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Copper complex with N-,O- architecture grafted graphene oxide nanosheet as a heterogeneous catalyst for Suzuki cross coupling reaction

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ABSTRACT

We report a straight forward synthesis of a heterogeneous catalyst by covalently immobilizing copper Schiff base complex on the surface of amino functionalized graphene oxide (AGO) for the Suzuki coupling of substituted aryl halides with arylboronic acids. The as-synthesized complex and subsequent catalyst were characterized for their structural features using suitable techniques. The analysis confirmed that the Cu bound to Schiff base (L) ligand via bi(N-,O-) linkage and Cu-L immobilization on AGO was due to its amino functionality. The catalyst exhibited excellent yield of 94% for Suzuki coupling reactions as analyzed by gas chromatography. The catalyst was recycled for 5 successive reactions with insignificant loss in efficiency. ICP-AES analysis showed the catalyst retained 87.5% of its active metal center after 5th iteration. High yield, environmentally benign, easy work-up procedure, easy separation of catalyst and mild reaction conditions are some of the important facets this catalyst offer.

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1. Introduction

Transition metal catalyzed cross coupling reactions ($C(sp^2)-C(sp^2)$) are an influential method for the synthesis of biaryl compounds [1]. Their applications include a broad range of synthetic target intermediates in manufacture of materials for the synthesis of natural products in the field of medicinal chemistry and pharmaceutical industries [2]. An advantage of Suzuki Coupling reaction over other cross-coupling reactions is its wide accessibility of organoboron compounds *i.e.*, a broad functional group variation and are considerably stable as well as environmentally benign [3]. From its discovery 1989, till now the Suzuki-Miyaura cross-coupling involving the coupling of an organoboron reagent and organic halides or pseudo halides became one among the important methods in the formation of carbon-carbon bonds and also suitable synthetic procedure in organic chemistry, because of the starting materials stability, resistance to various functional groups and suitable to a wide range of solvents [2,4].

Among the various comprehensively well-studied approaches, in order to yield a maximum product with aryl bromides to form carbon-carbon bond, with the improvement of transition metal complexes onto different supports, a viable approach is heterogenization of various carbon supports such as carbon nan-

otube, graphite and fullerenes [5,6]. Graphene, a novel member to graphitic carbon family which consist of 2D nanosheets of sp^2 hybridized carbon atoms and possess fascinating properties such as high thermal and mechanical stability as well as optical properties with an excellent surface area of 2030 m^2/g to become a promising candidate as a catalyst or catalyst support [7].

During the previous years, metal grafted on heterogeneous support has an extensive progress than the homogeneous catalysts due to its reusability, stability and selectivity [8,9]. Copper is a 3d group element which has variable oxidation potential and possess unique properties that helps in tuning the size of Cu nanoparticles [10,11]. Wang et al. reported that Graphene or Graphene oxide (GO) as a solid support for N-heterocyclic-carbene palladium complex as an efficient catalyst for Suzuki coupling reaction [12]. Scheuermann et al. reported the palladium nanoparticle preparation by in-situ strategy and supported onto graphite oxide and chemically derived graphene derivatives [13]. They studied the catalytic activity along with the morphology of catalytic surface during the Suzuki coupling reaction and a low leaching of palladium. Rothenberg and his coworkers first reported that the bimetallic clusters Cu/Pd catalyzed the Suzuki coupling reaction of aryl halides and phenylboronic acids with a high product yield [14]. They investigated that the copper-based catalysts enhanced the catalytic activity and these are environmental friendly and less costly which can be an alternative to precious metals. Mao et al. reported that the copper powder in presence of PEG-400 and K_2CO_3 base as an effective catalyst for Suzuki coupling with a high yield for aryl iodides

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[15]. They performed the catalytic reactions in presence of Iodine as co-catalyst to get moderate yield for aryl bromides. Deng et al. reported the copper oxide effectively co-catalyzed the coupling of arylboronic acids with ethyl bromoacetate in presence of palladium species as catalyst to give good yields without using any poisonous base thallium carbonate [16]. Li et al. and Su et al. reported the synthesis of copper and other metals which are immobilized graphene oxide for the styrene epoxidation catalytic studies [17,18].

Graphene oxide (GO) which possess large surface area having many oxygen functional groups along with distinct 2D nanostructure are helpful in amalgamation with various metal salts and polymers [19]. Due to its unique nanostructure, it has applications in the areas such as composites, sensors, solar cells, heterogeneous catalysis and gas storage devices [20]. The hybrid materials possessing both the amino binding group and graphene oxide as an added benefit for the strong bonding of copper other than unfunctionalized GO [21]. The modification in functionalized GO boosts the catalytic performance along with more dispersion [22]. Furthermore, these functionalized GO retains binding capability by enhancing the active sites on the catalytic surface [23]. The leaching of metal does not occur during the reaction because of the metal remains intact firmly with the functionalized GO [22]. These groups make the GO maintain its acidic, basic and oxidative properties without any destruction of its chemical and catalytic performance [3].

GO nanosheets which are formed from graphite using improved Hummers method were bestowed with many oxygen functionalities [24]. GO surface is covalently modified using silane coupling agent 3-aminopropyl trimethoxysilane (APTMS) has an amino group which enhances the chemical modification of GO hydroxyl groups through silylation [25].

In present work, we report an in-situ synthesis of copper Schiff base complex grafted onto graphene oxide. The chemical modification of GO is to support the imino(methyl)benzaldehyde ligands, i.e., condensation of 2-amino phenol with terephthalaldehyde. The aminosilane functionalized GO reacted with copper complexes simultaneously to form metal chelates. It is anticipated that the barrier effect of CAGO complex combination along with its catalytic activity lead to the increase in the formation of carbon-carbon bond using aryl halides in Suzuki coupling reaction and the stability of the prepared nanocomposite which helps in recyclability of the catalyst.

