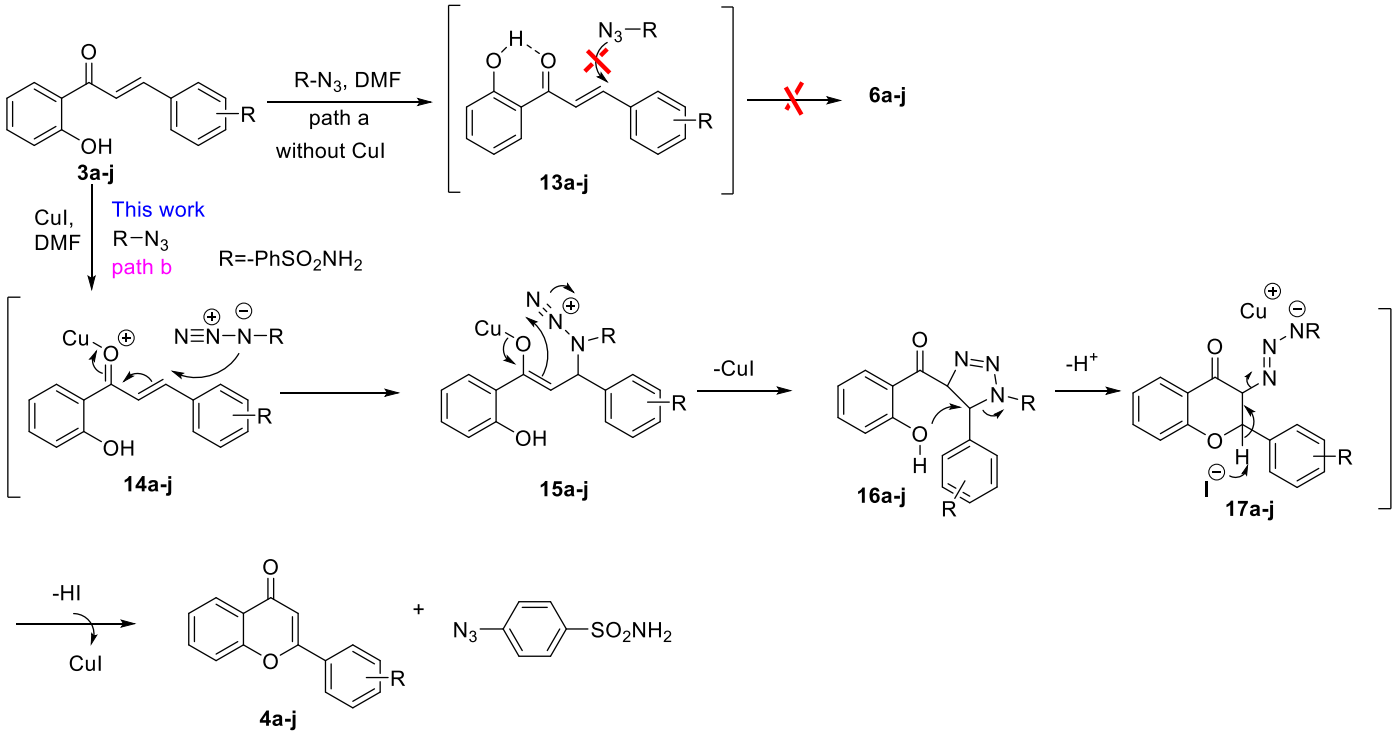


Figure 8. Plausible mechanisms of chalcones **3a-j** react with 4-azidonbenzenesulfonamide with or without CuI in formation of flavones **4a-j**

Table 1 addressed two very important issues: (1) the azide must undergo Michael addition, followed by intramolecular attack by the hydroxyl group and subsequent elimination; (2) the addition of CuI was essential, whether in catalytic or stoichiometric amounts. Notably, the use of a stoichiometric amount of CuI enhanced the yields and reduced the reaction time.

