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The Synthesis of Quinazolinones from Olefins, CO and Amines over Heterogeneous Ru-clusters/Ceria Catalyst

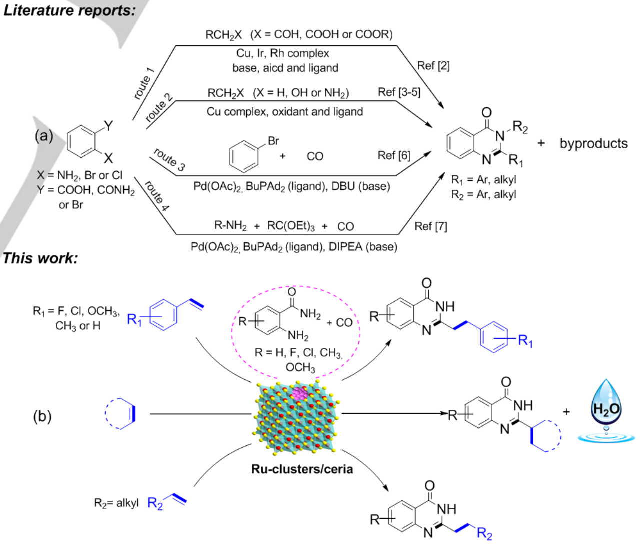
*Jinghua An, Yehong Wang, Zhixin Zhang, Zhitong Zhao, Jian Zhang and Feng Wang**

***Abstract:** Quinazolinones, one class of important heterocyclic compounds, have been widely used in pharmaceuticals due to their biological activities. However, until now, to synthesize quinazolinones efficiently and economically is still a challenge. Herein, a novel synthetic approach is developed to produce quinazolinones from olefins, CO and amines over heterogeneous Ru-clusters/ceria catalyst in the absence of acids, bases and oxidants. Furthermore, H_2O is generated as the only by-product. A series of quinazolinones with either aromatic or non-aromatic substituents, can be obtained in up to 99% isolated yields. The catalysis here is truly heterogeneous and Ru-clusters/ceria can be reused for at least four times. The analysis of E-factor (environmental impact factor) for the synthesis of 2-ethyl quinazolinone is conducted and it suggests that this system is more friendly to the environment compared to other processes reported previously.*

Quinazolinones are one class of important six-membered nitrogen heterocycles and have been widely used in anticancer, antimalarial, anti-inflammatory and antituberculosis due to their diverse biological activities.[1] Traditionally, they are synthesized via the acid/base-promoted condensation reactions from aldehydes, carboxylic acids, esters with amides (Scheme 1(a), route 1).[2] However, these methods generally suffer from drawbacks such as the complex homogeneous catalytic systems or expensive feedstocks. Recent examples of oxidative condensation reactions from alcohols,[3] methyl arenes[4] and amines[5] often require excess amounts of oxidants (Scheme 1(a), route 2). Wu and coworkers reported a palladium-catalyzed carbonylation reaction to synthesize quinazolinones from *o*-aminobenzamides, bromobenzenes and carbon monoxide (CO) (Scheme 1(a), route 3).[6] Only 2-aryl quinazolinones could be obtained via this method. In addition, they also reported the synthesis of quinazolinones from 2-bromoanilines, trimethyl orthoformate, amines and CO, but ligands and alkaline additives were indispensable (Scheme 1(a), route 4).[7] Therefore, to develop an efficient and economical method to synthesize quinazolinones is still a challenge.

Olefins, especially alpha-olefins, are important hydrocarbons with wide sources. For example, ethylene and propylene can be obtained from coal,[8] biomass,[9] methanol[10] and syngas.[11] As building blocks, olefins have been used in the synthesis of high value-added chemicals such as esters,[12] amides[13] and aldehydes.[14]. Further extending the transformation of olefins not only has great significance in academic research, but also presents broad prospects in industrial application.