2. Experimental

2.1. Materials

Graphite, 3-aminopropyl trimethoxy silane (APTMS), $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$, terephthalaldehyde and 2-aminophenol, arylboronic acids and aryl halides were purchased from sigma-Aldrich. Ethanol, toluene, acetonitrile, DMSO, hydrogen peroxide, sulphuric acid, and phosphoric acid were obtained from Finar chemicals limited. Caesium carbonate, potassium carbonate, sodium hydroxide, potassium hydroxide, sodium nitrate, sodium carbonate and potassium permanganate are obtained from Lobo chemie limited.

2.2. Synthesis of 3-aminopropyl trimethoxysilane functionalized-graphene oxide (AGO)

GO was prepared according to the improved Hummers method [24]. 1000 mg GO was suspended in 50 ml of dry toluene using ultrasonic bath and refluxed with 4 ml APTMS in N_2 for 24 h [26]. The black product was filtered and washed with dry toluene and ethanol to remove any residual APTMS. The sample was dried in vacuum oven around 90 °C for 6 h.

2.3. Synthesis of Schiff base ligand (L) and copper Schiff base complex (Cu-L)

The Schiff base (L) was synthesized by refluxing 470 mg terephthalaldehyde (1.0 mmol) and 229 mg 2-amino phenol (0.6 mmol) dissolved in 20 ml ethanol. The product was filtered and washed with ethanol before drying in an oven (Yield: 86.2%). In order to study the structure of pure copper Schiff base complex (Cu-L), the complex was synthesized. In typical procedure, 100 mg L and 100 mg $\text{Cu}(\text{Ac})_2 \cdot \text{H}_2\text{O}$ were dissolved in ethanol and stirred at room temperature for 24 h. The product was filtered and washed with ethanol several times before drying (Yield: 82%). (NMR and Mass Spectrometry results are provided in Supplementary Information, Fig. S1–S3. FTIR of Schiff base ligand L is given as Fig. S4).

2.4. Synthesis of Cu-L immobilized Graphene oxide (CAGO)

The CAGO was prepared in a two-step reaction. In first step, the Schiff base was bound to AGO at $-\text{NH}_2$ sites. This was done by refluxing the suspension amino functionalized GO (500 mg, 0.05 mmol) with Schiff base ligand L (100 mg, 0.01 mmol) in 660 mL ethanol and refluxed at 80 °C for 16 h. To this, 100 mg $\text{Cu}(\text{Ac})_2 \cdot \text{H}_2\text{O}$ was added and further refluxed for 24 h at 60 °C. The product brought to room temperature followed by filtration and ethanol wash. The filtered CAGO then dried in an oven at 50 °C.

2.5. Suzuki coupling reaction and yield

Aryl halide (1 mmol) and phenylboronic acid (1.5 mmol) taken in a flask along with the catalyst (0.36 mol%) and base K_2CO_3 (3 mmol) in ethyl alcohol (5 ml), and refluxed at 80 °C for 12 h. The product was analyzed using Gas Chromatography (GC) (Shimadzu 2014, Japan) fitted with siloxane Restek capillary column (30 m length and 0.25 mm diameter) and flame ionization Detector (FID).

