Journal of Molecular Catalysis A: Chemical 371 (2013) 135–140

Contents lists available at SciVerse ScienceDirect

Journal of Molecular Catalysis A: Chemical

journal homepage: www.elsevier.com/locate/molcata

# Microwave-accelerated three components cyclocondensation in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones promoted by Cu-CNTs

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ARTICLE INFO

*Article history:*
Received 21 December 2012
Received in revised form 27 January 2013
Accepted 28 January 2013
Available online xxx

*Keywords:*
Dihydroquinazolin-4(1*H*)-one
Synthesis
Catalyst
Cu-CNT composites
Microwave

ABSTRACT

Cu-CNTs efficiently catalyzes condensation reaction of isatoic anhydride, aldehydes, and primary amines or ammonium acetate to afford the corresponding 2,3-dihydroquinazolin-4(1*H*)-one derivatives successfully in high yield. The protocol proves to be efficient and environmentally benign in terms of very easy work-up, high yields, and ease of recovery of catalyst. In addition, the present method is superior in terms of green media, the amount of catalyst and reaction time.

## 1. Introduction

Heterocyclic chemistry occupies an important place in organic chemistry research worldwide [1–4] and forms the basis of many pharmaceutical, agrochemical and veterinary products. In the broad class of heterocyclic compounds, the nitrogen heterocycles play an important role. One of such systems is quinazolinones as a class of N-heterocycles which have an important place in medicinal and biological chemistry [5]. One of the main group of quinazolinones, are 2,3-dihydroquinazolin-4(1*H*)-ones, which have in particular have various biological activities, and are also key intermediates for the synthesis of quinazolin-4(3*H*)-ones as another member of this biologically important family [6–8]. These exhibit broad spectrums of pharmacological and biological activities, such as antibacterial, antifertility, antifungal, antitumor, and mono amine oxidase inhibitory activity [9]. In view of these useful properties, various procedures have been reported for preparing 2,3-dihydroquinazolin-4(1*H*)-ones in literature, which including: (a) the condensation reaction of anthranilamide with aldehyde or ketone [6], (b) the reductive cyclization of o-nitrobenzamide or o-azidobenzamide with aldehydes or ketones [10], (c) desulfurization of 2-thioxo-4(3*H*)-quinazolinones [11], (d) a two-step synthesis starting from isatoic anhydride and amines, followed by annulation with ketones [12], (e) reaction of isatoic anhydride with Schiff-bases [13], condensation of anthranilamide with benzil [14] and the condensation of isatoic anhydride, aldehydes, and ammonium acetate or primary amine [15,16]. More attractive and convenient method for synthesis of such significant heterocycles is three-component condensation of isatoic anhydride, aldehydes, and amines. In this context, various catalysts have been reported [17–28]. Some of these methodologies involve strongly acidic conditions, toxic catalysts, hazardous organic solvents and long reaction times. Thus, there is a need for an acidic and novel catalyst that could overcome the above drawbacks.

CNTs have become one of the most active fields of nanoscience and nanotechnology due to their exceptional properties that make them suitable for many potential applications as high specific surface areas, chemical stability and electrical conductivities [29]. The use of carbon nanotubes as support for transition metal catalysts has recently been demonstrated [30–33]. Recently, CNT-based composites have attracted much attention due to their unique properties and promising applications [29,34,35]. In addition, the advantages Cu-CNT composites are good thermal conductivity, lower density than copper, good machinability and low coefficient of thermal expansion [36,37]. Therefore combining Cu with CNTs to form metal/CNT nanocomposites produces synergistic properties from both the metals and the CNTs. Some researchers reported that the catalysts metal supported on multi-walled carbon nanotubes exhibited much better activity than other carbon-based supports for some hydrogenation reactions [38]. It is well known that for CNTs without surface modification, there are insufficient binding sites for anchoring the precursors of metal ions or metal nanoparticles, which usually leads to poor dispersion and large metal nanoparticles, especially under high loading