Herein, we developed a novel method for the synthesis of quinazolinones from olefins, CO and amines over heterogeneous Ru-clusters/ceria catalyst (Scheme 1(b)). Compared with the processes reported previously, there are some significant features listed in the following: 1) high activity and wide scope of substrates. A series of quinazolinones with either aromatic or non-aromatic substituents can be obtained in up to 99% isolated yields; 2) easy to be scaled up. 12-fold scale transformation of *o*-aminobenzamide was carried out and 1.042 g of 2-ethyl substituted quinazolinone could be obtained in 99% isolated yield; 3) heterogeneous catalysis and good stability of catalyst. The catalysis here is truly heterogeneous and the catalyst can be reused for at least four times; 4) friendly to environment. The values of s*E*-factor, c*E*-factor and *E*-factor are much lower than that of other processes reported previously.



Scheme 1. Different catalytic systems for the synthesis of quinazolinones.

Initially, the synthesis of 2-ethyl substituted quinazolinone (**3**) from ethylene (**2**), CO and *o*-aminobenzamide (**1**) was carried out with tetrahydrofuran (THF) as solvent (Table 1). No target product (**3**) was obtained in the absence of catalyst (Table 1, entry 1). No reaction occurred when $Pd(OAc)_2$, the most efficient catalyst reported,[6] was used as catalyst even increasing the reaction temperature from 120 °C to 160 °C (Table 1, entries 2-3). In our previous work, we have reported the excellent catalytic performance of Ru-clusters/ceria in the carbonylation reaction transforming olefins to esters.[15] Thus here, Ru-clusters/ceria was also applied in the synthesis of quinazolinones from olefins, CO and amines. The results showed that the reaction could

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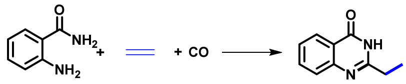
proceed smoothly in 38% GC yield of product **3**. And then it achieved higher than 99% when increasing the temperature to 160 °C (Table 1, entries 4-7). By comparison, $RuCl_3{\cdot}nH_2O$, the precursor of Ru for Ru-clusters/ceria, and ceria support showed poor catalytic performances (Table 1, entries 8-9). These results suggested that Ru-clusters/ceria is the most efficient catalyst in the synthesis of quinazolinone. The removal of Ru-clusters/ceria catalyst in 1 h upon ~ 40% yield of **3** nearly stopped the reaction, suggesting the real heterogeneous catalysis of this reaction over Ru-clusters/ceria (Figure S1(a)). The Ru-clusters/ceria catalyst could be reused for at least four times with slight decrease in activity, suggesting the stability of the catalyst (Figure S1(b)). Additionally, we hypothesized that the solvent tetrahydrofuran (THF) might join the reaction. And then this possibility was excluded by using THF-D_8 as solvent instead of THF, because no deuterium-labelled products were observed (Figure S2).

Table 1. Synthesis of 2-ethyl substituted quinazolinone (**3**) from ethylene (**2**), CO and *o*-aminobenzamide (**1**).[a]



Entry	Catalyst	Temperature / °C	Yield of **3** / %
1	No catalyst	160	0
2[b]	$Pd(OAc)_2$	120	0
3[b]	$Pd(OAc)_2$	160	0
4	Ru-clusters/ceria	130	38
5	Ru-clusters/ceria	140	76
6	Ru-clusters/ceria	150	83
7	Ru-clusters/ceria	160	>99
8[b]	$RuCl_3{\cdot}nH_2O$	160	13
9	ceria	160	0

[a] Reaction conditions: catalyst (0.1 g), **1** (0.5 mmol), ethylene (0.5 MPa), CO (0.5 MPa), THF (2.0 mL), 12 h. Yield was determined by GC and calculated based on **1**. [b] Catalyst (0.02 mmol).

