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Synthesis, Characterization and Catalytic Activity of Melamine-Based Dendrimer Encapsulated Pd/Cu Bimetallic Nanoparticles

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ABSTRACT

A melamine-based dendrimer was used to prepare a new class of heterogeneous Pd/Cu bimetallic nanoparticles (NPs). The physicochemical characteristics of Pd/Cu bimetallic NPS were studied with the aid of SEM, EDX, XRD, TG& DSC techniques. Leaf-like surface morphology and the presence of required palladium and copper ion were observed from SEM and EDX analysis respectively while TG& DSC showed good thermal stability, as well as XRD, revealed nano-shape FCC structure. The recycling ability of the catalyst was examined for five successive runs without any noteworthy loss of activity. The synthesized Pd/Cu bimetallic NPS exhibited high catalytic activity in the synthesis of ynone in which synthetic route was phosphine ligand-free.

Keywords: Melamine, Dendrimer, Bimetallic Nanoparticles, Ynone

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Introduction

Dendrimers are highly branched, spherical, multivalent, monodisperse molecules with synthetic versatility and a broad range of implementations from catalysis to electronics and drug delivery [1-4]. Melamine-based dendritic structures usually show external groups that can take part in bonding with hydrogen. Fréchet and coworker illustrated in an elegant example the self-assembly of greater-ordered dendrimer structures by adding two complementary hydrogen-bonding moieties to the focal point of various dendrons [5, 6].

Moreover, bimetallic nanoparticles, consisting of two different metals, display enhanced catalytic output and implementations in so many industrial applications, predominantly in fuel industries or environmental catalytic processes, and lately in C–C cross-coupling reactions [7]. Bimetallic catalysts constitute a remarkable class of catalysts as a result of which one metal can modify and/or alter catalytic properties of the other owing to the electronic and structural interactions [8].

Furthermore, they are effective due to the adjustment of their surface electrons comparative to that of individual metals [9, 10]. The composition, framework and more exploitation of catalytic activity of bimetallic particles were shown to be of tremendous interest [11, 12]. It has been demonstrated that metals interacting with either another metal or metal compound at the nano level can shape nanoparticles with greater activities not found in bulk alloys [13].

On the other hand, numerous metal-catalyzed cross-coupling reactions involve either another metal to be present as a co-catalyst to support the entire process. Sonogashira reaction between an aryl halide and terminal alkynes requires a lot of Pd and Cu as a catalyst in which Cu⁺ plays a catalytic role in switching the alkynyl group to Pd [14].

In addition, the outstanding redox characteristics of the Pd / Cu system are due respectively to the

electron donor and acceptor character of Cu and Pd [15, 16]. The noticeable "Cu-effect" in the Pd-catalyzed cross-coupling reaction induced the progression of many heterogeneous bimetallic NPs utilizing available in the market insoluble polymeric supports and has been used satisfactorily in numerous cross-couplings [17, 18].

However, bimetallic nanoparticles catalysts inserted in/on insoluble supports with ligand-free and negligible or no leaching are extremely desirable from the perspective of decreasing environmental burdens as well as cost-effectiveness. The major challenges for a heterogeneous catalyst remain procedural brevity, uniform dispersion, lower cost and reliable dendrimeric surface, better efficiency and life cycle.

Yet, to the best of our knowledge, there is no study obtainable in the literature on the synthesis of bimetallic NPs encapsulated with melamine based dendrimer and the utilization in cross-coupling reaction such as ynone preparation reactions of their heterogeneous catalytic activity. Here, we have reported the synthesis, characterization, and utilization as a catalyst in the synthesis of biologically and pharmaceutically important intermediates ynone compound of a new class of heterogeneous melamine based dendrimer encapsulated Pd / Cu bimetallic NPs.

Experimental

Materials and Methods

All the solvents such as DMF, DMSO, CH₃CN, C₂H₅OH, DMF, and bases, for example, NaOH, KOH, KO^tBu, Na₂CO₃, and Et₃N, all the reagents such as melamine, Anisoyl chloride, 1-heptyne, CuCl₂·2H₂O, and PdCl₂ were purchased from Aldrich and were directly used without further purification. The IR spectra were taken on a Shimadzu FTIR 8400S Fourier Transform with KBr pellets. NMR spectra were taken using tetramethylsilane as an internal standard on the BRUKER DPX-400 spectrophotometer. Analytical thin layer chromatography (TLC) was

silica gel 60 F 254 coated on 25 TCC aluminum sheets (20 × 20 cm). Silica gel column chromatographic separations were made on silica gel 60 N (neutral, 40-100 μM). The thermal stability of nanoparticles was ascertained by a thermogravimetric analyzer (NETZSCH STA 449F3) from 26 to 600 °C. TG and DSC data were observed under a nitrogen atmosphere by using aluminum oxide crucible at a heating rate of 10 k/min and at a flow rate of 40 and 60 mL/min. SEM and EDX of Pd/Cu bimetallic NPs 4 were taken by the JEOL-JSM-7600F. Particle size and phase were identified with a PANANALYTICAL x-ray diffractometer. Elemental analyses were carried out with a Fisons EA 1108 CHNS-O apparatus. Particle size and phase identification were performed with a PANANALYTICAL x-ray diffractometer. The leaching study was done by Perkin Elmer ELAN DRC E ICP/MS.

Synthesis of 2,4,6-tris(di-4-methoxybenzamido)-1,3,5-triazine 3

0.492 g (0.0039 mol) of melamine was dissolved in 10 ml of dimethylsulfoxide (DMSO) in a 250 ml round bottle flask and then 2.16 g (0.024 mol) of anisoyl chloride was added to it. The reaction mixture was allowed to stir with reflux at 90 °C temperature for 8 hours. Reaction advancement was monitored with TLC. After completion of the reaction, the flask was allowed to cool at room temperature and some pieces of ice were poured in the flask very carefully. After a while, it was filtered on a Buchner funnel and washed with Sodium bicarbonate solution to remove completely any remaining acid. Finally, the compound was purified by recrystallization with ethanol.