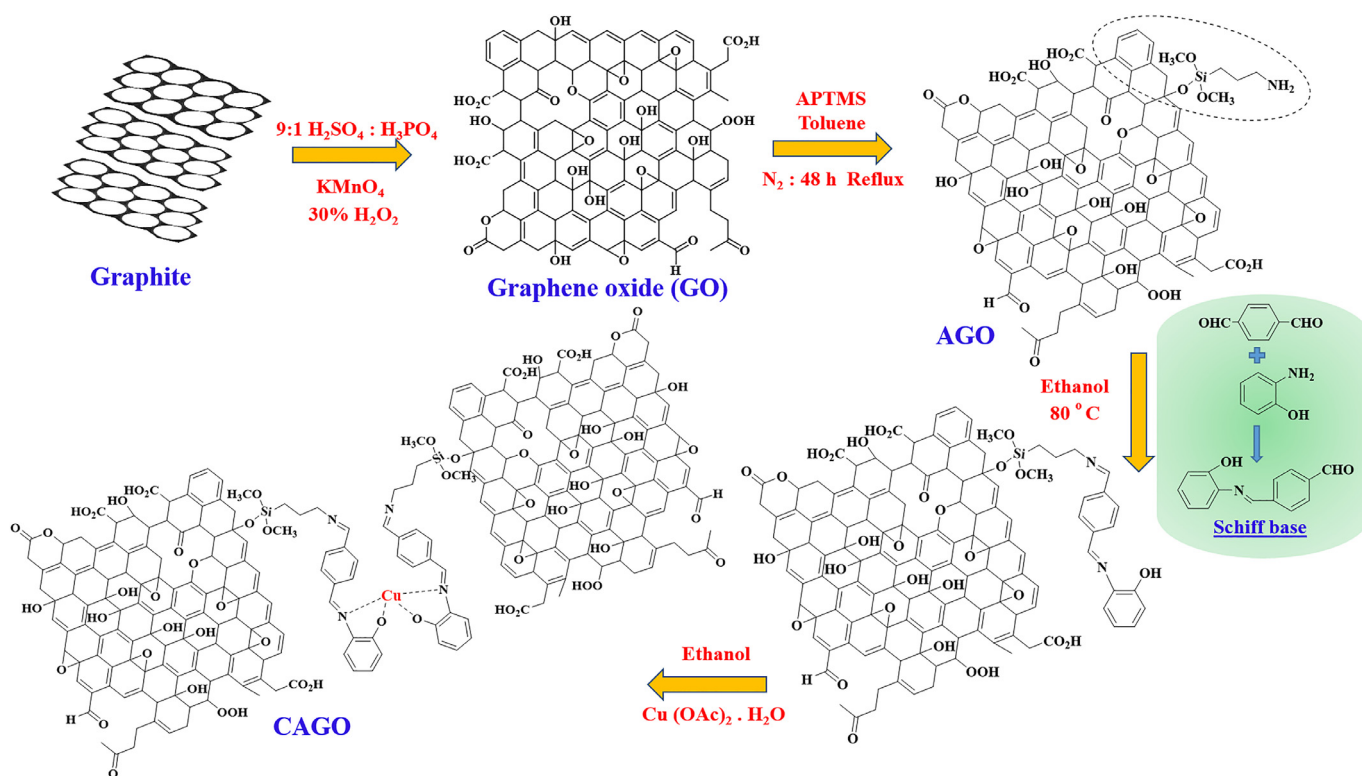
2.6. Characterization

Prepared materials were characterized using ICP-AES, FTIR, DR-UV, TGA, XRD, SEM, EDS, TEM and XPS to understand their structure, composition and stability. The FTIR spectra were recorded on a Bruker-Alpha ECO-ATR FTIR in the range 500–4000 cm^{-1} using KBr method. UV-vis spectra were recorded from 200–800 nm on Analytik Jena SPECORD S600. XRD measurements were carried out using Cu K α radiation on a Rigaku 600 diffractometer Miniflex equipped with Ni-filtered $\text{CuK}\alpha$ radiation (40 kV, 30 mA, and $\lambda = 1.54056$ nm), within diffraction angles of 5° - 80° at the scanning speed of 2° per minute. The surface morphology of the synthesized materials was studied on Zeiss Sigma SEM with oxford EDS. HR-TEM Analysis were taken on a FIE Tecnie-F20 with an acceleration voltage of 200 kV. X-Ray photoelectron spectroscopy (XPS) was measured on a Kratos XSAM800 X-ray electron photometer using Al K α radiation. TG analysis was carried out on a Hitachi DTA-6300 working in a N_2 stream with a heating rate of 5 °C per min in the range of 25–800 °C. The samples were heated at a constant rate of 10 °C/min under N_2 flow from 200–800 °C. Metal content was estimated by inductively coupled plasma atomic emission spectroscopy (ICP-AES) analysis on an Agilent 5100 Synchronous Vertical Dual View ICP-AES with Dichroic Spectral Combiner (DSC) Technology Scheme 1.

3. Results and discussions

3.1. Vibrational spectroscopy

Fig. 1 show the FT-IR spectra of the pure GO, AGO and CAGO. GO exhibit characteristic strong band at 3394 cm^{-1} and 1725 cm^{-1}



Scheme 1. Synthesis of CAGO catalyst.

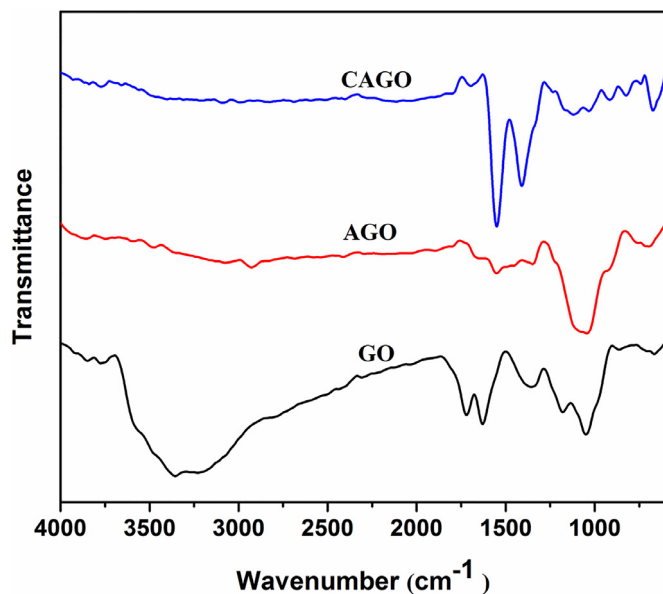


Fig. 1. FTIR spectra of (a) GO, (b) AGO, and (c) CAGO.

representing -OH and C=O bonds of hydroxyl, carbonyl and carboxylic acid groups along with the bands at 1058, 1220, and 1621 cm^{-1} indicating the epoxy (C-O), phenol, C=C bonds in their scaffold [26,27]. AGO presents a doublet band at 2850 and 2917 cm^{-1} exhibiting the presence of symmetric and asymmetric stretching vibrations of the methyl and methylene alkyl chains. The bands at 1121.5, 1034, and 1620 cm^{-1} justify the Si-O-Si, Si-O-C, and C-N vibrations due to the grafting of APTMS onto GO [26,28]. In contrast, CAGO shows a sharp peak at 1438 cm^{-1} for CO which confirmed the binding of copper with acetate groups

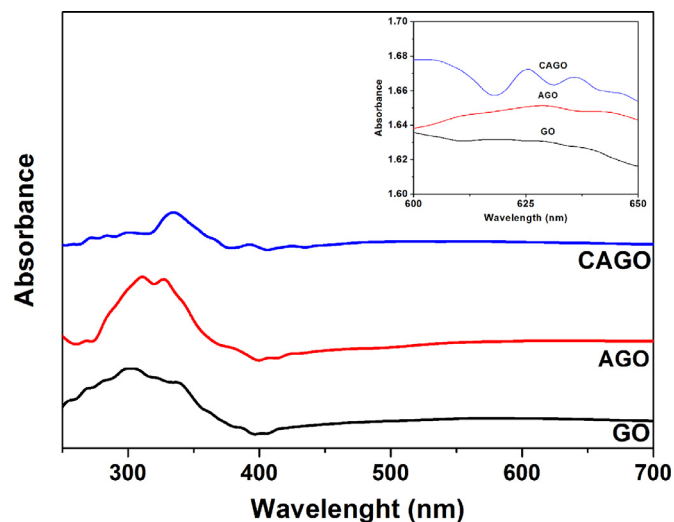


Fig. 2. UV spectra of (a) GO, (b) AGO, and (c) CAGO.