Then, ^{13}C NMR spectroscopy was used to track the reaction (Figure 1). Initially, before the reaction, a series of signals were observed, all of which were assigned to the chemical shift of C atoms for substrate (**1**). Additionally, no impurity was found (Figure 1(a)). Then, the reaction was carried out at 160 °C for 2 h and the reaction mixture was obtained to be examined by ^{13}C NMR (Figure 1(b)). Except for the signals assigned to the chemical shift of C atoms for the unreacted substrate (**1**), a number of new signals could be also observed. By comparison, partial new signals located at 164.2, 157.6, 149.3, 134.8, 127.1, 126.4, 126.3, 120.5, 29.1 and 11.5 ppm were assigned to the chemical shift of C atoms for product (**3**) (Figure 1(c)). Consideration of the high selectivity in the synthesis of **3** in the Ru-clusters/ceria catalytic system (Table 1, entry 7), we proposed the left new signals at 171.9, 171.5, 139.7, 133.7, 132.2, 126.5, 121.5, 120.4, 30.5, 8.6 ppm were probably assigned to the C atoms for the reaction intermediate. According to the references, the signals at 30.5 and 8.6 ppm were assigned to the chemical shift of C atoms for ethyl.[2i,4] And the signals appearing at 139.7, 133.7, 132.2, 126.5, 121.5 and 120.4 ppm were assigned to the chemical shift of C atoms for benzene ring.[2a] Two signals at 171.9 and 171.5 ppm were assigned to the chemical shift of C atoms for carbonyl group.[16] On the basis of the assignments of these signals, we concluded that the reaction intermediate was probably the amide species (**4**), which was generated via the carbonylation of N-H for $–NH_2$ group connected directly to benzene ring. The generation of amide species during the reaction as the reaction intermediate is also verified by the result of the time course for the synthesis of **3** over Ru-clusters/ceria catalyst (Figure S3).

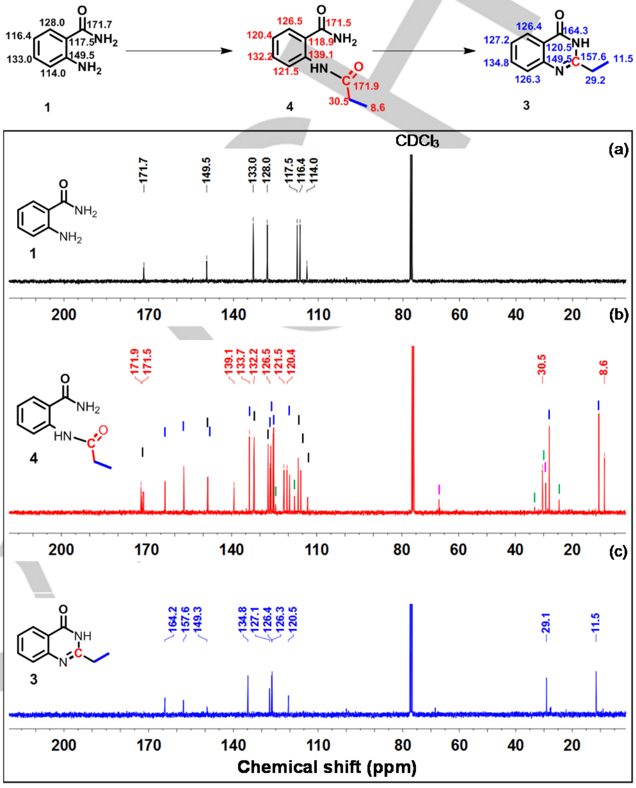
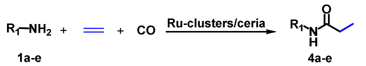
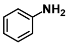
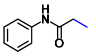
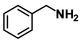
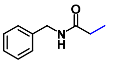
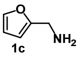
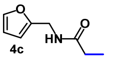
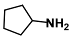
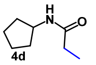
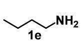
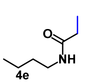


Figure 1. ^{13}C NMR of the substrate **1** (a), the reaction mixture obtained after reaction at 160 °C for 2 h (b) and product **3** (c). The symbols of |, |, | and | shown in Figure 1(b) were the assignments for substrate **1**, product **3**, the trace amount of THF and trace amount of 2,6-di-tert-butyl-4-methylphenol (BHT) in solvent, respectively.