White crystalline solid; yield: 94%, IR (KBr): ν_{\max} = 3015.00, 1765.21, 1615.50, 1415.20, 1290.40 cm^{-1} . ^1H NMR (400 MHz, CD_3OD): δ = 6.87 (d, 12H, $J=8.8$ Hz), 7.73 (d, 12H, $J=9.2$ Hz), 3.72, (s, 18H) ppm. ^{13}C NMR (100 MHz, CD_3OD): δ = 169.99, 144.97, 130.80, 130.09, 129.10, 21.58 ppm. Anal. Calcd. for $\text{C}_{51}\text{H}_{42}\text{N}_6\text{O}_{12}$; C 65.80, H 4.55, N 9.03; found: C 65.70, H 4.45, N 9.00

Synthesis of melamine Based Dendrimer Encapsulated Pd/Cu Bimetallic NPs 4

0.100 g 2,4,6-tris(di-4-methoxy benzamido)-1,3,5-triazine **3** (0.12 mmol), 0.00096 g $\text{PdCl}_2 \cdot \text{NH}_2 \cdot \text{NH}_2 \cdot \text{H}_2\text{O}$ (1.5 mmol) were stirred in CH_3CN (10 mL) in a round bottom flask at 90 °C for 1 hour under reflux condition. Then 0.01536 g of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (Pd/Cu weight ratio of 1:16) and $\text{NH}_2 \cdot \text{NH}_2 \cdot \text{H}_2\text{O}$ were added drop wise to some extent in excess of the equivalent amount of copper salt to the reaction mixture. The NaOH solution (0.3 M) was gradually added to the reaction mixture in order to maintain pH 12 and the suspension was robustly stirred at 90 °C for 2 hrs with a magnetic stirring bar. The black residue was originated after the reaction mixture had been settled down. The residue was filtered after the reaction mixture was centrifuged (4000 rpm for 20 min), washed with distilled water and acetone and dried overnight at 130 °C in an oven.

Application of Pd/Cu bimetallic NPs 4 as a catalyst in the synthesis of ynone

Synthesis of 1-phenyloct-2-yn-1-one 7

In $\text{C}_2\text{H}_5\text{OH}$ (10 mL) a combination of benzoyl chloride **5** (1.0 mmol) with 1-Heptyne **6** (1.2 mmol), Pd/Cu bimetallic NPs 4 (1.5 mol %) and triethylamine (1.2 mmol) were stirred for 30 minutes in a round bottom flask in a nitrogen atmosphere at 70 °C. The advancement of the reaction was monitored by TLC. The reaction mixture was evaporated under reduced pressure to dryness at the end of the reaction and the residue was eliminated with chloroform. Washed with distilled water, the chloroform extract was dried over anhydrous MgSO_4 , filtered and concentrated under reduced pressure. Then it was purified with n-hexane/chloroform (8:1) through silica gel column chromatography. Solid white product was obtained, yield % 90; mp. 70-72°C, IR (KBr): ν_{\max} = 3312.85, 2975.30, 2938.65, 2739.97, 2492.11, 2357.09, 2162.09, 1474.63, 1398.64, 1171.79 cm^{-1} ; ^1H -NMR (CDCl_3 , 400 MHz): δ 7.45 (d, $J=8.0$, 2 H); 7.11 (t, $J=8.0$, 2H); 6.37-6.44 (m, 3H); 2.42 (t, $J=8.0$, 2H); 1.92-1.90 (m, 2H); 1.44-1.37 (m, 4H); 0.93

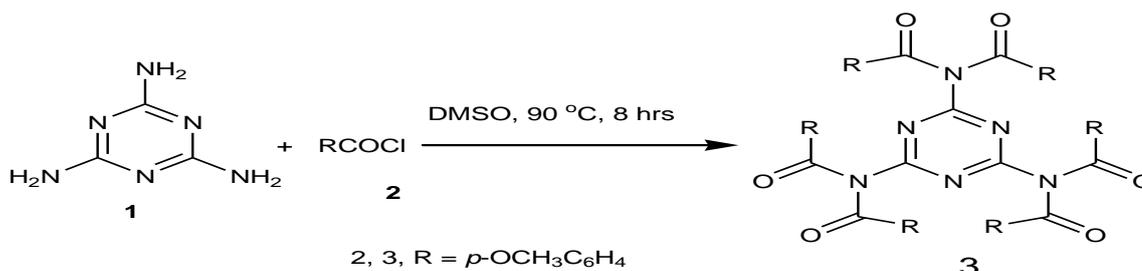
(t, $J=7.6$, 3H). ^{13}C -NMR (CDCl_3 , 400 MHz): δ 175.28, 135.11, 134.23, 130.13, 129.23, 94.33, 30.09, 28.27, 18.90, 14.44

Results and Discussion

Synthesis and characterization

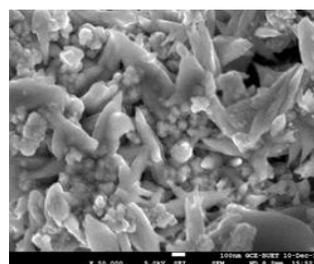
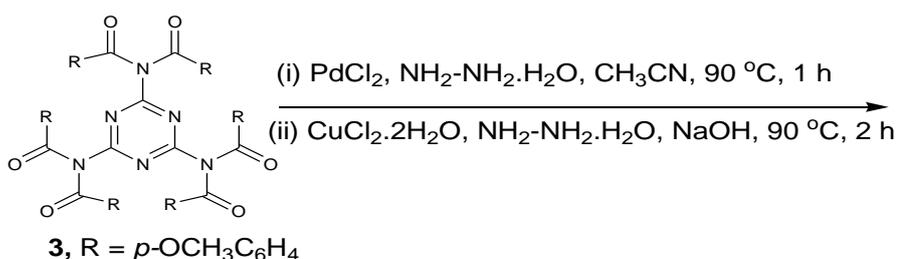
Dendrimer 3 was produced by the reaction of 0.492 g (0.0039 mol) of melamine 1 with 2.16 g (0.024 mol) of anisoyl chloride in anhydrous DMSO at 90 °C temperature for 8 hours under a

nitrogen atmosphere (Scheme 1). The growth of the reaction was monitored by thin layer chromatography (TLC) and after the completion of the reaction, the solid reaction mixture was washed with sodium bicarbonate solution and recrystallized with ethanol that gave the dendrimerized product 3. The product was analyzed by using IR, ^1H NMR, ^{13}C NMR (S1-S3 in the supporting information).



