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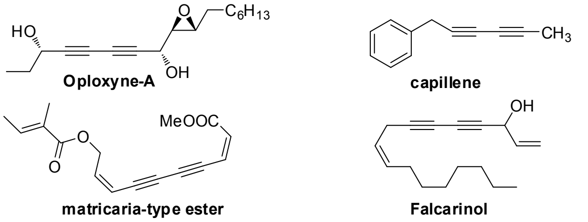
Visible-light-activated Copper(I) catalyzed Oxidative C_{sp}-C_{sp} Cross-Coupling Reaction: Efficient Synthesis of Unsymmetrical Conjugated Diynes without Ligands and Base

Arunachalam Sagadevan,[1] Ping-Chiang Lyu,[2] and Kuo Chu Hwang[1]*

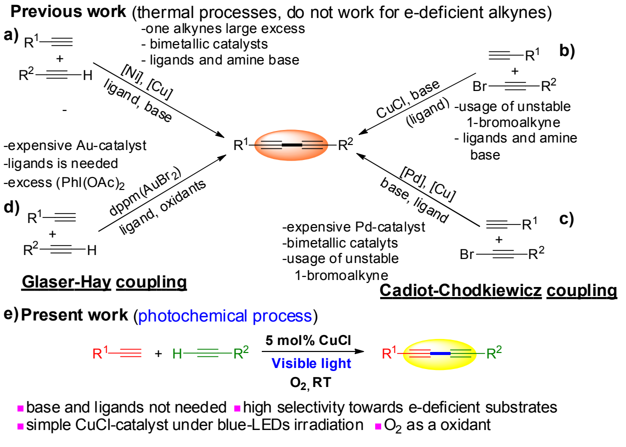
A novel visible-light-promoted copper-catalysed process for C_{sp}-C_{sp} cross-coupling reaction of terminal alkynes at room temperature is described. The current photochemical method is simple, high functional group compatible, and more viable towards the construction of bio-active 1,3-unsymmetrical conjugated diynes without the need of bases/ligands, additives and expensive palladium/gold catalysts.

The development of new and mild synthetic approaches for the construction of unsymmetrical 1,3-conjugated diynes and polyynes is of great importance in synthetic chemistry. The scaffold of 1,3-conjugated diynes is a valuable building block in various natural products, pharmaceuticals (see, Scheme **1**), organic materials and supramolecular materials.[1] Glaser-Hay coupling[2] and Cadiot-Chodkiewicz coupling[3] are two of the earliest and the most remarkable methods for preparation of conjugated diynes. However, these processes require toxic organic amine bases and excess of alkynes. In addition, Cadiot-Chodkiewicz coupling (see, Scheme **2b**) involving pre-functionalized alkyne substrates (e.g., unstable 1-haloalkyne), which is the leads to increase the overall cost for the synthesis of unsymmetrical diynes.[4]



Scheme 1 Unsymmetrical 1,3-diynes in natural products.

Recently, it was reported that C_{sp}-C_{sp} cross-coupling reaction can be achieved with 1-bromoalkyne using an expensive Cu/Pd bimetallic catalyst with or without air sensitive phosphorous ligands (Scheme **2c**).[5] From an environmental and economic viewpoint, direct coupling of two terminal alkynes is greener and more cost-effective.[6] Cu/Ni bimetallic catalytic system have shown to facilitate oxidative coupling of two terminal alkynes in the presence of organic base and ligands with usage of large excess of one alkynes (5.0 eqv)(see, Scheme **2a**).[6a] Very recently, gold catalysed selective heterocoupling of terminal alkynes for the synthesis of 1,3-conjugated diynes in presence of ligands and oxidants (see, Scheme **2d**).[6b] Despite their increasing attention, the method is still limited to cross-coupling reaction of alkyl-aryl alkynes, moreover, this process requires the use of expensive gold-catalyst along with excess oxidants ($PhI(OAc)_2$) and ligand (phenanthroline), thus make the overall cost and is accompanied with the formation of undesired side products/ wastes.



Scheme 2 Transition metal-catalyzed oxidative cross-coupling of alkynes.