Table 2. Synthesis of amides from ethylene, CO and various amines.[a]



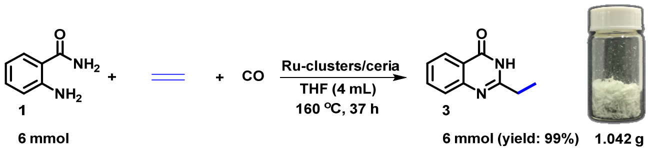
Entry	Amine	Product	Yield[b] (%)
1	 **1a**	 **4a**	78 67[c]
2	 **1b**	 **4b**	>99
3	 **1c**	 **4c**	>99
4	 **1d**	 **4d**	>99
5	 **1e**	 **4e**	>99

[a] Reaction conditions: Ru-clusters/ceria (0.2 g), amine (0.5 mmol), ethylene (0.5 MPa), CO (0.5 MPa), THF (3.0 mL), 160 °C, 24 h. [b] GC yield. [c] Isolated yield. ^{1}H NMR and ^{13}C NMR spectra of **4a** were shown in Supporting Information.

When aniline was used as the substrate to react with ethylene and CO, N-phenyl propionamide (**4a**) could be successfully obtained in 67% isolated yield (Table 2. entry 1). Besides, other amines including benzylamine, furfuryl amine, cyclohexylamine and *n*-butylamine were also tolerated and the corresponding amides were obtained in high yields (>99%) (Table 2. entries 2-5). These results confirmed that it is feasible for the generation of intermediate amide over Ru-clusters/ceria catalyst. And then the target product quinazolinone was easily obtained via the dehydration of intermediate amide, which could be catalyzed by acid sites on Ru-clusters/ceria.[17]

To show the practical value of the present procedure, 12-fold scale transformation of substrate (**1**) was carried out and 1.042 g of product (**3**) could be obtained in 99% isolated yield (Scheme 2).

Then, s*E*-factor, c*E*-factor and *E*-factor, metrics to quantify the economy and sustainability of a process,[18] were used to compare the synthesis of **3** in this work with that in other processes reported previously. And the results showed that the values of s*E*-factor, c*E*-factor and *E*-factor were much lower than that of processes reported (Ref [2i], [2m], [2j], [4] and [2n]), suggesting that this process produces less waste (Figure 2, Table S2-S3).



Scheme 2. The scaled-up reaction to synthesize product (**3**).

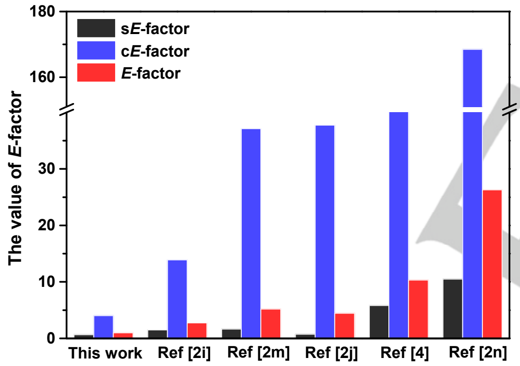
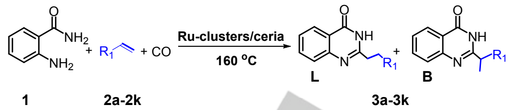
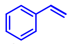
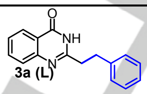
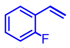
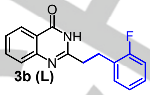
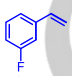
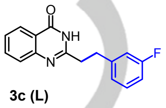
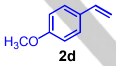
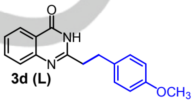
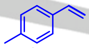
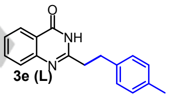
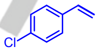
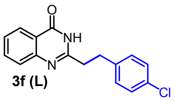
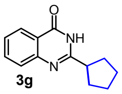
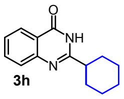
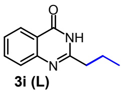
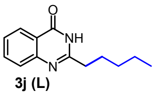
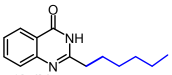


Figure 2. s*E*-factor, c*E*-factor and *E*-factor analysis of the synthesis of product (**3**). Calculation details were shown in Supporting Information.