[29,30]. Additionally, band at 674 cm^{-1} signifies the Cu-N bond stretching due to the coordination of copper with N, O sites on the catalytic surface [18]. The FTIR spectrum of ligand is provided in Supplementary Information, (Fig. S4). As observed, the characteristic peaks corresponding OH at 3500 cm^{-1} , and peaks of aldehyde C=O at 1670 cm^{-1} and imine peak at 1620 cm^{-1} , which results in a broad multiplet. This confirms the formation of Schiff base ligand.

3.3. UV-vis spectroscopy

The UV-vis spectra of the pure GO, AGO, and CAGO has absorption bands around 232 nm and 300 nm, indicating the transitions of both p-p* and n-p* on C=C and C=O (Fig. 2). The absorption

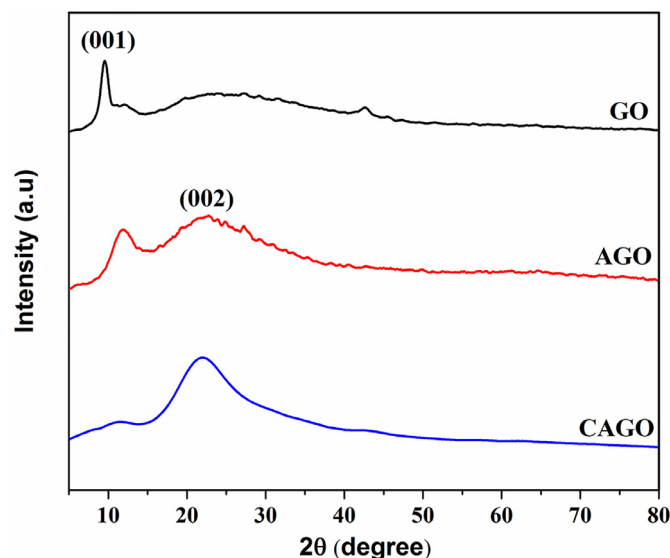


Fig. 3. XRD graph of GO, AGO, and CAGO complex.

bands from 260–430 nm indicated the ligand to metal charge transfer (MLCT) and the ligand charge transfer around pi bond electron and the non-bonded electrons in the catalyst [18]. The band at 320 nm in AGO is due to the amino functionalization of GO surface. Additionally, a weak band around 630 nm in CAGO represents to the d-d transitions of the central copper ion, confirmed the incorporation of metal ion that the transition metal complexes have been successfully anchored on GO [18].

3.4. XRD analysis

The XRD spectra (Fig. 3) of GO, AGO and CAGO shows a broad peak at 10.91 for the (001) reflection of GO [13]. The reminiscence of this peak for AGO and GO states that the presence of GO in both forms. Additionally, AGO and CAGO exhibit a broad peak at 22.24 due to the amino functionalization of GO [27]. The GO peak around 10.9 becomes weak after the introduction of organometallic species which confirms the formation of CAGO [17]. The broad diffraction peaks indicate amorphous nature of the catalyst.

3.5. Electron microscopy

The surface morphology and microstructural features of GO, AGO, and CAGO were analyzed using SEM and EDS (Fig. 4). GO and the CAGO exhibited twists in nanosheets which are not in an ordered phase with crumpled features. The sp^2 hybridized carbon of GO possess folded edges and protrusions [26] which facilitates the immobilization of Cu-L. These reactive areas act as idealistic patterns which help the reactant substrates to form their target modification of GO. The amino functionalized GO surface had network like structures which is the result of solvents used for covalent functionalization. However, the stacking of CAGO was much greater than that of GO was due to the interaction of amino groups at the surface or in the basal planes of GO [31]. The EDS analysis of CAGO exhibited the presence of Cu (Fig. 4(d)) which was absent in AGO (Figure S4). This further emphasis successful immobilization of Cu-Schiff base complex on AGO. Table 1 provides different elemental composition (in atomic %) of GO, AGO and CAGO as measured from EDS. The apparent excess of Cu in CAGO may be due to the presence of Cu in the form of unreacted $Cu(OAc)_2$ which will be deposited in the GO matrix. Additionally, $Cu(OH)_2$ is another species which is formed as a byproduct of the reaction and gets deposited on GO. The composition of Cu element

Table 1
Elemental Composition of GO, AGO, and CAGO from EDS.

Sample	Element (Atomic %)					
	C	O	N	Si	Cl	Cu
GO	33.33	66.67	–	–	–	–
AGO	19.28	38.27	37.89	3.97	0.59	–
CAGO	25.73	51.46	2.82	1.77	–	18.22

was found to be 0.34 mmol/g in CAGO from ICP-AES analysis. These results suggest that the metal complex supported on GO.

TEM of GO shows typical few layer nanoscopic feature (Fig. 5(a)) [32]. The amino functionalization of GO in AGO (Fig. 5(b)) resulted in a layered morphology due to the presence of silane group on its surface. The TEM of CAGO (Fig. 5(c and d)) depicts the retained layered feature with crumpling feature thus suggesting the agglomeration. The SID pattern (Inset Fig. 5(d)) confirmed the same.

3.6. X-ray photoelectron spectroscopy (XPS)

XPS of CAGO was obtained to further understand the nature of binding of C, Si, N and Cu and O in the composite. C1s scan (Fig. 6(a)) shows typical peaks at 284 eV for C–C and C–O–C, and 289 eV for O–C=O binding which is mainly due to the GO support which is further supported by O1s scan with peak at 532.5 eV for C–O binding [18]. The Si 2p and N 1s scan (Fig. 6 (b and c)) represents the silylation between APTMS with the surface hydroxyl groups on the GO support. The prominent peak at 102.25 eV in Si 2p scan corresponds to the SiO linkage while the shoulder at 101 eV is from N–Si linkage. This N–Si linkage can be confirmed by the presence of a doublet corresponding to 398 eV and 400 eV which are due to N–Si₃ and N–Si₂O linkage respectively. The Cu 2p spectra (Fig. 6(d)) shows two typical bands at 933.9 which is indicative of Cu–O bond whereas 953.8 eV represents Cu–N bond of Cu(II) ions. Additionally, a Cu 2p_{3/2} satellite peak at 943.0 eV is observed which is a result of the electrostatic interaction between the ligand and the copper metal ion [29,33]. Fig. 6(f) gives the specific binding energy of Cu, N, O, C and Si elements in the CAGO. These observations support the predicted binding of various elements in the prepared complex.