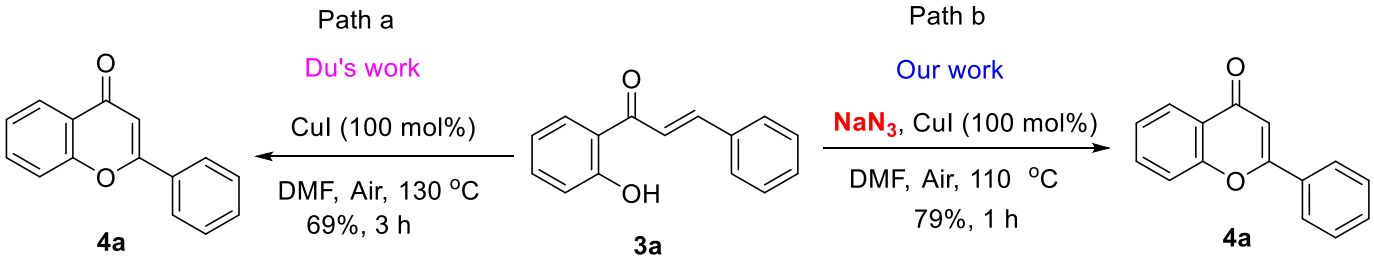
We proposed that Cu(I) most likely coordinates first with the carbonyl group (Figure 7, path b), increasing the electrophilicity of the *β*-position and thereby facilitating the Michael addition step. When CuI (10 mol%) was employed, sodium azide reacted more rapidly but furnished lower yields (Table 1, condition b). In contrast, full coordination of the carbonyl group by a stoichiometric amount of CuI stabilized the transition state and afforded higher yields (Table 1, condition c). Without the assistance of CuI, 4-azidobenzenesulfonamide did not undergo any reaction for 72 h (Table 1, condition d). However, when CuI

(10 mol%) was introduced, the reaction proceeded, albeit requiring a longer time and affording only moderate yields (Table 1, condition e). When stoichiometric CuI was employed, 4-azidobenzenesulfonamide (Table 1, condition f) afforded comparable yields with using NaN_3 and the reaction proceeded slightly faster.

These behaviors can be rationalized by considering both nucleophilicity and leaving-group ability of respective azides. In this study, sodium azide acts as a stronger nucleophile because it exists as a free anion with a high negative charge density, which explains its faster reaction in the presence of either catalytic or stoichiometric amounts of CuI. In contrast, 4-azidobenzenesulfonamide is a weaker nucleophile due to its neutral character, bulky structure, and the strong electron-withdrawing inductive effect of the sulfonamide group, resulting in a slower reaction. Thus, full coordination of Cu(I) with the carbonyl group plays a significant role in stabilizing the transition state. Moreover, 4-azidobenzenesulfonamide, compared to azide, provides a neutral and bulky substituent that serves as an effective leaving group, ultimately furnishing better yields.

Another issue that arises is whether azides play a critical role during the course in synthesis of flavones. According to Du's work, CuI was employed as the sole catalyst (100 mol%) for the synthesis of flavones from chalcones in DMF at 130 °C for 3 h, affording compound **4a** in 69% yield. (Scheme 2, Path a). Our method demonstrated that, in the presence of CuI (100 mol%) and NaN_3 in DMF, the reaction proceeded in a shorter time (1 h), at a lower temperature (110 ^oC), and with a higher yield (79%), thereby underscoring the advantage of our approach (Scheme 2, Path b). On the other hand, the yields of condition b versus condition e, and condition c versus condition f in Table 1 should be comparable if azides did not participate in the Michael addition. However, the present results indicated that they differed significantly-not only in yields but also in

reaction time. When Cu(I) coordinated with the carbonyl group, the *β*-carbon became more electrophilic, thereby accelerating the reaction with both azides, which are more nucleophilic than the hydroxyl group of A ring. This observation supports our proposed mechanism, which aligns with pathway c-a route not suggested by Du. Consequently, azides must participate in the initial formation of 1,2,3-triazole intermediates; however, these intermediates are not isolated because they undergo subsequent intramolecular attack by the hydroxy group at the C2 position of the A-ring of chalcones, ultimately leading to flavone formation.



Scheme 2. A comparison of the synthesis of compound **4a**, either with the assistance of NaN_3 or without, by CuI (100 mol%) highlights the differences between Du's work and our own.