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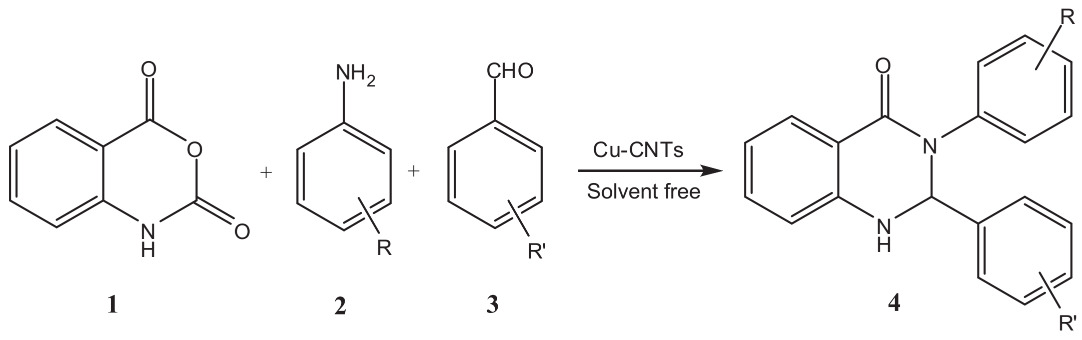
http://dx.doi.org/10.1016/j.molcata.2013.01.031



**Scheme 1.** Cu-CNTs catalyzed microwave-activated synthesis of 2,3-disubstituted-2,3-dihydroquinazolin-4(1H)-ones from isatoic anhydride, aromatic amine and aldehyde.

conditions [39,40]. Herein we investigated the possibility of synthesizing 2,3-dihydroquinazolin-4(1*H*)-one derivatives via one pot three-component condensation of isatoic anhydride, aldehydes and amines, in the presence of a catalytic amount of Cu-CNTs composites under microwave irradiation (Schemes 1 and 2).

## 2. Experimental

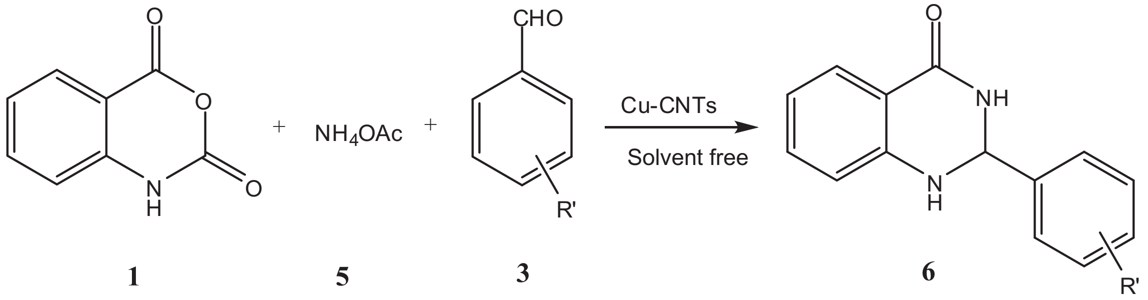
All chemicals and reagents were obtained from Merck and Aldrich and were used without further purification. MWNTs were purchased from Nanotech Port Co. (Taiwan). These MWNTs were produced via the chemical vapor deposition (CVD, or sometimes called catalytic pyrolysis) method. The outer diameter of CNT was between 20 and 40 nm. Melting points were determined on an Electrothermal MK3 apparatus and were uncorrected. $^1$H NMR and $^{13}$C NMR spectrums were recorded on Bruker DRX–400 spectrometer at 400 and 100 MHz in DMSO-$d_6$, respectively. The chemical shifts are expressed in $\delta$ ppm using TMS as an internal standard and coupling constants ($J$) are reported in hertz (Hz). Spin multiplicities are given as s (singlet), d (doublet), t (triplet) and m (multiplet) as well as b (broad). FT-IR spectra were recorded on a Perkin Elmer FT–IR 550 spectrophotometer using KBr pellets and absorbencies are reported in cm$^{-1}$. Microwave oven reactions were performed on a CEM–Discover Focused Monomode, microwave input power 300 W and microwave frequency 2450 MHz. ESI-MS analyses were carried out on a MICROMASS QUATTRO II triple quadrupole mass spectrometer. X-ray diffraction analysis was carried out using a Holland Philips Xpert X-ray powder diffraction (XRD) diffractometer (CuK, radiation, $\lambda = 0.154056$ nm), at a scanning speed of 2°/min from 10° to 100° ($2\theta$). Scanning electron microscopy (SEM) images were obtained using a FEI Quanta 200 SEM, operated at a 20 kV accelerating voltage. Purity of the compounds synthesized was monitored by TLC), visualizing with ultraviolet light. Elemental analysis was performed using a Heraeus CHN-O-RAPID elemental analyzer. UV–vis spectra of compounds were recorded on a UV-Vis Varian Perkin-Elmer UV 550-S spectrophotometer. The known products were characterized by comparison of their spectral ($^1$H NMR, $^{13}$C NMR and MS) and physical data with those of authentic samples. All yields refer to isolated products after purification.