In the IR spectrum the presence of vital peaks confirmed the structure of compound 3. The peak at the stretching bands 3015, 2965 and 1510 cm^{-1} indicated the existence of aromatic C-H, aliphatic C-H and aromatic C=C respectively whereas the band was originated at 1680-1770 cm^{-1} due to C=O groups. Here it is

noteworthy that there's no band for N-H near about 3400–3200 cm^{-1} which was proved that 1° amino group of melamine was changed into 3° amido group. In ^1H NMR and ^{13}C NMR spectra offered the preferred dendrimer 3. There is no ^1H NMR peak for NH or NH₂ group, which was additional established the structure of 3.

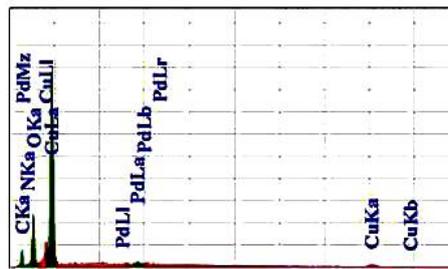
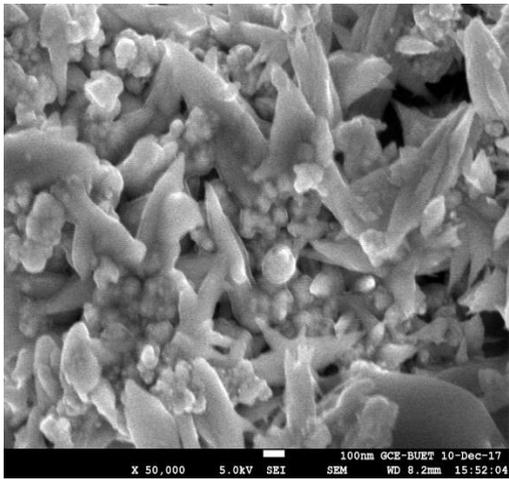


Pd/Cu bimetallic NPs, 4

Scheme 2. Synthesis of Pd/Cu bimetallic NP 4

The melamine-based dendrimer supported Pd / Cu bimetallic NPs⁴ was produced by using a sequential loading approach (Scheme 2) and thus described with various physicochemical techniques including SEM, EDX, XRD, TGA, and DSC. The dendritic globular or leaf shape was shown in the SEM images of Pd-Cu bimetallic NP 4 (Figure 1). The presence of

copper was 60.75 % of the mass and palladium 4.40 % of the mass of the synthesized NPs (Figure 2) was identified by EDX analysis. Furthermore, the EDX results confirmed that the sequential loading technique might be quite successful in preparation the melamine based-dendrimer template Pd / Cu bimetallic nanoparticle 4.



Acquisition Parameter
 Instrument : 7600F
 Acc. Voltage : 10.0 kV
 Probe Current: 1.00000 nA
 PHA mode : T3
 Real Time : 30.28 sec
 Live Time : 30.00 sec
 Dead Time : 0 s
 Counting Rate: 1044 cps
 Energy Range : 0 - 20 keV

ZAF Method Standardless Quantitative Analysis

Fitting Coefficient : 0.0837

Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K ^α	0.277	14.02	0.25	33.48				5.6763
N K ^α	0.392	1.61	0.15	3.30				1.7853
O K	0.525	19.22	0.34	34.44				21.5728
Cu L	0.930	60.75	0.63	27.87				67.8324
Pd L ^α	2.838	4.40	0.30	0.91				3.1331
Total		100.00		100.00				

Figure 1 SEM of Pd/Cu bimetallic NPs 4. Figure 2 of EDX of Pd/Cu bimetallic NPs 4.

The particle structures of Pd-Cu bimetallic NPs 4 was additional described by the XRD patterns (Figure 3). The peaks at 2theta(o) values of 52.70 and 73.40 degrees relate to (200) and (220) copper planes respectively those directed for the creation of FCC copper nanoparticles and associated with the JCPDS General Powder Diffraction Card, Copper Record No (04—0836) [17] while the peaks at 2theta(o) values of 39.10, 55.00 and 67.30 correspond to (111), (200) and (220) planes of Palladium respectively those regarded as the formation of FCC Palladium nanoparticle and Palladium report no (46—1043) [18], (figure 3), compared to the well-known JCPDS powder diffraction card. As a

result, these results were revealed in Pd-Cu bimetallic NP's FCC structure. The Pd-Cu bimetallic NP 4 XRD pattern shows the average 18.60 nm particle length calculated through the Debye-Scherrer formula.

. Thermal steadiness is the main characteristic for catalysts to maintain their activity in long reaction times. It was detected from the TG and DSC curves of the Pd / Cu bimetallic NP 4 (Figure 3). The material of nanoparticle changes with its structure and composition and is measured through the thermo-gravimetric diagram depending on the weight loss of these elements.

