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# Carboxylic acid addition to terminal alkynes utilizing ammonium tagged Hoveyda-Grubbs catalyst supported on magnetically separable core/shell silica: A highly reusable and air compatible catalytic system

Bengi Özgün Öztürk*, Didar Gürcü, Solmaz Karabulut Şehitoğlu**

*Hacettepe University, Faculty of Science, Chemistry Department, 06800, Beytepe, Ankara, Turkey*

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## ABSTRACT

In this study, the performance of ammonium tagged Hoveyda-Grubbs catalyst supported on magnetically separable core/shell silica gel was tested on carboxylic acid addition reactions to terminal alkynes using a variety of carboxylic acid derivatives under air atmosphere. The catalytic system was found to be compatible with air atmosphere and can tolerate even non-degassed solvents. The reaction parameters such as temperature, substrate/catalyst ratio and the effect of carboxylic acid on the selectivity and yield of the reaction were investigated in details. The reaction of arylacetylenes with acetic acid yielded the corresponding E-isomer with conversion values up to 99% with a catalytic loading of 1% Ru. The reusability of the catalyst was tested using acetic acid/benzoic acid and phenylacetylene in toluene at 85 °C under air atmosphere. The catalyst was found to be highly reusable and maintained its activity up to 11th run, reaching a conversion value of 83% with minimum ruthenium leaching.

## 1. Introduction

Carboxylic acid addition reactions to terminal alkynes are of great importance owing to their ability to form unique C—O bonds through the enol-ester formation in an efficient manner. Different transition metals such as Ru, Rh, Ir, Re, Pd, and Au are used efficiently in enol-ester synthesis [1,2]. Among these transition metal-based catalysts, ruthenium complexes were commonly used as highly active and selective catalysts in carboxylic acid addition to terminal alkynes [3–5] (Scheme 1).

The first example of carboxylic acid addition to terminal alkyne reaction was reported by Shvo and Rotem utilizing $Ru_3(CO)_{12}$ as an efficient catalyst [6]. Dixneuf and coworkers demonstrated the effect of the catalyst structure on the selectivity of the addition reaction using $RuCl_2(\eta^6\text{-arene})(PR_3)]$ and $[\{Ru(\mu{-}O_2CH){-}(CO)_2(PPh_3)\}_2$ to obtain Marknovnikov product. On the other hand, $[Ru(\eta^3\text{-}2\text{-}C_3H_4Me)_2\{\kappa^2(P,P)\text{-}Ph_2P(CH_2)_nPPh_2\}]$ yielded mainly Z-isomer of the addition product [7–9]. Recently Stark et al. reported a cationic ruthenium catalyst: $[RuCl(2,6\text{-diacetylpyridine})(PPh_3)_2]BArF$ giving predominantly gem isomer in carboxylic acid addition reactions to alkynes [10]. The dimeric bis(allyl)ruthenium (IV) complex and mononuclear trans-$[RuCl_2(\eta^3{:}\eta^3\text{-}C_{10}H_{16})PPh_3]$ were found to be efficient catalysts for addition reactions of aromatic/aliphatic carboxylic acids to a variety of alkynes such as enynes, diynes and terminal alkynes [11]. $[Ru(dppe)_2(CH_3CN)Cl][BF_4]$ complex was reported as an anti-Markovnikov selective catalyst for carboxylic acid addition reactions to terminal alkynes [12]. In 2016, a highly regioselective protocol (up to 99% Markovnikov addition product) utilizing $[Ru(CO)_2(P(p{-}C_6H_4{-}X)_3)_2(O_2CPh)_2]$ (1a, X = $CF_3$; 1b, X = Cl; 1c, X = H; 1d, X = Me; 1e, X = OMe) for carboxylic acid addition to terminal alkyne reactions was reported [13]. In addition to metathesis reactions, Grubbs type catalysts can be used in various non-metathetical catalytic transformation reactions [14]. The thermolysed Grubbs catalyst was found to be an efficient catalyst for carboxylic acid addition reactions to alkynes giving Markovnikov addition products and the same study also demonstrated the pH-dependent selectivity of the reaction [15]. Our research group reported the activity of Grubbs 2nd generation analog on carboxylic acid addition and terminal alkyne dimerization reactions [16]. The analog of Grubbs catalyst bearing N-heterocyclic carbene ligands showed high Z selectivity yielding Markovnikov products in carboxylic acid addition reactions and can even catalyze the carboxylic acid addition to internal alkynes. A limited amount of study was focused on the carboxylic acid addition to internal alkynes which are

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* Corresponding author.
** Corresponding author.
*E-mail address:* bengi04@hacettepe.edu.tr (B.Ö. Öztürk).