3.7. Thermal analysis

TGA of GO (Fig. 7) depicts two degradation steps in GO. The loss within 170–250 °C is attributed to the loss in H₂O and CO₂ due to the removal of OH and C–O–C functional groups which is usually the case in thermal reduction of GO. The gradual loss in the region from 280–600 °C presumably due to the degradation of the left over C–O and C–H moieties [34,35]. The weight loss in the CAGO are observed in three steps (Fig. 7). First, a loss of 4.59% at 90–190 °C represented the removal of physically adsorbed water. Another weight loss of 33% around 250–450 °C corresponds to the thermal decomposition of undigested functionalities of oxygen along with unreacted APTMS [36]. This suggest that CAGO can be utilized as a heterogenous catalyst upto 250 °C without undergoing degradation. Final, the weight loss region corresponds to the degradation of GO.

3.8. Suzuki coupling reaction

The catalytic performance of the synthesized catalyst was explored using the Suzuki coupling of certain phenylboronic acid and phenyl iodide as the model reaction. The summary of CAGO catalyzed reactions with different solvents and base are given as

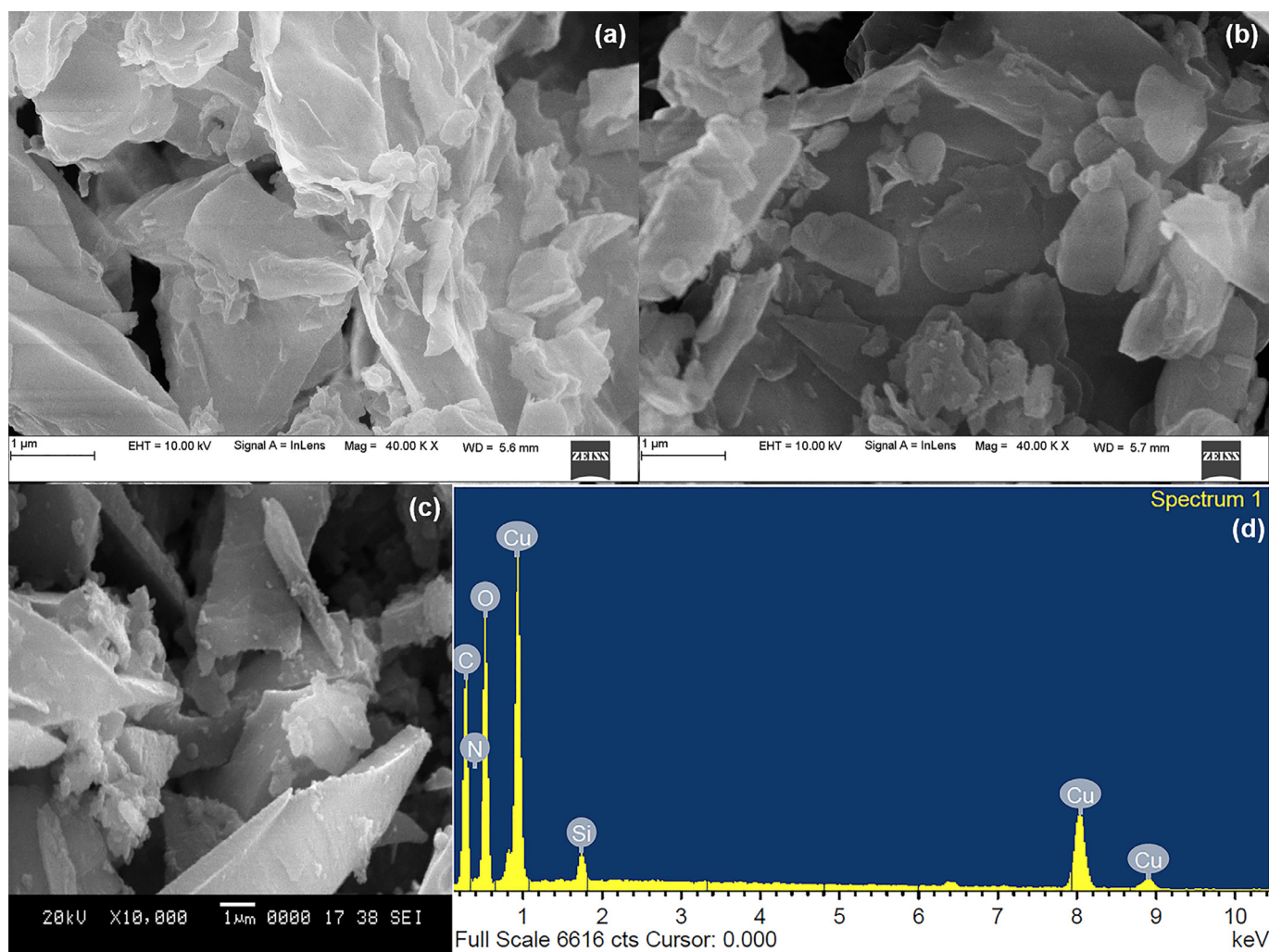


Fig. 4. SEM of (a) GO, (b) AGO, (c) CAGO, and (d) EDS image of CAGO.