Next, the scope of olefins were examined for this reaction. Various styrenes with electron-donating (-OCH_3 or -CH_3) or electron-withdrawing groups (-F or -Cl) reacted with substrate (**1**) and CO to give 72-94% isolated yields. And the linear product was majorly generated (Table 3, entries 1-6). Higher selectivities (95% and 86%) of linear products were achieved when *ortho* or *meta*-substituted styrenes were used as substrates. Moreover, cycloolefins proved to be efficient coupling partners and were converted into the corresponding quinazolinones in 79% yield for cyclopentene and 51% yield for cyclohexene (Table 3, entries 7-8). Even in the transformation of alpha-olefins, the corresponding quinazolinones could be achieved in 96% yield starting from 1-propylene, 57% yield from 1-pentene and 37% yield from 1-hexene (Table 3, entries 9-11). To the best of our knowledge, this is the first example to synthesize quinazolinones employing alpha-olefins as the substrates over a heterogeneous catalyst.

Table 3. Substrate scope of olefins.[a]



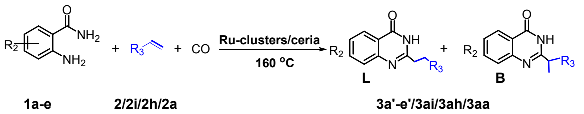
Entry	Olefin	Product	Yield[b] (%) (**L**/**B**)
1	 **2a**	 **3a (L)**	**77 (79/21)**
2	 **2b**	 **3b (L)**	**81 (86/14)**
3	 **2c**	 **3c (L)**	**94 (95/5)**
4	 **2d**	 **3d (L)**	**72(58/42)**
5	 **2e**	 **3e (L)**	**76 (64/16)**
6	 **2f**	 **3f (L)**	**80 (64/16)**
7[c]	 **2g**	 **3g**	**79**
8[c]	 **2h**	 **3h**	**51**
9[d]	 **2i**	 **3i (L)**	**96 (56/44)**
10[c]	 **2j**	 **3j (L)**	**57 (83/17)**
11[e]	 **2k**	 **3k (L)**	**37 (67/33)**

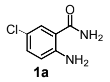
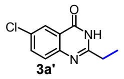
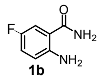
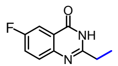
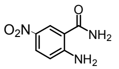
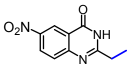
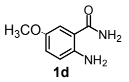
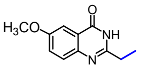
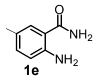
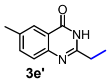
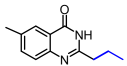
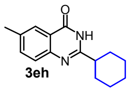
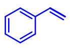
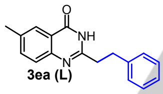
[a] Reaction conditions: Ru-clusters/ceria (0.2 g), **1** (0.5 mmol), olefin (2.0 mmol), CO (1.0 MPa), THF (3.0 mL), 160 °C, 16 h. **L** and **B** represent linear and branched product, respectively. [b] Isolated yield of quinazolinones including linear (**L**) and branched product (**B**). Ratios of **L**/**B** were determined by the peak areas in GC-MS analysis. [c] 24 h. [d] Propylene (0.5 MPa), CO (0.5 MPa), 24 h. [e] Ru-clusters/ceria (0.3 g), 36 h. ^1H NMR and ^{13}C NMR of the linear products were given in the Supporting Information.