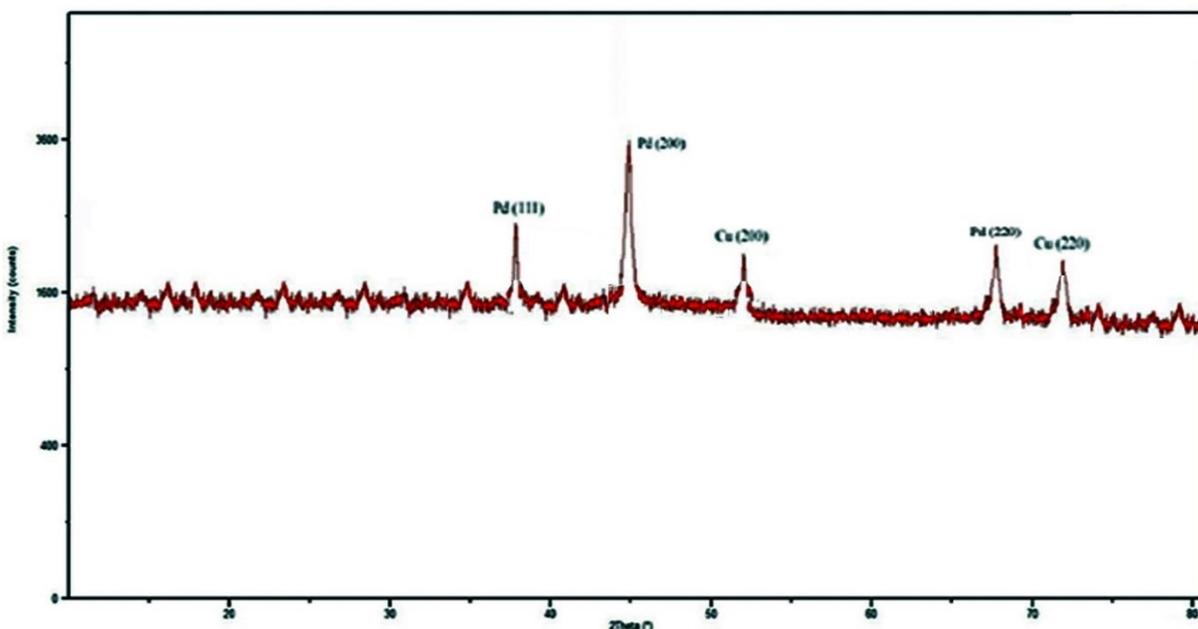


Figure 3 XRD pattern of Pd-Cu bimetallic NPs 4

From the TG curve, it was recorded that the overall weight loss of compound 4 from 330 °C to 395 °C and the DSC values are just about comparable to the TG values. The melting point

was indicated at approximately 330 °C from the DSC endothermic curve. The final step of decay revealed about 19.10 % ash residue of the catalyst 4.

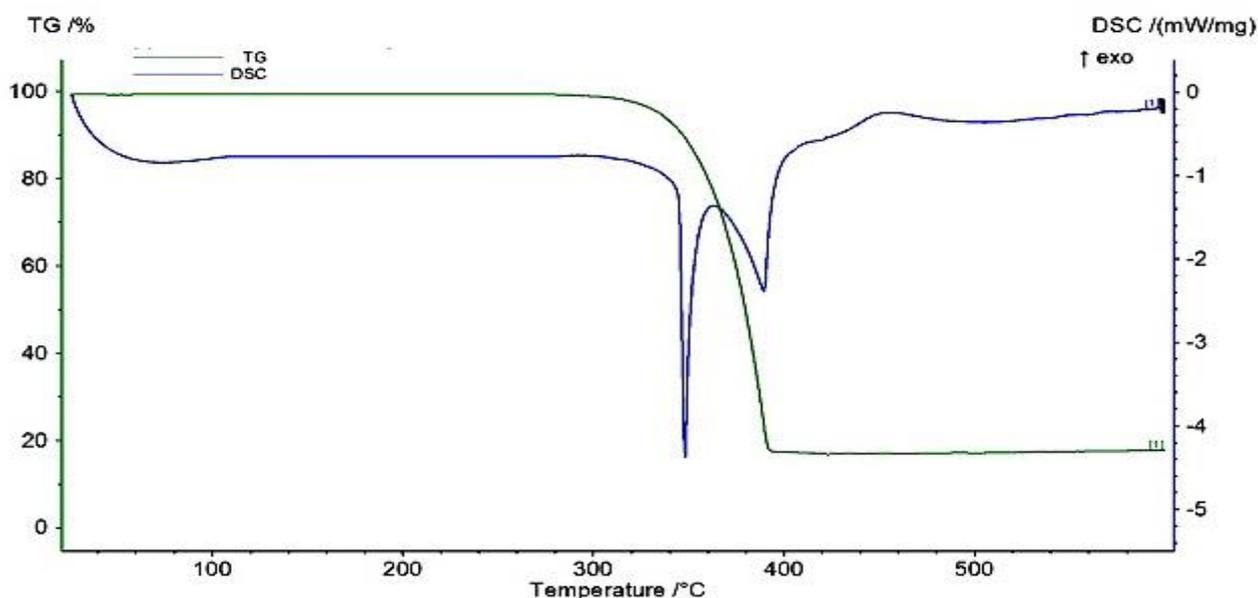
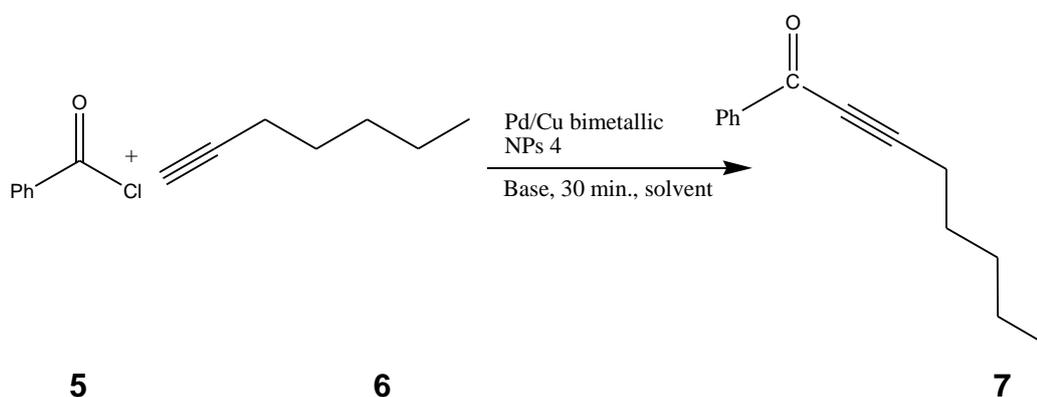