Despite their own uniqueness and advantages, all reported processes[6] share common limitations, such as: (a) all processes work well only for e-rich/neutral aryl/aliphatic alkynes; (b)

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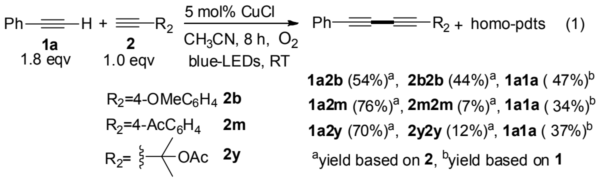
require toxic amine base and ligands along with bimetallic catalyst, which leads to environmental issues on the synthesis of 1,3-conjugated diynes (large scale). Therefore, it is highly desirable to develop simple, inexpensive catalytic system (e.g., Cu) for an effective C_{sp}-C_{sp} cross-coupling reaction of two different terminal alkynes under low energy visible light irradiation with absence of expensive catalyst (Pd or Au), ligands and base is great importance in organic synthesis.

Visible light-activated photoredox catalysis (Ru- or Ir-complexes) has emerged as a new reaction modality for organic reactions.[7] Photoredox copper catalysis has been demonstrated to be extremely successful in various coupling reactions, such as, alkyne-azide cycloaddition (CuAAC) reactions,[8] C-C cross-coupling,[9] and various C-N, C-S and C-O cross-coupling reactions.[10] We have previously developed a visible light-activated Cu-catalyzed process for efficient C-C, C-N cross-coupling, and C-H annulation reactions.[11] Herein, we report simple Cu-catalyzed direct C_{sp}-C_{sp} cross-coupling reaction of terminal alkynes between e-rich and e-poor aryl alkynes under blue-LEDs light irradiation at room temperature. The reactions proceeded with high yields (64-95%) and good selectivity without the needs of expensive Pd, ligands, and bases. The significance of the present chemistry includes: 1) an unprecedented visible-light-induced copper-catalysed C_{sp}-C_{sp} cross coupling reaction of terminal alkynes for rapidly accessing a variety of unsymmetrical 1,3-conjugated diynes at room temperature; 2) the current photochemical method does not require the use of precious expensive metal catalyst (e.g., Au-, Pd-catalyst), external oxidants/additives, ligands and base (see, Scheme **2**); 3) This is the first literature example that direct aerobic oxidative coupling of two different terminal alkynes for construction of unsymmetrical 1,3-conjugated diynes under visible light induced copper-catalysed process.

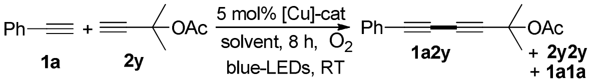
Our initial study was focused on coupling of phenylacetylene (**1a**) with 4-OMe-phenylacetylene (**2b**) in acetonitrile in the presence of O_2 and the absence of any base under blue-LEDs irradiation. The reaction yields 54% of the desired heterocoupling product along with a mixture of homocoupling products [see eqn**.** (**1**)]. To our delight, the C_{sp}-C_{sp} hetero-coupling products, **1a2m** and **1a2y**, were obtained in good yields (76 and 70%, respectively) when electron-deficient aliphatic alkynes (**2m** and **2y**) were used as substrates [see eqn**.(1**)].



After optimization (see Table 1), we found that acetonitrile is the best solvent (85% yield) for the cross-coupling reactions; other polar solvents, such as, MeOH, DMF, and THF, also work well (entries 5-7, Table 1). Non-polar solvents, such as, toluene, fail to produce the heterocoupling product (see entry 8, Table 1). In addition, we found that low substrate concentrations can significantly improve the yield of hetero-coupling product **1a2y** to 85%, and suppress the homo-coupling product **2y2y** to 11% (see entry **1,** Table 1). However, cross-coupling reaction of 1:1.2 ratio of two different alkynes provides mixture of hetero and homocoupling products (entry **4**, Table **1**). Upon optimization, an excess one alkyne (up to 1.8 eqv.) is necessary to achieve good selectivity (**1a2y**) (entry **1**, Table **1**) for the heterocoupling product. In the screening of catalysts, CuX (X: Cl, Br) affords the highest yield of the hetero coupling product. However, $CuCl_2$ fails to catalyze the formation of the desired cross-coupling product (entries 1-3, Table 1). It is worthy to note that the formation of the homocoupling product (entry **4, Table 1**) increases at higher substrate (2y) concentrations. Meanwhile, control experiments also reveal that exclusion of either light, CuCl or O_2 results in no product formation (entries 10-12, **Table 1**).