### 2.1. Catalyst preparation

Detailed procedure for preparing the Cu-CNT nanocomposites is described elsewhere [41]. In brief, The CNTs were treated by adding 40 mL of nitric acid and sulfuric acid (3:1 in volume) solution, then dropping 10 mL of ethanol into the solution slowly, to increase their surface chemical activity via oxidation of the defect sites before coating with copper. The solution was stirred at 70 °C for 24 h. Then in a chemical reduction process, equal amounts of 0.01 M of $Cu(NO_3)_2{\cdot}3H_2O$ aqueous solution and ethanol added to the basic solution of NaOH and $Na_2CO_3$ (3:1 in volume) with pH 12. Acid-treated MWNTs were added to above aqueous solution followed by dropping of a solution of 37% formaldehyde (10:1 of formaldehyde and copper nitrate) under sonication for 2 h. Then, the precipitate was separated by centrifugation and washed with deionized water until pH 7. The solid samples were then calcined at 350 °C for 2 h (Fig. 1).

### 2.2. General procedure for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones under microwave irradiation

Cu-CNTs nanocomposite (0.04 g) as an efficient catalyst was added to the mixture of isatoic anhydride (1 mmol), primary aromatic amine (1.1 mmol) or ammonium acetate (1.2 mmol) and aldehyde (1 mmol). The resulting mixture was heated at microwave oven under solvent free conditions with stirring for appropriate time until reaction complete. After the complete disappearance of the starting material, as monitored by TLC (eluent: petroleum ether:ethyl acetate: 4:1), hot ethanol (15 mL) was added and the catalyst was removed by filtration. The filtrate containing the catalyst was evaporated under reduced pressure to give a solid, and the recovered catalyst could be reused without a loss of catalytic



**Scheme 2.** Cu-CNTs catalyzed microwave-activated synthesis of 2-substituted-2,3-dihydroquinazolin-4(1H)-ones from isatoic anhydride, ammonium acetate and aldehyde.