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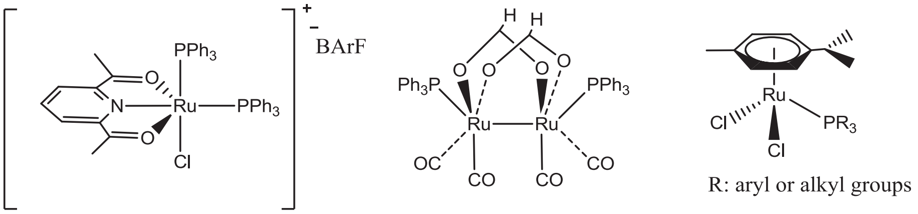


**Scheme 1.** Ruthenium based catalysts for carboxylic acid addition to terminal alkynes.

known as challenging substrates [17–20]. Although a variety of different catalysts were employed in carboxylic acid addition reactions, only a few catalytic systems were found to be reusable [21–23]. A reusable heterogeneous catalyst system based on phosphine modified Ru/$CeO_2$ for carboxylic acid addition reactions to terminal alkynes was reported by Nishiumi et al. [22]. Recently our research group has reported a novel support material based on core/shell structured magnetically separable silica gel for ammonium tagged Hoveyda-Grubbs catalysts [24] as a robust and highly reusable catalytic system for ring-closing metathesis reactions [25]. Here we report the activity of ammonium tagged Hoveyda-Grubbs catalysts immobilized on magnetically separable core/shell silica supports for the carboxylic acid addition reactions to arylacetylenes. The homogenous ammonium tagged Hoveyda-Grubbs catalyst and its supported analog was compared in means of selectivity. The supported catalyst Ru-1@SiO2 catalyzed the acetic acid/benzoic acid addition reactions to phenylacetylene up to 11th run without any significant decrease in both activity and selectivity.

## 2. Experimental

Otherwise noted all chemicals were purchased from Sigma-Aldrich and used as received. Toluene was dried over Na wire/benzophenone and distilled under a nitrogen atmosphere. Ammonium tagged Hoveyda-Grubbs catalyst; (1,3-bis(mesityl)imidazolidin-2-ylidene)(2-((1-(methoxy(methyl)amino)-1-oxopropan-2-yl)oxy)benzylidene)ruthenium(VI) chloride were purchased from Strem and used as received. Core/Shell structured magnetically separable silica gels and the supported catalyst; Ru-1@$SiO_2$-magn. were synthesized according to the literature [25]. Gas chromatography-mass spectrometry (GC-MS) analyses were performed with a Shimadzu GC-MS 2010 Plus using a Restek Rxi-5 Sil column (30 m × 0.25 mm × 0.25 μm) and temperature range of 50–320 °C with a constant helium flow rate of 1 ml/min $^1H$ and $^{13}C$ NMR spectra were recorded at 25 °C with a Bruker GmbH 400-MHz high performance digital FT-NMR spectrometer using $CDCl_3$ as the solvent.

### 2.1. The representative protocol for carboxylic acid addition to terminal alkynes

Alkyne (0.18 mmol) and carboxylic acid (1.8 mmol) was added to the Schlenk reactor. Toluene (1 mL) and Ru-1@$SiO_2$-magn. (20 mg, 0.0018 mmol Ru) were added to the reaction medium and reaction mixture were stirred at 85 °C under air atmosphere. Aliquots taken from the reaction mixture was diluted with methanol and analyzed by GC-MS using n-tetradecane as an internal standard. Once the reaction was finished, the catalyst was separated from the reaction mixture with the aid of a magnet. The volatiles were removed by high vacuum. The remaining solution was diluted by dichloromethane and extracted with saturated $NaHCO_3$ solution followed by extraction with water. The organic layer was dried with $MgSO_4$ and then volatiles were removed by high vacuum. The resulting final product was characterized by means of $^1H$ NMR spectroscopy and GC-MS analysis.