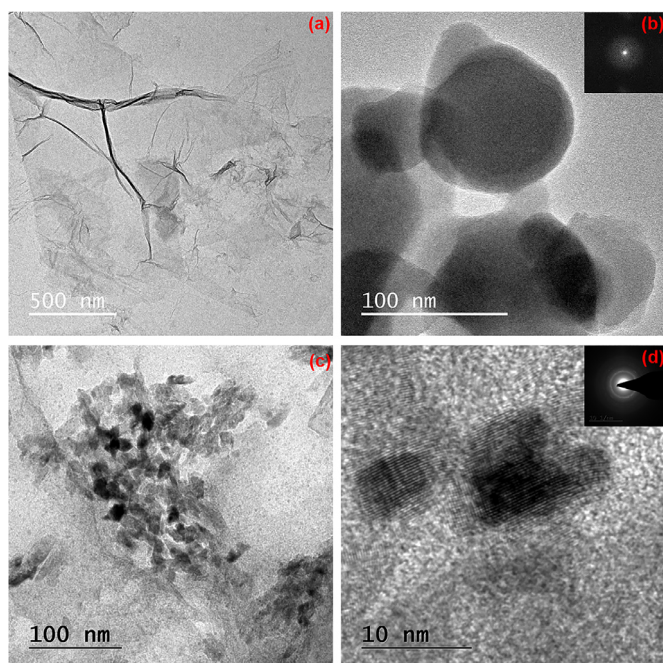


Fig. 5. TEM images of (a) GO, (b) AGO, (c) CAGO, and (d) Hi-Res image of CAGO.

Table 2

Effect of various solvents and bases on yield at temp 80 °C.

Sl. No.	Solvent	Base	Temperature	Catalyst (mol%)	Yield (%)
1	EtOH	K ₂ CO ₃	80	0.36	94
2	EtOH	Na ₂ CO ₃	80	0.36	88
3	EtOH	Et ₃ N	80	0.36	54
4	EtOH	KOH	80	0.36	65
5	EtOH	NaOH	80	0.36	58
6	Acetonitrile	K ₂ CO ₃	80	0.50	75
7	Dimethyl sulfoxide	K ₂ CO ₃	80	0.50	71
8	Tetrahydrofuran	K ₂ CO ₃	80	0.50	60.5
9	Toluene	K ₂ CO ₃	80	0.50	52

Table 2. EtOH and K₂CO₃ were found to be the best solvent and base pair for the reaction with GC yield of 94%. Further, the effect of catalyst on the yield of reaction for different substituted aryl halides and phenylboronic acids are given as Table 3. The results suggest that the amount and time taken for the product formation for CAGO catalyzed reaction depends on the nature of substituent group. Aryl bromides having donor moieties require a long time to complete their reaction because of the polarity in molecule which is due to the transfer of electron through π -bond on aryl bromides. In fact, the product formation is less for aryl chlorides because of the high bond dissociation energy of the C–Cl bond (96 kcal/mol) [37]. Henceforth, the reaction between phenyl iodide

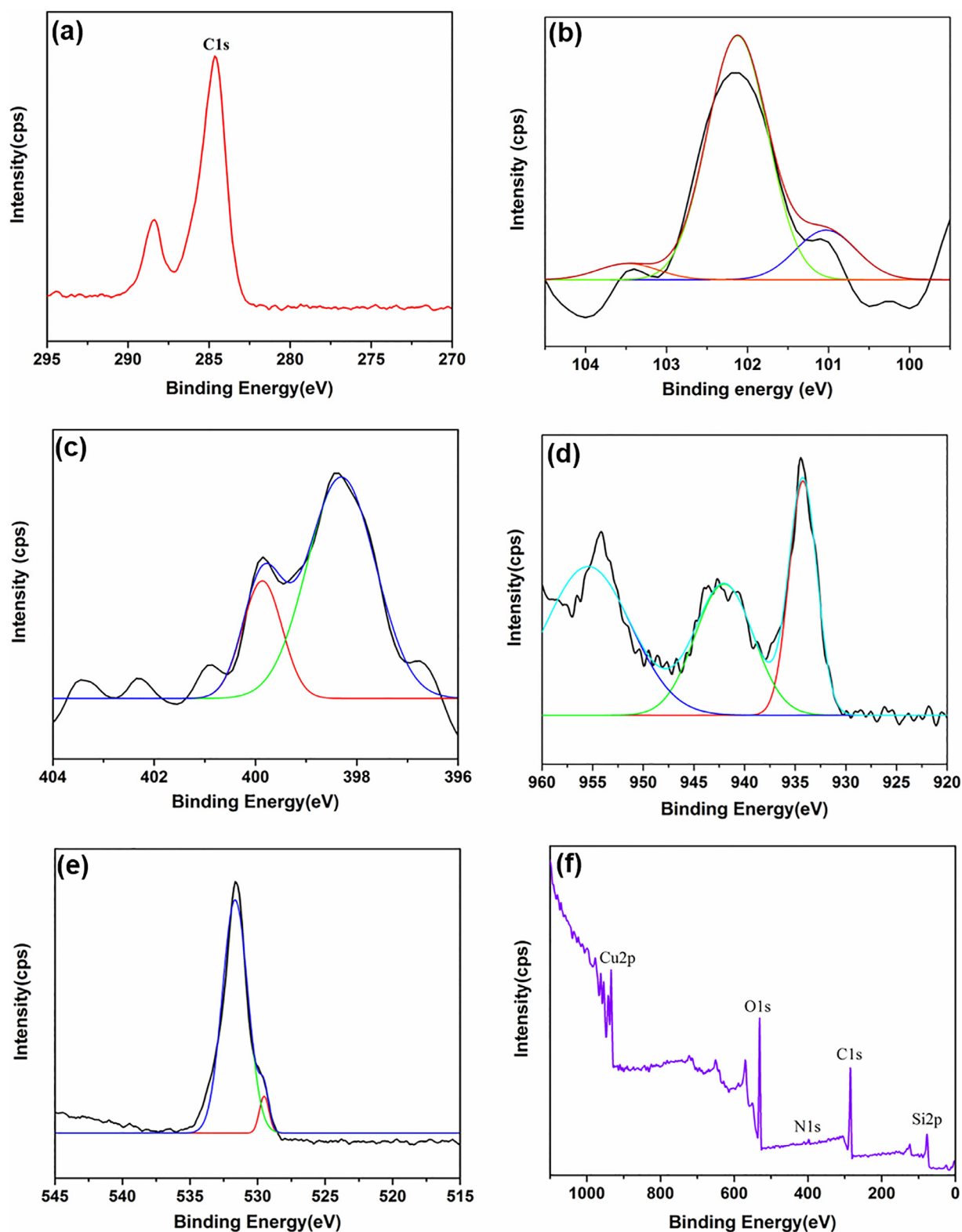


Fig. 6. XPS analysis of CAGO composite with (a) C 1s scan (b) Si 2p scan (c) N 1s scan, (d) Cu 2p scan, (e) O 1s scan, and (f) elemental survey.