Conclusion

Sodium azide (N_3^-) ions are commonly used as nucleophiles in substitution reactions and are seldom employed as leaving groups [28]. However, in our study, azides participate in a Michael addition to form intermediates **9a-j →11a-j**, which subsequently undergoes elimination to generate flavones (Figure 7, path b). This reactivity differs from previously reported procedures in which sodium azide reacts with chalcones in which there were no 2-hydroxy group at their A rings to form 1,2,3-triazoles [27]. In addition, 4-azidobenzenesulfonamide followed a similar reaction mechanism to NaN_3 used. In those cases, strong intramolecular hydrogen bonding prevented the Michael addition pathway without presence of CuI (Figures

7 and 8, path a). In contrast, under the present conditions, copper(I) plays a crucial role by coordinating with the carbonyl group and disrupting the hydrogen-bonding network, thereby accelerating the reaction. The involvement of azides in the presence of CuI leads to a distinct mechanism not supported by Du. Importantly, in this transformation, azides do not behave as traditional nucleophiles; instead, they act as reactive intermediates that drive flavone formation. Our study provides compelling evidence to support path c (Figure 6) when azides are present. Furthermore, the hydroxy group at the C2 position of the A ring is essential for intramolecular attack during cyclization.

In summary, Du previously demonstrated that CuI (10 mol%) in an ionic liquid under oxygen (1 atm) at 50 °C afforded compound **4a** in 92% yield after 48 h. In contrast, our study employed the same loading of CuI but, with the assistance of NaN_3 in DMF at 110 °C, delivered **4a** in 58% yield within only 1 h.

Experimental

All chemicals were purchased from Acros and Alfa of Uni-Onward Corp. in Taiwan and used without further purification except otherwise mentioned. The reaction progress was monitored by thin layer chromatography (Macherey-Nagel DC-Fertigplatter Durasil-25UV plates) eluted with designated solvents, visualized by UV light (254 /365 nm), and stained with $KMnO_4$ or *p*-anisaldehyde solutions. Purification was conducted by flash column chromatography (SiO_2, 230-400 mesh, SiliaFlash). The melting points were measured on Fargo MP-2D without correction with open capillary tubes. ^1H NMR and ^{13}C NMR spectroscopic data were recorded on Bruker UltraShield 600 MHz or Bruker advance 300 MHz instruments. The chemical shifts were reported in part per million (ppm) relative to the residual solvent: $CDCl_3$, ^1H NMR (300 MHz and

600 MHz), 7.26 ppm; ^{13}C NMR (75 MHz or 150 MHz), 77.0 ppm. The high-resolution mass spectrometry (HRMS) data was recorded on Orbitrap Fusion Lumos Tribrid mass spectrometer (ThermoFisher Scientific) equipped with a heated electrospray ionization source (HESI).

General procedure in synthesis of flavones 4a-j

Sodium azide (0.20 mmol) or 4-azidobenzenesulfonamide (0.20 mmol) was dissolved in DMF (1.0 mL), followed by the addition of either a catalytic amount of CuI (0.020 mmol) or a stoichiometric amount of CuI (0.20 mmol). The mixtures were heated at 110 °C (sand bath), and the reaction progress was monitored by TLC until completion. After completion, the mixture was filtered through celite, and the residue was washed with Et_2O. The combined filtrate was diluted with H_2O, and the organic layer was separated, dried over $MgSO_4$, concentrated, and purified by flash column chromatography (EtOAc:hexane = 1:40→1:30→1:20→1:10→1:1) to give the target compounds.

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Conflict of interest

The authors declare no conflict of interest.

Author Contributions

Sheng-Xiang Chen: data curation; formal analysis; investigation; validation. Yu-Jui Su: data curation; formal analysis; investigation; validation. Hsin-Ying Wu: data curation; formal analysis; investigation; Sy-Chyi Cheng: data curation. Tzenge-Lien Shih: conceptualization, writing, funding acquisition, investigation; project

administration; resources; supervision

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Supporting Information

Products characterization data and the spectroscopic data of ^{1}H and ^{13}C NMR for compounds **4a-j**.

Data Availability Statement

All data supporting the finding of this study are available in the article and its Supporting Information.

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