Under the optimized condition (entry 1, Table 1), phenylacetylene (**1a**) was used as one of the partner for coupling with all substrates (see results in Table **2**). In the optimized condition, the reaction does not work as efficiently for electron rich aromatic acetylenes (**2a – 2e**) (as compared to electron deficient aryl alkynes) with lower yields of the desired cross-coupled products (**1a2a-1a2e**). In the case of halo-substituted aryl acetylenes, F, Cl, and I react smoothly to generate the corresponding cross-coupling products in good to excellent yield 73 to 88% (**1a2f-1a2h)**. The presence of these halogen functionalities is useful for further synthetic modifications.[12] Other aromatic alkynes (**2i-2k**), including electron-donating group at the meta position, can undergo cross-coupling reaction to generate the desired products in good yields (**1a2i-1a2k**).

Table 1 Optimization of reaction condition.[a]



Entry	[Cu] Catalyst	Solvent	conversion [%][b]	yield (%)		
				1a2y[c]	**2y2y**[c]	**1a1a**[d]
1	CuCl	CH_3CN	96	85	11	48
2	CuBr	CH_3CN	95	85	10	48
3	$CuCl_2$	CH_3CN	10	-	-	trace
4[e]	CuCl	CH_3CN	98	70	28	56
5	CuCl	CH_3OH	76	64	12	44
6	CuCl	DMF	86	72	14	43
7	CuCl	THF	68	42	26	30
8	CuCl	toluene	24	20	trace	21
9[f]	CuCl	CH_3CN	82	75	7	42
10[g]	CuCl	CH_3CN	trace	-	trace	12
11[h]	CuCl	CH_3CN	n.r	-	-	-
12[i]	CuCl	CH_3CN	trace	-	trace	-

[a] Unless otherwise mentioned, reaction conditions are as follows; 0.6 M of **1a** (1.2 mmol), 0.35 M of **2y (**0.7 mmol**)**, 5 mol% of [Cu] catalyst. The mixture was irradiated with blue LEDs (power density: 40 mW/cm^2 at 460 nm) for 8 h in an O_2 atmosphere (1 atm.). [b] Total conversion of the isolated product **1a2y** based on **2y**. [c] Yield of the isolated product **1a2y** based on **2y.** [d] Yield of the homocoupling product **1a1a** based on **1a.** [e] Reaction condition: 0.6 M (1.2 mmol) of **1a**, 0.5 M (1 mmol) of **2a**. [f] irradiation with an ambient white light bulb for 15 h (power density: 8 mW/cm^2 at 460 nm). [g] Reaction

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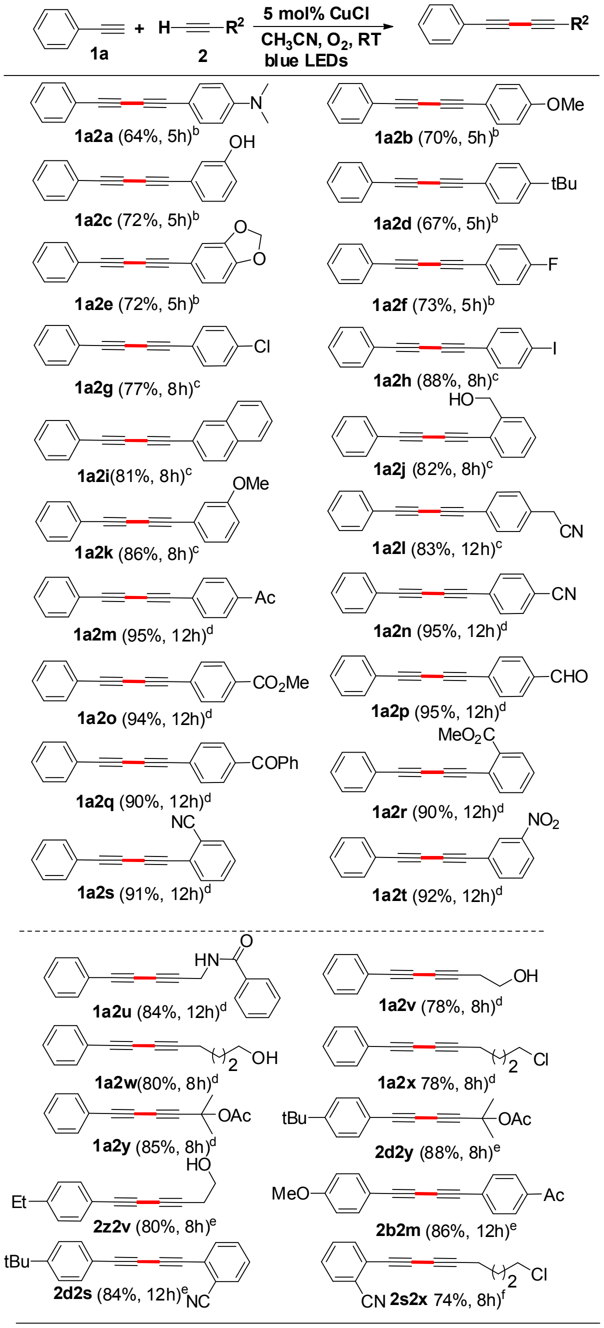
was conducted in the dark at RT. [h]In the absence of CuCl catalyst. [i]In the absence of O_2.