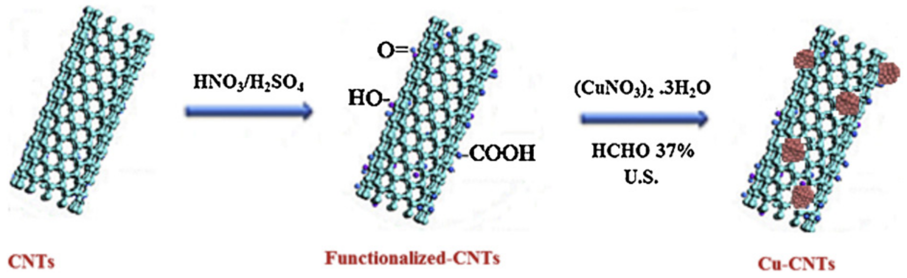


**Fig. 1.** Schematic presentation of functional group formation and copper decoration on multi walled carbon nanotubes.

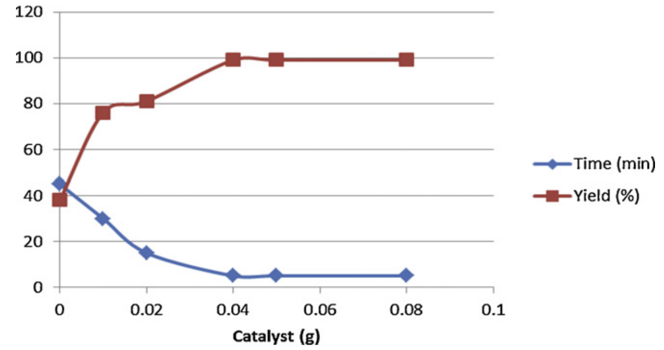


**Fig. 2.** Investigation of the amounts of catalyst effect on the preparation of **4a**.

**Table 2**
Optimization of the condensation reaction power for synthesis of compound **4a** in various times.

| Entry | P (w) | Time (min) | Yield (%) |
|---|---|---|---|
| **1** | 30 | 35 | 56 |
| **2** | 45 | 20 | 72 |
| **3** | 50 | 15 | 87 |
| **4** | 65 | 5 | 99 |
| **5** | 70 | 5 | 95 |
| **6** | 85 | 3 | 92 |

activity. The corresponding pure product was obtained by recrystallization from ethanol.

## 3. Results and discussion

In order to exploit potential of Cu-CNTs composite for the synthesis of 2,3-dihydroquinazolin 4(1*H*)-one derivatives, for the first time three components one-pot condensation of isatoic anhydride, aldehydes, and $NH_4OAc$ or primary amines in the presence of a catalytic amount of Cu-CNTs composite as an efficient catalyst under microwave-activated conditions was investigated. We studied the potential use of Cu-CNTs as the catalytic agent on the condensation. To improve the yield and optimize the reaction conditions, the effect of amount of catalyst on the condensation using the model reaction of isatoic anhydride, benzaldehyde and aniline was studied by varying the amount of Cu-CNTs without solvent under microwave irradiation (Fig. 2). The results indicated clearly that Cu-CNTs was a superior catalyst and successfully promoted this three components condensation with high yields. As it was shown from Fig. 2, the best result has been obtained with an amount of 0.04 g Cu-CNTs composite. It is worth mentioning that all these results clearly showed the efficiency of this catalytic system in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones. Different molar ratios of reagents were examined and the best result was obtained with 1:1:1.1:1.2 ratios of isatoic anhydride, aldehyde, and aniline or ammonium acetate in the presence of 0.04 g of Cu-CNTs composite. Presently organic reactions in solvent free media have attracted the attention of researchers. Further because of the toxicity of organic solvents, we considered the green media. When the reactions were conducted in solvent free conditions, the expected products were obtained with excellent yields in low reaction times. In order to generalize the procedure, a variety of substituted aromatic aldehydes were screened under optimized conditions. To expand the scope of amine substrates, a variety of amine derivatives were applied to this protocol. Interestingly, in all cases, both electron-rich and electron-deficient substrates worked well, and the desired reactions took place successfully to afford the desired products in good yields. These values are tabulated in Table 1. All these observations indicate that the electronic properties and the position of the substituent group on the phenyl ring had no obvious influence on this conversion.

The reaction temperature also played an important role in this reaction. Therefore, we tried to optimize the reaction temperature in various times for the model reaction. As could be seen on Table 2, the reaction at power 65 and 5 min proceeded in highest yield. Using these optimized reaction conditions, the above mentioned cyclocondensation reaction proceeded well and afforded the desired products in good to excellent yields. The catalytic activity of Cu-CNTs composite was evaluated for this model reaction, and the results showed that Cu-CNTs composite could be used for

**Table 1**
Cu-CNTs composite mediated synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives under microwave irradiation without any solvent.