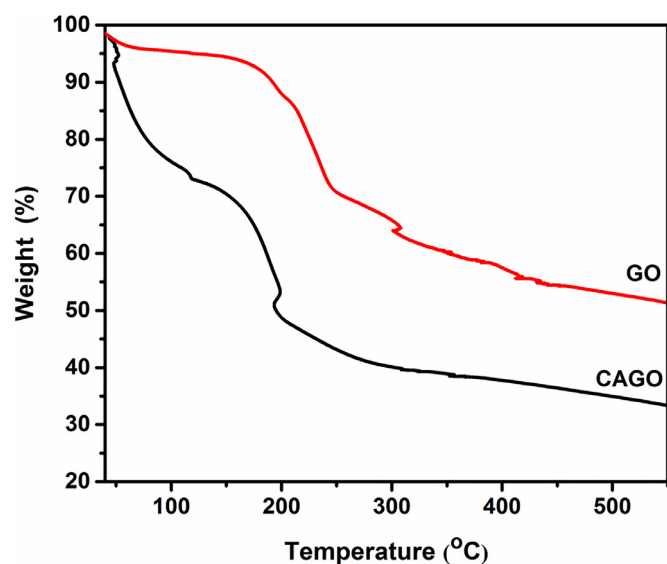


Fig. 7. TGA of the (a) GO, and (b) CAGO.

and phenyl boronic acid was monitored to study the performance of CAGO as catalyst.

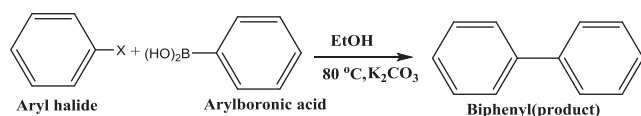


Table 4 represents the comparison of catalytic yield of CAGO with GO + CuL as control catalyst. As observed from the % yield, it can be seen that CAGO provides highest yield among all the three catalyst which is due to the absorptive nature of the catalyst. In case of GO + CuL catalyst, the yield was found to be very low which can be attributed to the fact that Cu-L is not bound to GO therefore it goes into liquid phase whereas the reactants get adsorbed on GO there by limiting the yield. This can be further ascertained by the use of only Cu-L complex as catalyst which showed improvement over the GO + CuL catalyst because the reactant and catalyst were in same phase. Therefore, from this experiment it can be seen that, Cu-L is active catalyst for the Suzuki cross coupling reaction, and when it is bound to AGO, the synergistic effect of catalytic activity and adsorption increase the overall reaction yield.

The influence of the amount of catalyst was investigated, and it was found that the biphenyl product formation enhanced from 2% to 94% when the catalytic amount of catalyst gradually raised from 0.01 mol% to 0.36 mol% under the optimal reaction conditions (Fig. 8(a)). Further increase in the amount of catalyst showed no significant improvement in yield. Additionally, the catalyzed reaction was observed to give highest yield for 8 hours of reaction after which there was no further improvement (Fig. 8(b)).

The effectiveness of a heterogenous catalyst in nanocatalysis depends on its particle size, irrespective of the coupling reaction mechanism. The smaller the nanoparticle is, better will be its catalytic activity. Therefore, CAGO displays more catalytic activity due to the good dispersion and small size of the Cu-L nanoparticles. GO which possess rich oxygen functional groups, aids in tuning the surface of Cu-L nanoparticles without aggregating and maintained catalyst performance throughout the reaction.

The catalyst recyclability studies (Fig. 9) were further done to prove the reusability of the heterogeneous catalyst, and were found to be beneficial for reliable practical applications. 0.36 mol% of CAGO catalyst was used for this reaction. The study suggests that CAGO catalyst can be reused for 4 times without any significant loss of its catalytic activity. However, a gradual decrease

Table 3
GC yield of the products of various aryl halide and arylboronic acids at 80 °C.

Sl. No.	Aryl halides	Arylboronic acids	Time(h)	Yield (%)
1			8	94
2			12	92
3			24	80
4			24	76
5			24	72
6			12	89
7			12	87
8			12	84
9			24	80
10			24	74
11			24	64
12			24	69
13			24	88
14			24	18
15			24	23

Reaction Content: EtOH(5 ml), Aryl halides(1.0 mol), arylboronic acids(1.5 mol), K_2CO_3 (2 mol), and CAGO Catalyst (0.36 mol%).

Table 4
Comparison catalytic yield of CAGO with control catalyst GO + CuL and only Cu-L complex.