Figure 4. TG& DSC curves of Pd-Cu bimetallic NPs 4

Catalytic performance

The catalytic effect for the melamine-based dendrimer containing Pd/ Cu bimetallic NPs 4 through the reaction between acyl chlorides with a terminal alkyne. Using Pd/Cu bimetallic NPs 4 (Scheme 3), the reaction between benzoyl chloride and 1-Heptyne was chosen to optimize the synthetic method of ynone. The cross-coupling reaction was performed at 70 °C with the presence of NaOH as a base and DMF as a solvent to produce 20 % yield for the catalyst Pd/Cu bimetallic NPs 4

(entry 1). We elected another four bases for KOH, KO^tBu, Na₂CO₃, and Et₃N (Table 1, entries 2-4) and noticed that Et₃N was the best base for this reaction. The solvent effect was also examined by using H₂O, DMF, toluene and acetonitrile and ethanol and also ethanol was found as a best solvent. The optimized reaction condition was indeed found when 1.5 mol % of Pd/Cu bimetallic NPs 4 and Et₃N and C₂H₅OH were used at 70 °C (Table 1, Entry 5) under the nitrogen atmosphere. The product 7 was analyzed by IR, ¹HNMR, ¹³CNMR (Figures 4-6 in the supporting information).



Scheme 3

Table 1. Reaction condition optimization for the coupling reaction of benzoyl chloride with 1-heptyne catalyzed by Pd/Cu bimetallic NPs 4

Entry	Base	Temp (°C)	solvent	Catalyst, 4 (mol%)	Yield (%)
1	NaOH	70	DMF	1.5	20
2	KOH	80	Toluene	1.0	10
3	KO ^t Bu	R.T.	CH ₃ CN	1.0	N. R
4	Na ₂ CO ₃	80	H ₂ O	1.5	40
5	Et ₃ N	70	C ₂ H ₅ OH	1.5	90

Reaction conditions: benzoyl chloride (1 mmol), 1-heptyne (1.2 mmol), base (1.2 mmol), time-30 min, solvent (10 mL), b) Yield % of products is based on benzoyl chloride.

Test for Recyclability

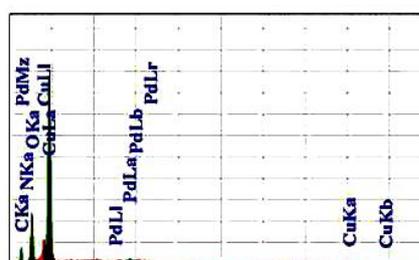
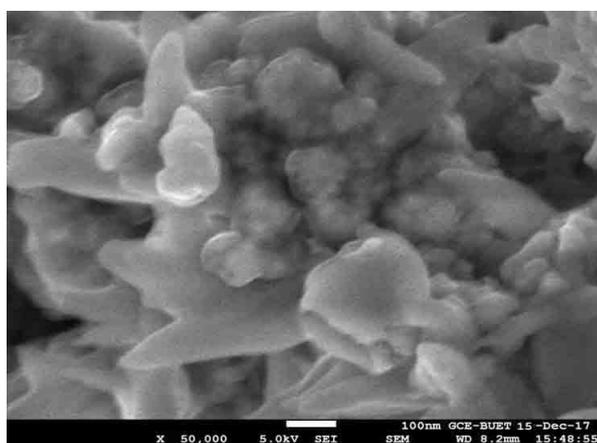
The reusability of the catalyst 4 was examined in the reaction between benzoyl chloride and 1-heptyne. After every catalytic run, the catalyst 4 was recovered from the reaction mixture by simple filtration, washed with water followed by acetone, and then dried under vacuum. The

recovered catalyst was reused five times without noteworthy losing its catalytic activity (Table 2).

SEM images of the recovered Pd-Cu bimetallic NPs 4 showed nearly retained surface morphology of original catalyst (Fig. 5) as well as Pd (4.00 % of mass) and Cu (59.05 % of mass) were found from EDX analysis (Fig. 6)

Table 2- Recyclability of Pd-Cu bimetallic NPs 4 of the coupling reaction between benzoyl chloride and 1-heptyne.

Entry	Run	Time (h)	Yield %
1	Fresh	0.5	90
2	1	0.5	88
3	2	0.5	86
4	3	0.5	82
5	4	0.5	80



Element	(keV)	Mass%	Sigma	Atom%	Compound	Mass%	Cation	K
C K*	0.277	15.72	0.25	35.07				5.6763
N K*	0.392	1.61	0.15	3.30				1.7853
O K	0.525	19.22	0.34	34.44				21.5728
Cu L	0.930	59.05	0.63	26.17				67.8324
Pd L*	2.838	4.00	0.30	0.51				3.1331
Total		100.00		100.00				

Figure 5 SEM image of the recovered NP 4 Figure 6 EDX analysis of the recovered NP 4

In addition, the catalyst's leaching study was performed due to checking heterogeneity. The reaction mixture was analyzed by ICP-MS after isolation of the catalyst and only 0.20 ppm of copper was observed in the reaction mixture. All the results showed that our catalyst was almost similar before the catalyst showed high stability and reusability.

Conclusion

In overview, we have synthesized heterogeneous Palladium-Copper bimetallic nanoparticles by sequential loading method templated by melamine-based dendrimer. Because of the low cost of melamine but still, most effective palladium with bimetallic nanocatalyst and therefore due to make a potentially valuable, recoverable, reusable Pd/Cu bimetallic nanocatalyst, a small quantity of palladium salt were used with a copper salt. All evaluative results (SEM, EDX, XRD, TG & DSC) verify their heterogeneous catalytic activity in the carbon-carbon cross-coupling reaction such as Sonogashira type reaction of the synthesis of ynone and the synthetic path was also observed to be phosphine-ligand free. Additional applications of this heterogeneous catalytic activity in numerous C–C coupling reactions is now under-investigated.