Table 2 Substrates scope[a]

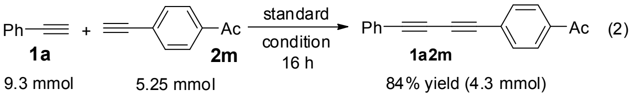


[a]Standard condition. Isolated yield based on **2** after purification by column chromatography on silica gel, [b]reaction condition: 1.0 mmol of **1a** (0.5 M), 0.8 mmol of **2** (0.4 M), [c]reaction condition: 1.2 mmol of **1a** (0.6 M), 0.7 mmol of **2g-2l** (0.35 M). [d]reaction condition: 1.3 mmol of **1a** (0.65 M), 0.7 mmol of **2m-2y** (0.35 M). [e]reaction condition: 1.3 mmol of **2b, 2d & 2z** (0.65 M), 0.7 mmol of **2y, 2v, 2m & 2s** (0.35 M). [f]reaction condition: 0.7 mmol of **2s** (0.65 M), 1.0 mmol of **2x** (0. 5 M).

In general, the C_{sp}-C_{sp} cross coupling between electron deficient aromatic terminal alkynes and phenylacetylene is very difficult to accomplish, since these groups have less π–basicity on the C≡C triple bond, and thus is less favorable towards soft Lewis acid catalysis, such as, Cu(I) ion.[13] However, the present catalysis system is effective for cross-coupling to generate the unsymmetrical 1,3-diynes in 83-95% yields (**1a2l-1a2t**). Thus, this visible light induced cross coupling protocol is very important and complementary to the literature reported thermal C_{sp}-C_{sp} coupling reaction.[5,6] It could be understood that the acetylene proton in terminal alkynes having electron deficient substituent groups is more acidic, and can be easily abstracted by photo-generated radical species, such as, superoxide radical anion (for details, please see the proposed mechanism below). Therefore, the C_{sp}-C_{sp} cross-coupling reaction between electron-rich/neutral and electron-poor aryl alkynes occurs upon photo-irradiation.

Moreover, aliphatic alkynes, such as, propargylamine derivative and linear alkynes, can also be effectively coupled with **1a** to generate desired unsymmetrical 1,3-diynes (**1a2u-1a2y).** In addition, electron rich aromatic groups (e.g., R= tBu & Et) can also effectively couple with aliphatic alkynes to generate the desired products in good yields (e.g., **2d2y & 2z2v**). In contrast to reported thermal methods, the current photochemical process works well for cross-coupling reaction between electron-rich and electron-deficient aryl alkynes to afford hetero-coupling 1,3-diynes products in good yields (e.g., **2b2m & 2d2m**). Interestingly, an electron-poor aryl alkyne (**2s**) can also effectively couple with aliphatic alkynes to generate the desired cross-coupling products in good yield (e.g., **2s2x**).