Sl. No	Catalyst	Amount of catalyst (mol%)	Yield (%)
1	CAGO	0.36	94
2	CAGO	0.72	94
3	CAGO	0.18	80
4	CAGO	0.09	62
5	GO + CuL	0.36	5
6	GO + CuL	0.72	13
7	GO + CuL	1.00	13
8	Cu-L	0.18	32
9	Cu-L	0.36	46
10	Cu-L	0.72	65
11	Cu-L	1.00	65
12	Cu-L	2.00	65

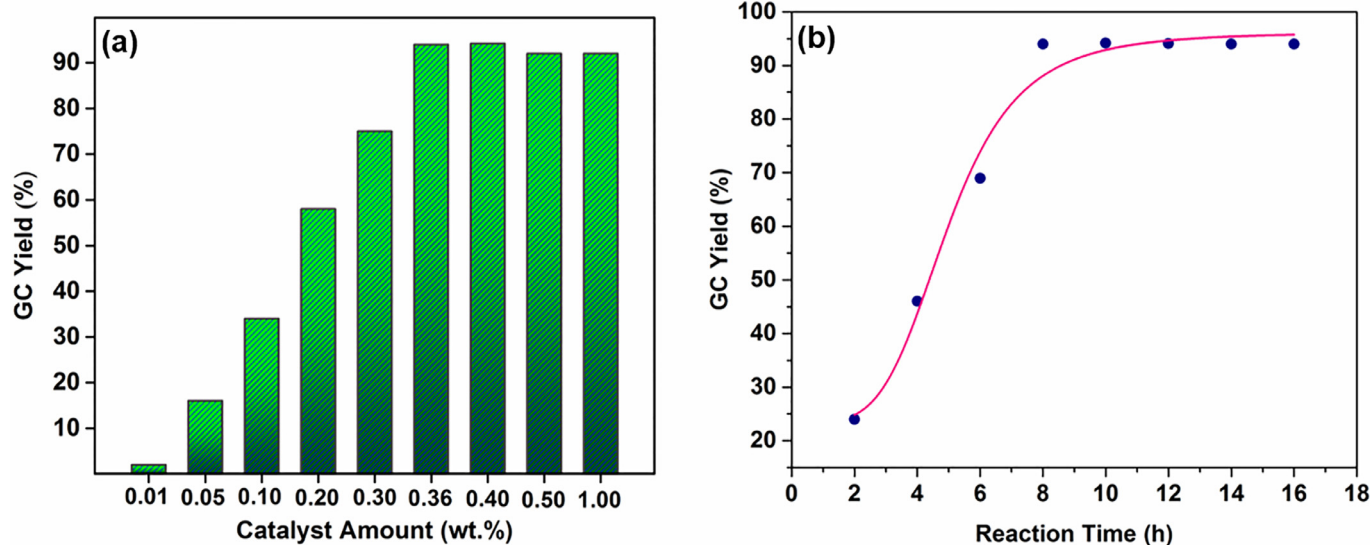


Fig. 8. The effect of (a) catalyst amount and (b) reaction time on the GC yield of Suzuki coupling reaction.

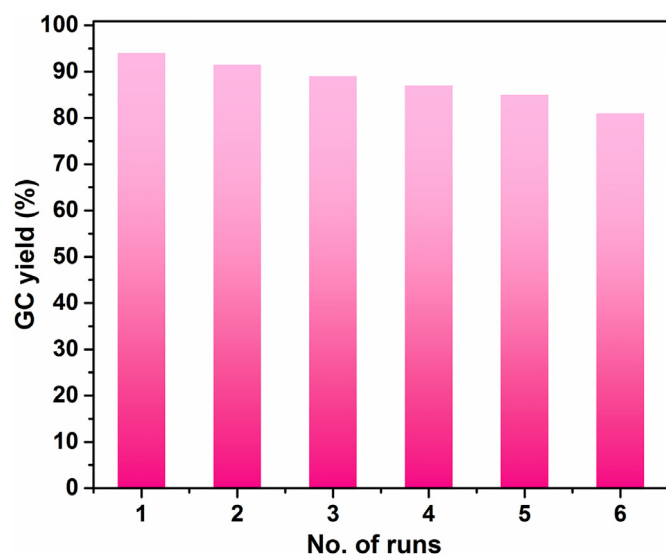


Fig. 9. Recycling ability of CAGO as catalyst for Suzuki coupling reactions.

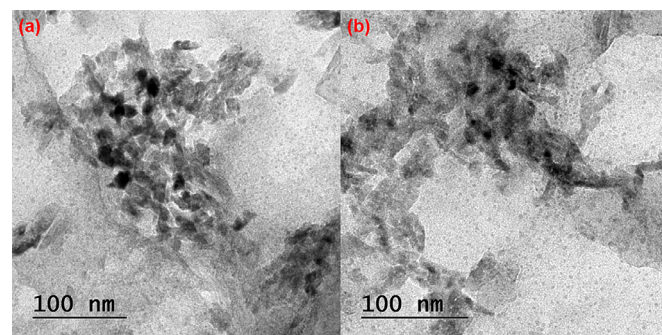


Fig. 10. TEM images of fresh and used catalyst after 5th cycle (a) fresh catalyst (b) used catalyst.

in the product yield was observed in subsequent reactions. The catalyst metal leaching during the reaction was studied using ICP analysis. The concentration of copper metal in CAGO catalyst and the recovered catalyst was found to be 0.28 mmol/g which confirmed that copper leaching of the catalyst was very less. The TEM image of the fresh catalysts and the used catalyst (Fig. 10(a and b)) proved that the metal leaching is less during the coupling reaction. The results proved that CAGO complex is stable which prevents the copper leaching.

4. Conclusions

An in-situ strategy has been developed for the synthesis of a copper Schiff base complex and simultaneously grafted on GO support through a surface modification. The as-prepared catalyst was found to be an efficient catalyst for the synthesis of biphenyl as products from the Suzuki cross-coupling reaction (C–C bond) of substituted aryl halides and phenylboronic acid. The 2D structure of GO support which owe to its excellent performance possessing outstanding properties and the robust synthetic methodology.

Therefore, GO acts as good supporting material having its unique properties and a powerful supporting agent for homogeneous metal complexes for their practical applications. Heterogeneous nanocatalysts is highly active, possesses high surface area, and its thermal and chemical stabilities were confirmed using various characterization technique through spectroscopic methods and surface analysis. This recoverable and reusable catalyst is benign, low cost and be handled easily. More wide and in-depth analysis in heterogeneous coordination catalysis were done to further studies in various organic reactions were anticipated.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jtice.2018.09.029.

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