Acknowledgments:

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Conflicts of Interest: The authors confirm no conflicts of interest.

References

1. Wang D, Li Y. Bimetallic nanocrystals: liquid-phase synthesis and catalytic applications. *Adv Mater* (2011); 23(9):1044-60.
2. Aulenta F, Hayes W and Rannard S. Dendrimers: a new class of nanoscopic containers and delivery devices. *Euro Polym J* (2003); 39(9): 1741-1771.
3. Greish K, Fang J, Inutsuka T, Nagamitsu A. and Maeda H. Macromolecular therapeutics. *Clinical pharmacokinetics* (2003); 42(13): 1089-1105.
4. Gupta U, Agashe H B, Asthana A. and Jain N K. Dendrimers: novel polymeric nanoarchitectures for solubility enhancement. *Biomacromolecules* (2006); 7(3): 649-658.
5. Liu M and Fréchet J M, Designing dendrimers for drug delivery. *Pharm sci & techno today* (1999); 2(10): 393-401.
6. Dahlous K, Almarhoon Z, Badjah-Hadj-Ahmed, A Y, AL Othman Z and El-Faham A. Microwave Irradiation Assists the Synthesis of a Novel Series of bis-Arm s-Triazine Oxy-Schiff Base and Oxybenzylidene Barbiturate Derivatives. *Molecules* (2018); 23(11): 2976.
7. Fréchet J.M. Dendrimers and supramolecular chemistry. *Proceedings of the National Academy of Sciences* (2002); 99(8): 4782-4787.
8. Pérez-Temprano M H, Casares J A and Espinet P. Bimetallic Catalysis using Transition and Group 11 Metals: An Emerging Tool for C–C Coupling and Other Reactions. *Chem–A Euro J* (2012); 18(7): 1864-1884.
9. Coq B and Figueras F. Bimetallic palladium catalysts: influence of the co-metal on the catalyst performance. *J Molec Catal A: Chem* (2001); 173(1-2): 117-134.
10. Han S W, Kim Y and Kim K. Dodecanethiol-derivatized Au/Ag bimetallic nanoparticles: TEM, UV/VIS, XPS, and FTIR analysis. *J collid Interf Sci* (1998); 208(1): 272-278.
11. Link S, Wang Z L and El-Sayed M A, Alloy formation of gold– silver nanoparticles and the dependence of the plasmon absorption on their composition. *J Phy Chem B* (1999); 103(18): 3529-3533.
12. Sinfelt J H and Cusumano J A Bimetallic catalysts. *Exxon monograph. Wiley, New* (1983) 44.
13. Sinfelt J H. Ruthenium-copper: a model bimetallic system for studies of surface chemistry and catalysis. *Int Rev Phys Chem* (1988); 7(4): 281-315.
14. Rodriguez J. Physical and chemical properties of bimetallic surfaces. *Surf Sci Rep* (1996); 24(7-8): 223-287.
15. Chinchilla R and Nájera C. The Sonogashira reaction: a booming methodology in synthetic organic chemistry. *Chem Rev* (2007); 107(3): 874-922.
16. Allred G D and Liebeskind L S. Copper-mediated cross-coupling of organostannanes with organic iodides at or below room temperature. *J Am Chem Soc* (1996); 118(11): 2748-2749.

17. Shih Z Y, Wang C W, Xu G and Chang H T. Porous palladium copper nanoparticles for the electrocatalytic oxidation of methanol in direct methanol fuel cells. *J Mater Chem A* (2013); 1(15): 4773-4778.
18. Myers S V, Frenkel A I and Crooks R M. X-ray absorption study of PdCu bimetallic alloy nanoparticles containing an average of ~ 64 atoms. *Chem Mater* (2009); 21(20): 4824-4829.
19. Sengupta D, Saha J, De G and Basu B. Pd/Cu bimetallic nanoparticles embedded in macroporous ion-exchange resins: an excellent heterogeneous catalyst for the Sonogashira reaction. *J Mater Chem A* (2014); 2(11): 3986-3992.