### 2.2. The representative protocol for reusability tests

Alkyne (0.18 mmol) and carboxylic acid (1.8 mmol) were added to the Schlenk reactor. Toluene (1 mL) and Ru-1@SiO2-magn (20 mg, 0.0018 mmol Ru) were added to the reaction medium and reaction mixture were stirred at 85 °C under air atmosphere. Aliquots taken from the reaction mixture was diluted with methanol and analyzed by GC-MS using n-tetradecane as the internal standard. Once the reaction was finished, the catalyst was separated from the reaction mixture with the aid of a magnet. The catalyst was kept in the reactor while the solution phase was taken out of the reactor via Pasteur pipet. The catalyst was washed with toluene and dichloromethane and then reused in carboxylic acid addition reactions.

## 3. RESULTS&DISCUSSION

Magnetic mesoporous core/shell silica gels and Ru-1@$SiO_2$-magn. were synthesized according to the literature (Fig. 1) [25]. Ru-1 (commercial name; Aquamet) was supported on core/shell silica gel (surface area: 630 $m^2$/g; pore size/volume; 2.88 nm/0.393 $cm^3$) in $CH_2Cl_2$ medium. The ruthenium content of the Ru-1@$SiO_2$-magn. was found to be 0.090 mmol Ru/g as determined by ICP-MS.

First tests were carried out using phenylacetylene and acetic acid with a catalytic loading of 1% Ru in toluene at 85 °C. In the first trial, acetic acid/phenylacetylene (mol/mol) ratio was varied between 1/1, 2/1, 5/1 and 10/1 and results were given in Table 1.

As it can be seen in Table 1, alkyne dimerization reaction can be suppressed by increasing acid/alkyne ratio. The amount of dimerization product had significantly decreased when acid/alkyne ratio was increased from 1/1 to 10/1. Dimerization reaction was completely suppressed at the acid/alkyne ratio of 10/1. On the next trial, the effect of solvent on carboxylic acid addition reactions was tested using both homogeneous and supported catalysts; Ru-1 and

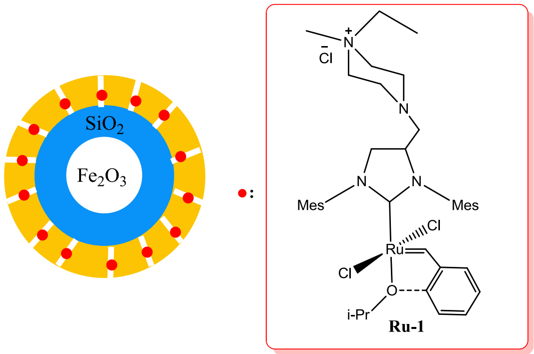
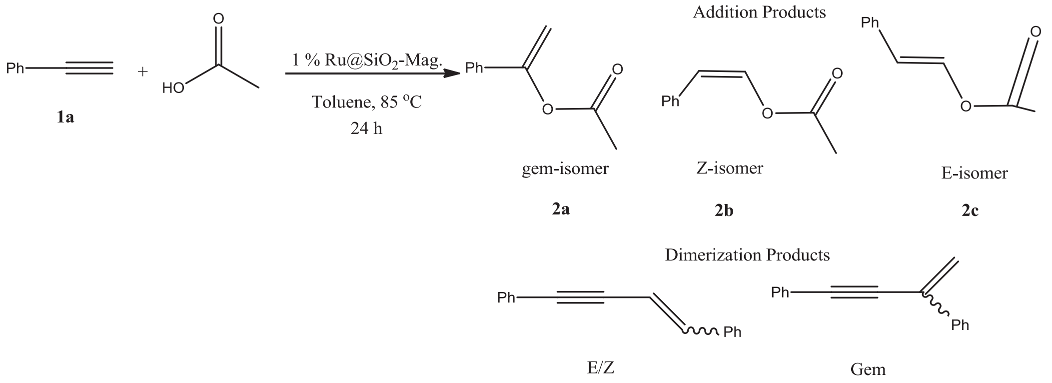