Table 3 Evaluation of green chemistry metrics for the synthesis of 1,3 diynes (**1a2m**)



Reactant A	phenylacetylene (**1a**)	0.95g	0.0093 mol	FW 102.05
Reactant B	1-(4-ethynylphenyl)ethanone (**2m**)	0.75g	0.0052 mol	FW 144.06
Solvent	ACN	11.79g	---	---
Auxiliary	---	---	---	---
Product	1,3-diynes (**1a2m**)	1.06g	0.0043 mol	FW 244.09

Product yield= 84%

$$\text{E-factor} = \frac{0.95\text{g} + 0.75\text{g} + 11.79\text{ g} - 1.06\text{g}}{1.06\text{ g}} = 11.7 \text{ kg waste/ 1 kg product}$$

$$\text{Atom economy} = \frac{244}{262} \text{ X } 100 = 93\%$$

$$\text{Atom efficiency} = 84\ \% \text{ X } 93\% / 100 = 78\%$$

$$\text{Carbon efficiency} = \frac{18}{8 + 10} \text{ X } 100 = 100\%$$

$$\text{Reaction mass efficiency} = \frac{1.06\text{ g}}{0.95\text{g} + 0.75\text{g}} \text{ X } 100 = 63\%$$

In addition, the current C_{sp}-C_{sp} cross coupling process can be readily scaled up to a gram scale (0.75 g, 5.25 mmol); 1.06 grams of **1a2m** (84% yield) can be obtained after 16 hours of irradiation with blue LEDs at room temperature (see eqn (2)). We have evaluated the green chemistry metrics[11c] for the synthesis of the unsymmetrical conjugated 1,3-diynes (**1a2m**) on a preparative scale (see Table 3). Overall, our green process can enable the 1,3-diynes (**1a2m**) on the preparative scale with an E factor of 11.7, 93% atom economy, 78% atom efficiency, 100% carbon efficiency, and 63% reaction mass

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efficiency, which are far better than previously reported process[6a] (E factor= 27, atom economy= 56%, atom efficiency= 48%, reaction mass efficiency: 27%, see, details in S.I.), since the current photochemical process does not require the any base/ligands and expensive metal catalyst and thus leads to a sustainable and green process for the synthesis of various bio-active unsymmetrical 1,3-diynes (especially in large scale).

A possible reaction mechanism for the visible light-initiated, CuCl catalysed C_{sp}-C_{sp} cross coupling reaction is proposed in Scheme **2**. The first step involves the formation of Cu^{I}-π–alkyne complex (**A**), of which the alkynes terminal proton becomes more acidic[13] and thus favors the in-situ formation of copper(I) phenylacetylide **(B).[14]** In the next step, addition of electron-deficient alkynes to the solution results in formation of a stable, bipolar ground state hetero-dimeric Cu^{I} complex (**C**) in polar organic solvents, as evidenced by isothermal titration calorimetry (ITC) measurements of heat release upon titration of free e-deficient terminal alkynes into a solution containing Cu(I) phenylacetylide (Cu^{I}-**1a**).(see more details in Figure **S3**). From the titration curve, a complexation constant, K_a, of ~ 199 μM^{-1} was obtained for the formation of a ground state bipolar hetero-dimeric Cu(I)-phenylacetylide complex. Such a hetero-dimeric Cu(I) complex contains an electron-rich/neutral and another electron-deficient alkyne, of which the dipolar character is stabilized by a polar solvent. ITC measurements also showed that such a bipolar hetero-dimeic Cu(I) complex was not formed in nonpolar organic solvents (such as, toluene). Similarly, homodimeric Cu(I) complexes do not form, even in polar organic solvents. The formation of the hetero-dimeric Cu(I)-**1a** complex is supported by the observation of a red shift in the absorption maximum of Cu(I)-**1a** from 479 nm to 490 nm in ACN upon addition of equal amount of electron deficient (4-ethynylphenyl) ethanone (**2m**) (see Figure **1**). It is reasonable that formation of the ground state bipolar hetero-dimeric complex is more favourable as compared to homo-dimeric complex in polar organic solvents, since the hetero-dimeric Cu^{I} complex is of dipolar nature (having both electron rich and electron poor moieties in same complex). Direct photo excitation of the complex (**C**) by blue light (λ_{max}= 479 nm) leads to the formation of a long-lived triplet excited hetero-dimeric Cu^{I} complex (**D**) via ligand to metal charge transfer (LMCT).[11c,d&15] The photoexcited Cu^{I}-**1a** complex (**D**) could transfer a single electron to the molecular oxygen to generate Cu^{II}–phenylacetylide complex (**E**) and superoxide,[11c] as evidenced by EPR detection of superoxide radical anion (see Figures S1-S2 in supporting information). Indeed, superoxide radical anion has a tendency to abstract an acidic proton from terminal alkynes (favored by electron withdrawing groups) to form the carbon centered alkynyl radical.[7a,16] Subsequent intra complex ligand-ligand cross coupling between the electron deficient alkynyl radical and electron rich/neutral copper(II) acetylide leads to formation of the desired cross-coupling product,[10a,11d] in accompany with the formation of hydrogen peroxide (as a by-product) and regeneration of CuCl catalyst. Such an intra-complex ligand-ligand cross coupling process dominates over bi-molecular coupling between the alkynyl radical and free alkynes in the solution, especially under low substrate concentration conditions.