Supporting Information

IR spectra of 2,4,6-tris(di-4-methoxybenzamido)-1,3,5-triazine 3

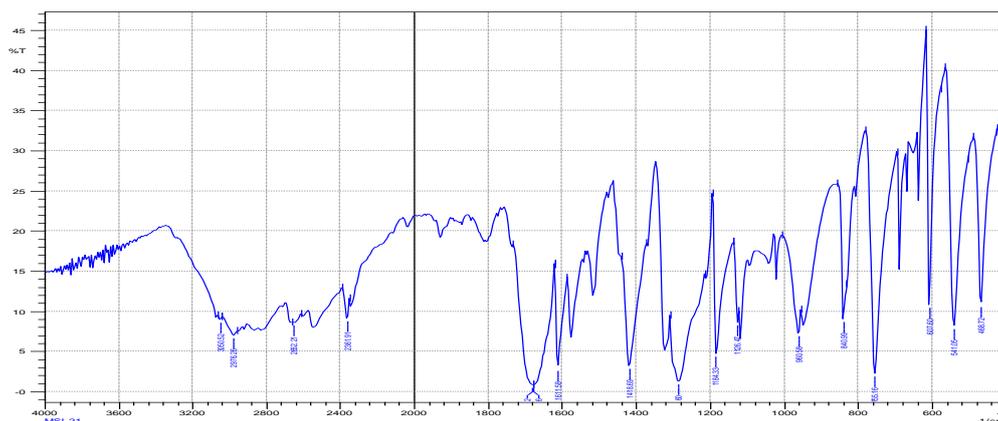


Figure. S1 IR spectra of 2,4,6-tris(di-4-methoxybenzamido)-1,3,5-triazine 3

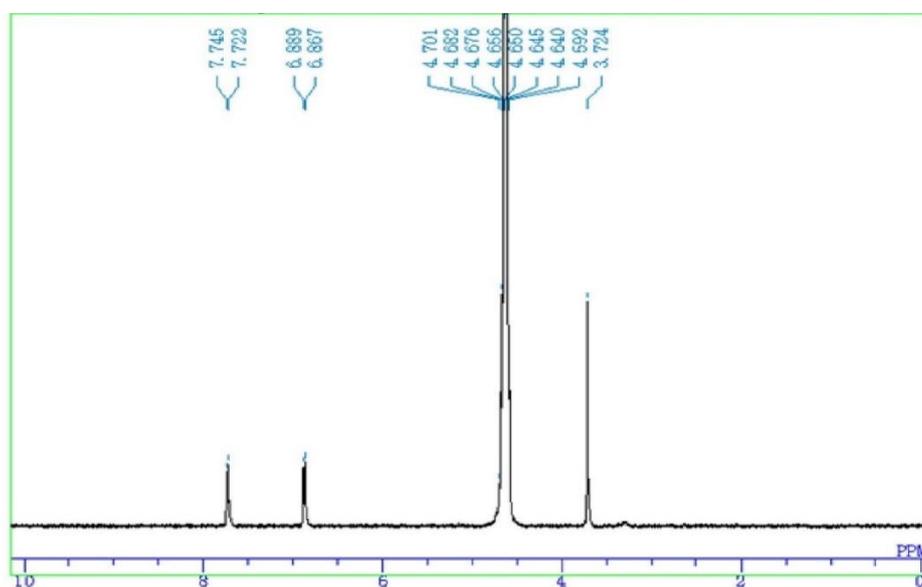


Fig. S2 ¹H NMR spectrum of the compound 3

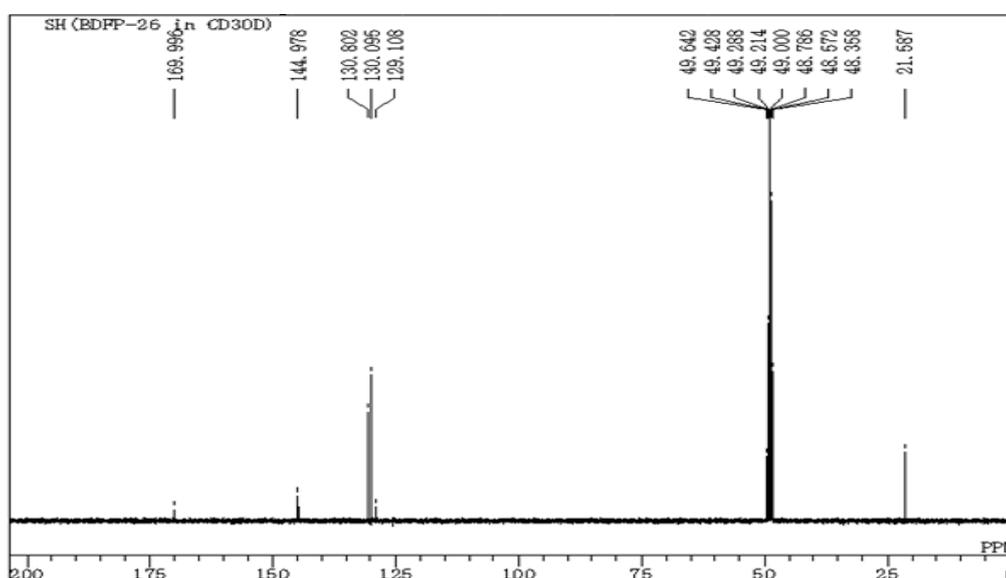


Fig. S3 ¹³C NMR spectrum of the compound 3

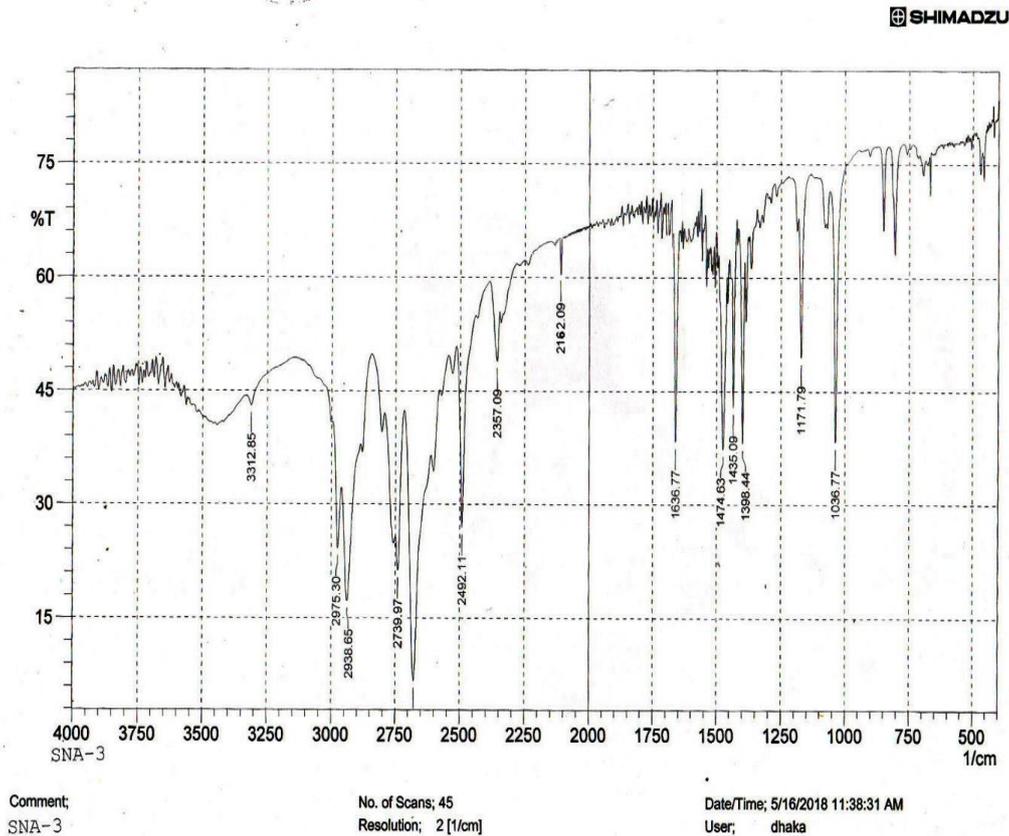


Fig. S4 IR of 1-phenyloct-2-yn-1-one 7

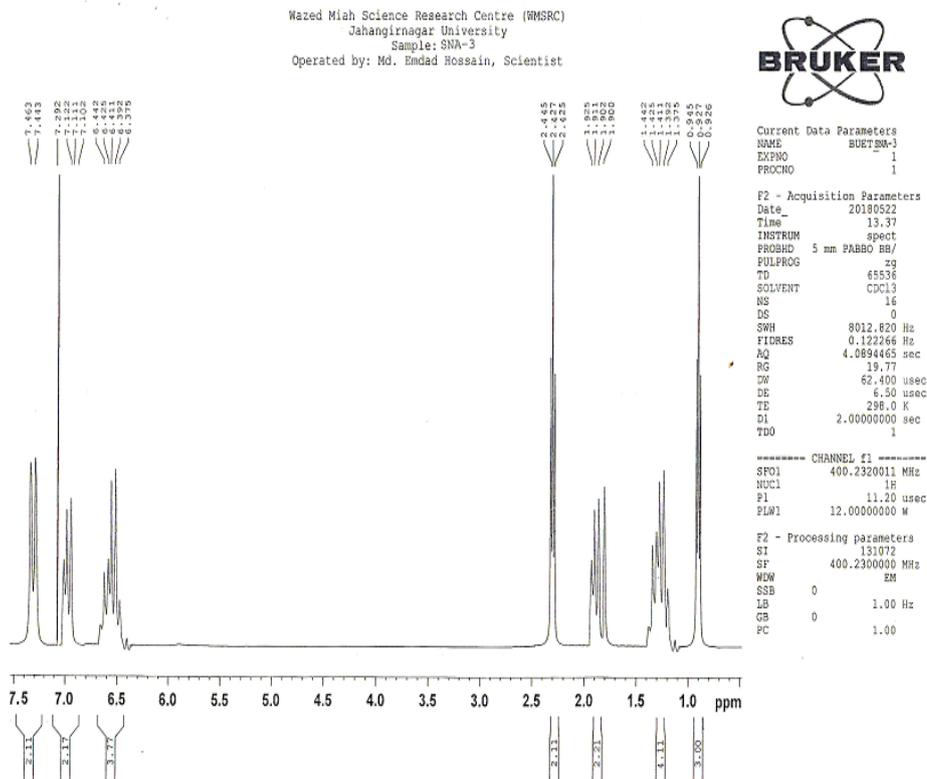
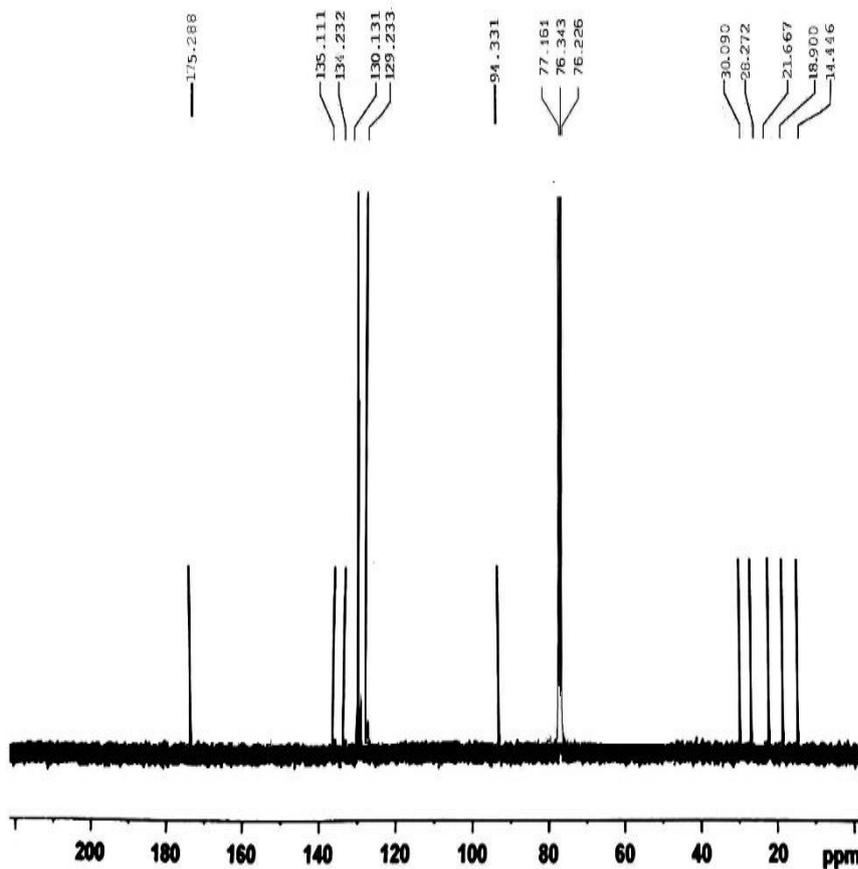


Fig. S5 ¹H nmr of 1-phenyloct-2-yn-1-one 7

Wazed Miah Science Research Centre (WMSRC)
 Jahangirnagar University
 Sample: SNA-3,13C
 Operated by: Md. Emdad Hossain, Scientist



Current Data Parameters
 NAME BUET_SNA-3
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180523
 Time 11.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 25.5
 BW 62.400 usec
 DE 6.50 usec
 TE 298.0 K
 D1 2.0000000 sec
 TDO :

===== CHANNEL f1 =====
 SF01 400.2320011 MHz
 NUC1 13C
 P1 11.20 usec
 PLW1 12.00000000 W

F2 - Processing parameters
 SI 131072
 SF 400.2300000 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

Fig. S6 ¹³C nmr of 1-phenyloct-2-yn-1-one 7