**Fig. 1.** Ru-1 and Ru-1@$SiO_2$-magn. catalysts.

**Table 1**
The effect of acid/alkyne ratio on carboxylic acid addition reaction.2



| Entry[a] | Acid/Alkyne | Conversion[b] % | Addition[c] Product (%) | 2a (%) (gem)[d] | 2b (%) (Z)[d] | 2c (%) (E)[d] | Dimerization[c] Product (%) | Z/E/Gem |
|---|---|---|---|---|---|---|---|---|
| **1** | 1/1 | 90 | 36 | 15 | 23 | 62 | 54 | 60/40/0 |
| **2** | 2/1 | 95 | 66 | 16 | 22 | 62 | 29 | 55/45/0 |
| **3** | 5/1 | 99 | 84 | 15 | 22 | 63 | 15 | 53/47/0 |
| **4** | 10/1 | 99 | 99 | 11 | 26 | 63 | – | – |

[a] Reaction time: 24 h.
[b] Determined by GC-MS using n-tetradecane as internal standard.
[c] Determined by GC-MS.
[d] İsomer ratios was determined using integration values of GC peaks of corresponding isomers. Alternatively, isomer ratio was also determined by $^1H$ NMR using the integration value of olefinic peaks of E, Z, gem isomers. E % = (Integration E/(Integration E + Integration Z + Integration gem))x100.

Ru-1@$SiO_2$-magn. in water, methanol, methanol/water (v/v: 1/1), toluene and no solvent media. As it can be seen in Table 2, the reaction exhibited no selectivity in means of gem, Z and E isomers of addition products in both water and water/methanol mixture. Gem/Z/E products at ratios of 16/41/43 (74% conversion in water) and 20/42/38 (60% conversion in methanol/water) were obtained using Ru-1@$SiO_2$-magn. The reaction proceeded more selectively in both toluene and no solvent media, reaching conversion values up to 99%. The reaction yielded E isomer as the main product (Table 2, Entry 6, 63% E) using Ru-1@$SiO_2$-magn. Homogenous catalyst Ru-1 exhibited low selectivity (gem/Z/E %; 44/26/30%) in addition reactions when compared to Ru-1@$SiO_2$-magn.

$^1H$ NMR spectrum of the reaction product also confirmed the presence of both E, Z and gem isomers (Fig. 2). Olefinic proton signals of E and Z isomer appeared at 7.84, 7.49 and 6.33, 5.69 ppm as doublets. Gem signals were observed at 5.40 and 4.95 ppm as singlets. $CH_3COO$– signals of E, Z and gem isomers appeared at 2.20, 2.27 and 2.36 ppm as singlets. Aromatic signals of both isomers were overlapped at 7.08–7.38 ppm region.

The performance of the catalytic system was tested on both air and nitrogen atmosphere using degassed dried toluene and non-degassed neat toluene at 85 °C and results were given in Table 3. No significant difference in means of conversion value and selectivity was observed when the reaction was carried out under air and nitrogen atmosphere. On the other hand, when a trace amount of water was present in the reaction media, acetophenone formation was inevitable.

Phenylacetylene (1a), 2-ethynyltoluene (1b), 4-ethynyltoluene (1c), 2-ethynylanisole (1d) and 4-ethynylanisole (1e) were reacted with acetic acid (Ac1) and benzoic acid (Ac2) in toluene at 85 °C with a catalytic loading of 1% Ru (Table 4). No significant selectivity difference was observed when using acetic acid and benzoic acid. In all cases, higher conversion values were obtained even using sterically hindered ortho-substituted alkynes (1b and 1d; 90% and 99%). E-isomer was formed as the major product during addition reaction. No trace amount of dimerization product was observed during the reaction. To further investigate the compatibility of the reaction with different acids; trans-cinnamic acid, trans-2-octenoic acid, 4-cyclooctene carboxylic acid were used on addition reactions of phenylacetylene under same reaction conditions and also

**Table 2**
Comparison of Ru-1 and Ru-1@$SiO_2$ on acetic acid addition to phenylacetylene using different solvents.

| *Entry*[a] | *Catalyst* | *Solvent* | *Conversion %*[b] | *gem %* | *Z %* | *E %* |
|---|---|---|---|---|---|---|
| ***1*** | Ru-1 | water | 94 | 38 | 47 | 15 |
| ***2*** | Ru-1@$SiO_2$-magn. | water | 74 | 16 | 41 | 43 |
| ***3*** | Ru-1 | MeOH/water (v/v: 1/1) | 70 | 27 | 40 | 33 |
| ***4*** | Ru-1@$SiO_2$-magn. | MeOH/water (v/v: 1/1) | 60 | 20 | 42 | 38 |
| ***5*** | Ru-1 | Toluene | 95 | 44 | 26 | 30 |
| ***6*** | Ru-1@$SiO_2$-magn. | Toluene | 99 | 11 | 26 | 63 |
| ***7*** | Ru-1 | No solvent | 94 | 36 | 27 | 37 |
| ***8*** | Ru-1@$SiO_2$-magn. | No solvent | 99 | 10 | 28 | 62 |

[a] The reaction conditions are as follows: A reactor was charged with 1% Ru (0.018 mmol), phenylacetylene (1.8 mmol) and acetic acid (18 mmol) in toluene (1 mL) and reacted under air atmosphere at 85 °C for 24 h.
[b] No dimerization product was observed.