To further explore the scope of amines, substrate (**1**) with electron-withdrawing (-Cl (**1a**), -F (**1b**) and -NO_2 (**1c**)) or electron-donating (-OCH_3 (**1d**) and -CH_3 (**1e**)) groups were also investigated in the presence of ethylene and CO (Table 4, entries 1-8). And the results showed that **1d or 1e** led to a higher yield of quinazolinones (88% and 87%) than **1a or 1b** (70% and 78%) (Table 4, entries 1-5). However, **1c**, the electron-withdrawing group (-NO_2) substituted on **1**, gave no product. It shows that *o*-aminobenzamide (**1**) with electron-donating groups are more favored to react than that with electron-withdrawing.

Further reaction using **1e** as the substrate were conducted and three quinazolinones could be obtained from propylene, cyclohexene and styrene in up to 99% isolated yields except for cyclohexene (52%) (Table 4, entries 6-8).

Table 4. Synthesis of quinazolinones from **1a-e**, olefins, and CO.[a]



Entry	1a /1b	Olefin	Product	Yield[b] (%) (L/B)
1[c]	 **1a**	 **2**	 **3a'**	**70**
2[c]	 **1b**	**2**	 **3b'**	**78**
3[c]	 **1c**	**2**	 **3c'**	**0**
4[c]	 **1d**	**2**	 **3d'**	**88**
5[c]	 **1e**	**2**	 **3e'**	**87**
6[c]	**1e**	 **2i**	 **3ei (L)**	**98 (58/42)[d]**
7	**1e**	 **2h**	 **3eh**	**52**
8	**1e**	 **2a**	 **3ea (L)**	**99 (83/17)[d]**

[a] Reaction conditions: Ru-clusters/ceria (0.2 g), **1a** or **1b** (0.5 mmol), olefin (2.0 mmol), CO (1.0 MPa), THF (2.0 mL), 24 h. [b] Isolated yields. Ratios of **L/B** were calculated based on the isolated yields of linear and branched products. [c] Olefins (0.5 MPa), CO (0.5 MPa). [d] ^{1}H NMR and ^{13}C NMR of the linear products were given in the Supporting Information.

In summary, we have developed an effective method to synthesize quinazolinones from olefins, CO and amines over a heterogeneous Ru-clusters/ceria catalyst in the absence of acids, bases, oxidants and with H_2O as the only by-product. Amide is verified as the reaction intermediate by ^{13}C NMR. With this method, a series of quinazolinones with either aromatic or non-aromatic substituents can be obtained in up to 99% isolated yields. Additionally, *E*-factor analysis shows that the values of s*E*-factor, c*E*-factor and *E*-factor for the synthesis of 2-ethyl quinazolinone are much lower than that of processes reported previously, highlighting the economy and sustainability of this reaction system. This method provides a new process with olefins as the readily available feedstocks in the synthesis of quinazolinone derivatives and related compounds, leading this method more attractive.

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Keywords: quinazolinones • Ru-clusters • ceria • olefins

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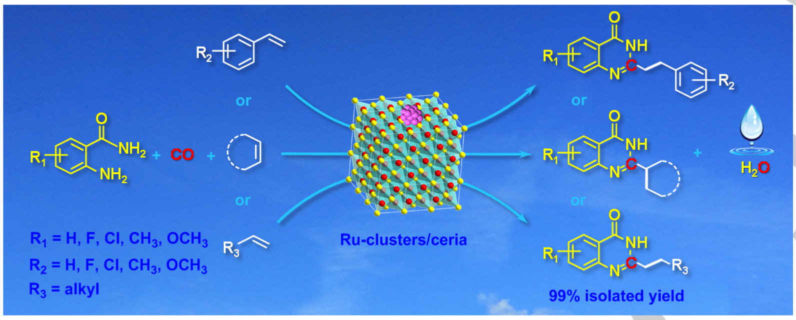
COMMUNICATION

The Synthesis of Quinazolinones from Olefins, CO and Amines over Heterogeneous Ru-clusters/Ceria Catalyst



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