Conclusions

We have developed a novel visible-light initiated CuCl catalyzed C_{sp}-C_{sp} cross-coupling of terminal alkynes for the facial synthesis of conjugated diynes without need for a base, ligands, and additives. This protocol can be used to prepare a variety of unsymmetrical conjugated diynes from readily available terminal alkynes without the need of pre-functionalized substrates. Meanwhile, the current process uses an excess one alkyne (up to 1.8 eqv.) to accomplish a good selectivity for the heterocoupling product. The key step toward the success of heterocoupling reaction is the formation of a stable ground state bipolar hetero-dimeric Cu^{I}-acetylide complex in polar organic solvents. Photo excitation provides sufficient energy for the ground state hetero-dimeric Cu^{I}-acetylide complex to overcome an energy barrier, to donate an electron to molecular oxygen, and to initiate the intra-complex hetero-ligands cross-coupling reaction. From synthetic point of view, exclusion of expensive Pd/Au catalysts, ligands, oxidants and amine base makes this method extremely simple and eco-friendly process for synthesis of a variety of bio-active unsymmetrical conjugated diynes.

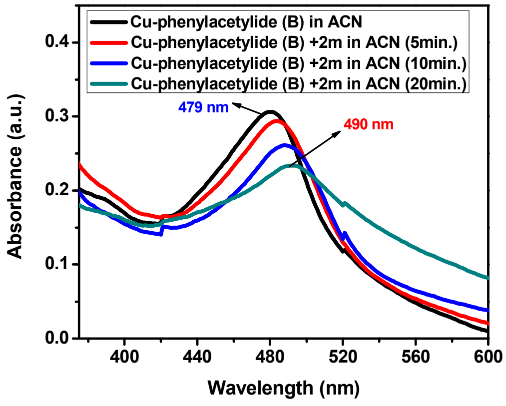
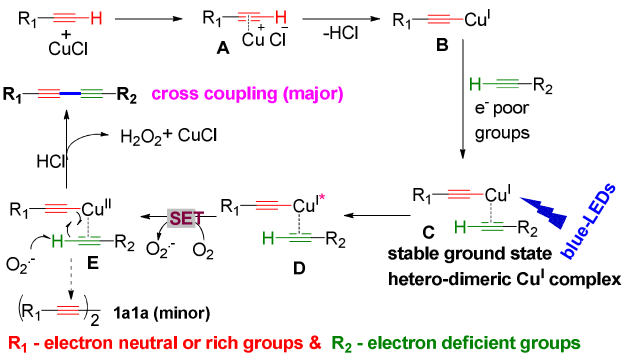


Figure 1 UV-visible spectra of 0.05 M Cu-phenylacetylide (**B**) alone in acetonitrile (ACN) (black curve) and spectra obtained at different time delay after addition of 0.055 M 1-(4-ethynylphenyl) ethanone (**2m**) into the solution.



Scheme 3 Proposed mechanism.

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Graphical Abstract

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An efficient and eco-friendly approach to C_{sp}-C_{sp} cross-coupling of terminal alkynes for construction of unsymmetrical conjugated diynes via visible-light-induced CuCl catalysed process at room temperature.