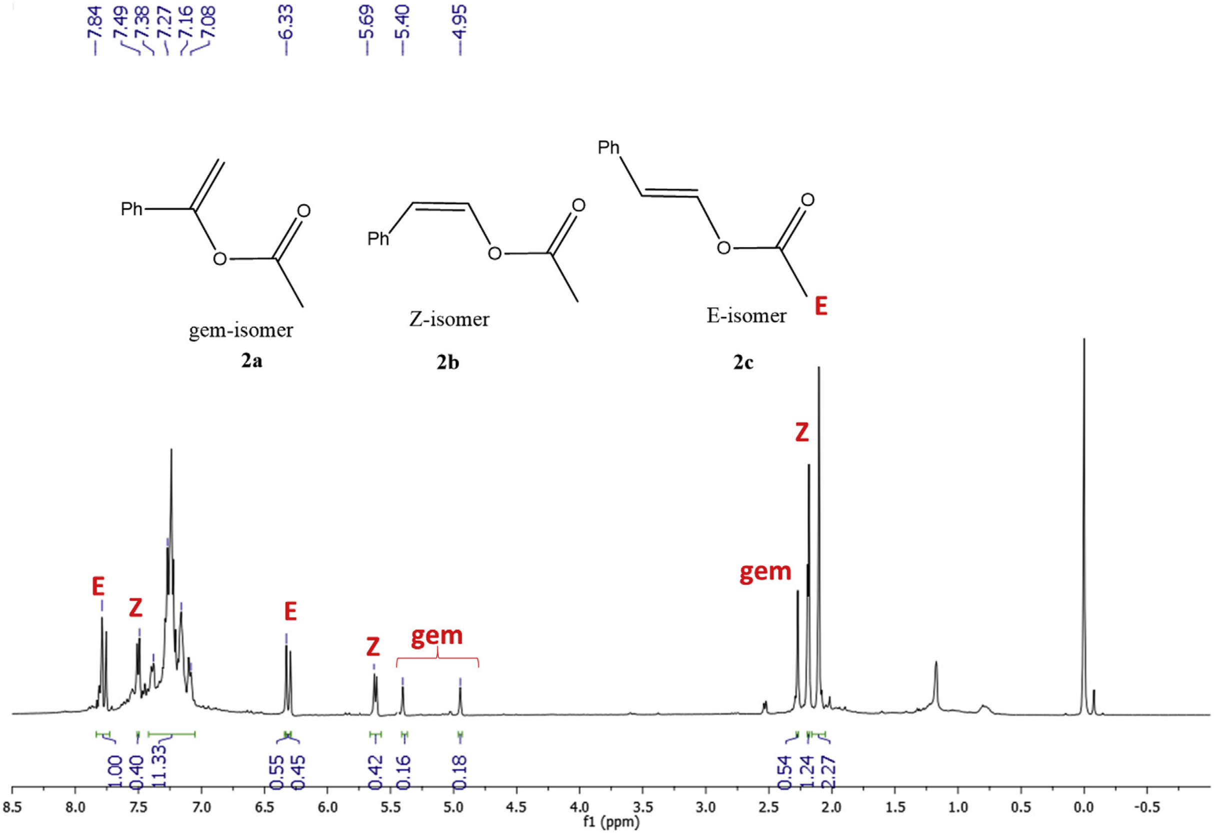


**Fig. 2.** $^{1}$H NMR spectrum of 2a/2b/2c in $CDCl_3$.

**Table 3**
Acetic acid addition to phenylacetylene under different reaction atmospheres.

| *Entry*[a] | *Atmosphere* | *Solvent* | *Conversion %*[b] | *Addition% (gem/Z/E)* | *Acetophenone %* |
|---|---|---|---|---|---|
| *1* | $N_2$ | Degassed dry toluene | 99 | 100 (11/26/63) | – |
| *2* | $N_2$ | Non-degassed toluene | 99 | 100 (11/27/62) | – |
| *3* | Air | Non-degassed toluene | 99 | 100 (11/26/63) | – |
| *4* | Air | Non-degassed toluene + trace amount of water | 90 | 84 (14/26/60) | 16 |

[a] The reaction conditions are as follows: 1% Ru (0.018 mmol), phenylacetylene (1.8 mmol) and acetic acid (18 mmol) in solvent (2 mL) under air atmosphere at 85 °C for 24 h.

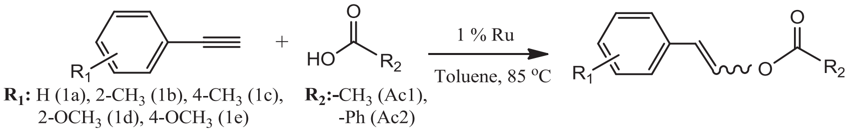
configuration retentions of carboxylic acids were investigated. All addition reactions were reached conversion values up to 99% under pre-determined reaction conditions (Table 5). Trans-2-octenoic acid, and trans-cinnamic acid retained their trans configurations during the reaction as confirmed by $^{1}$H NMR analysis (supporting information, Figs. S3–4). Although cyclooctene derivatives can undergo ring opening metathesis polymerization reactions in the presence of Grubbs type ruthenium alkylidene complexes, 4-cyclooctenoic acid didn't undergo any ring opening reaction during the carboxylic acid addition reactions (supporting information (SI), Fig. S5). The selectivity of the reaction was increased in the case of 4-cyclooctene carboxylic acid and trans-2-cinnamic acid.

Reusability of the catalyst was tested on both benzoic acid and acetic acid addition reactions to phenylacetylene under pre-determined reaction conditions. The catalyst was separated by applying magnetic field with an aid of a magnet. The catalyst was washed with toluene/dichloromethane twice and then used for the reaction. The reaction was terminated when no increase in conversion values was observed. As it can be seen on both Graph 1 and 2, Ru-1@$SiO_2$-magn. is highly usable in both acetic and benzoic acid addition to phenylacetylene (Graph 1 and 2). No significant activity loss and catalyst leaching were observed up to 11th catalytic turn in both reactions. Ruthenium leaching was found to be 1% Ru after 11th run as determined by ICP-MS.

## 4. Conclusion

The ammonium tagged Hoveyda-Grubbs catalyst (Ru-1) supported on magnetically separable core/shell silica gels was found to be an efficient, robust and air tolerant catalytic system for carboxylic acid addition to terminal alkynes. The reaction showed high E selectivity using arylacetylene derivatives with different carboxylic acids such as acetic acid, benzoic acid, trans-cinnamic acid, trans-2-octenoic acid and 4-cyclooctenecarboxylic acid. The olefinic groups of trans-cinnamic acid, trans-2-octenoic acid and 4-cyclooctenecarboxylic acid remained unreacted and preserved trans configuration during the addition reaction. The effect of support material on the selectivity of the reaction was investigated

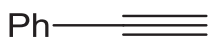
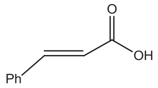
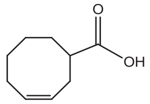
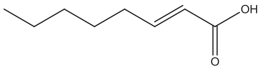
**Table 4**
Carboxylic acid addition to arylacetylenes.



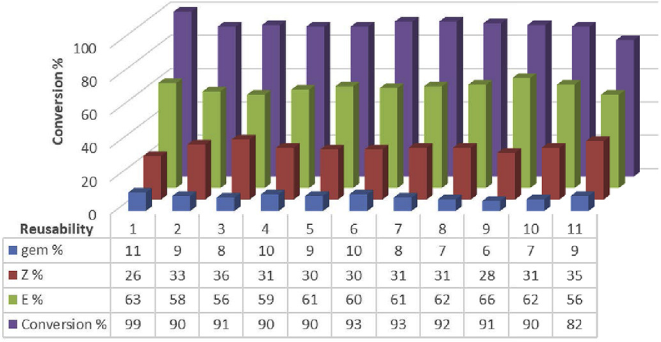
| Entry[a] | Alkyne | Temperature(°C) | Acid | Conversion %[b] | Gem % | Z % | E % |
|---|---|---|---|---|---|---|---|
| 1 | 1a | 50 | Acetic acid (Ac1) | 20 | 12 | 25 | 63 |
| 2 | 1a | 85 | | 99 | 11 | 26 | 63 |
| 3 | 1a | 110 | | 70 | 12 | 30 | 58 |
| 4 | 1b | 85 | | 90 | 10 | 23 | 67 |
| 5 | 1c | 85 | | 99 | 7 | 24 | 69 |
| 6 | 1d | 85 | | 99 | 11 | 28 | 61 |
| 7 | 1e | 85 | | 99 | 9 | 27 | 64 |
| 8 | 1a | 85 | Benzoic acid (Ac2) | 99 | 9 | 29 | 62 |
| 9 | 1b | 85 | | 99 | 15 | 26 | 59 |
| 10 | 1c | 85 | | 98 | 23 | 22 | 55 |
| 11 | 1d | 85 | | 99 | 10 | 28 | 62 |
| 12 | 1e | 85 | | 99 | 9 | 27 | 64 |

[a] Please see experimental section for the synthesis protocol.
[b] No dimerization product was observed.

**Table 5**
The addition reaction of phenylacetylene with different carboxylic acids.

| Alkyne[a] | Acids | Conversion %[b] | gem/Z/E (%)[b] |
|---|---|---|---|
|  |  | 99 | 9/17/74 |
| |  | 99 | 3/19/78 |
| |  | 95 | 7/23/70 |

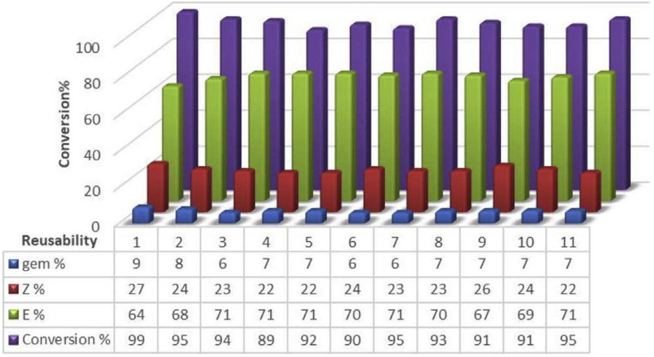
[a] The reaction conditions as follows: A glass reactor was charged with 1% Ru (0.018 mmol), phenylacetylene (1.8 mmol) and corresponding carboxylic acid (18.0 mmol) in toluene (2 mL) under air atmosphere and stirred for 24 h at 85 °C.
[b] Determined by GC-MS using n-tetradecane as internal standard.



| Reusability | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 🟦 gem % | 11 | 9 | 8 | 10 | 9 | 10 | 8 | 7 | 6 | 7 | 9 |
| 🟥 Z % | 26 | 33 | 36 | 31 | 30 | 30 | 31 | 31 | 28 | 31 | 35 |
| 🟩 E % | 63 | 58 | 56 | 59 | 61 | 60 | 61 | 62 | 66 | 62 | 56 |
| 🟪 Conversion % | 99 | 90 | 91 | 90 | 90 | 93 | 93 | 92 | 91 | 90 | 82 |

**Graph 1.** Reusability of Ru-1@SiO2-magn on acetic acid addition to phenylacetylene.

by comparing the selectivity of both homogenous catalyst Ru-1 and its supported analog Ru-1@$SiO_2$-magn, the latter one was found to be an E-selective catalyst for addition reactions. Ru-1@$SiO_2$-magn. catalyzed the addition reactions of acetic acid and benzoic acid to phenylacetylene up to 11th run without any significant activity loss.



| Reusability | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 🟦 gem % | 9 | 8 | 6 | 7 | 7 | 6 | 6 | 7 | 7 | 7 | 7 |
| 🟥 Z % | 27 | 24 | 23 | 22 | 22 | 24 | 23 | 23 | 26 | 24 | 22 |
| 🟩 E % | 64 | 68 | 71 | 71 | 71 | 70 | 71 | 70 | 67 | 69 | 71 |
| 🟪 Conversion % | 99 | 95 | 94 | 89 | 92 | 90 | 95 | 93 | 91 | 91 | 95 |

**Graph 2.** Reusability of Ru-1@SiO2-magn. on benzoic acid addition to phenylacetylene.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jorganchem.2019.01.005.

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