| Entry | Product | Amine(R)/Ammonium salt | R′ | Time (min) | Yield (%) | M.p (°C) | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | | | | | | Found | Reported | |
| **1** | **4a** | H | H | 5 | 99 | 214–215 | 214–216 | [42] |
| **2** | **4b** | H | 4-Me | 12 | 90 | 223–225 | 222–224 | [43] |
| **3** | **4c** | 4-Me | 4-OMe | 17 | 93 | 241–243 | 244–245 | [44] |
| **4** | **4d** | 4-OH | 4-Me | 20 | 87 | 248–250 | 249–252 | [25] |
| **5** | **4e** | H | 4-Br | 10 | 95 | 220–222 | 219–223 | [27] |
| **6** | **4f** | 4-Me | 4-Cl | 23 | 89 | 269–272 | 270–274 | [45] |
| **7** | **4g** | H | 4-F | 15 | 92 | 235–237 | 235–238 | [27] |
| **8** | **5h** | $NH_4OAc$ | 2,3-$Cl_2$ | 15 | 89 | 223–225 | 225–227 | [46] |
| **9** | **5i** | $NH_4OAc$ | 2-F | 12 | 91 | 265–267 | 266–267 | [47] |
| **10** | **5j** | $NH_4OAc$ | 4-$NO_2$ | 10 | 95 | 198–200 | 199–201 | [48] |

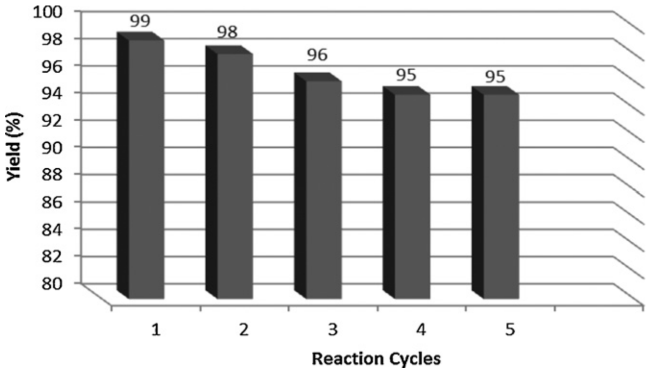


**Fig. 3.** Recyclability of the catalyst for the one-pot synthesis of **4a**.

five successive runs without significant decrease in its catalytic activity (Fig. 3). In general, the target compounds were obtained in excellent yields under the optimized conditions and the desired products were isolated by a clean and simple work-up procedure. All of the products are known compounds and their structure were confirmed by comparison of their physical and spectral data with those of authentic samples.

Characterization of Cu/CNTs has been performed by X-ray diffraction. As shown in Fig. 4, the peaks of CNT were 26.48 and 44.68, and broad diffraction peaks with 2 at 43.78, 48.42 and 74.95, corresponding to Cu. Fig. 5 shows the TEM and SEM images of CNTs and Cu coated on CNT. The CNTs are very pure and their surface is smooth. In comparing b with c, when CNTs coated with Cu, the surface of CNTs was rough. Also SEM images show that the CNTs are dispersed uniformly and combined well in the Cu.

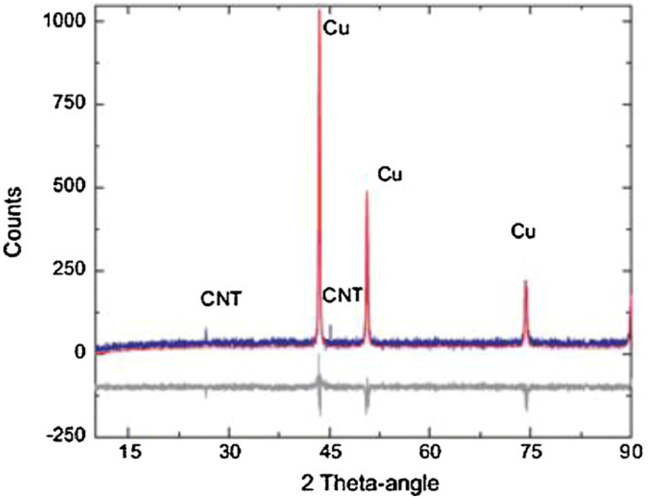


**Fig. 4.** XRD diffraction of Cu-CNT composites.

On the basis of our experimental results and by referring to the literature, a plausible mechanism of the three components reaction is proposed in Scheme 3. Catalyst also might assist in improving reactivity of carbonyl units and finally in promoting cyclization to form the quinazolinone core. First the isatoic anhydride is activated with coated metal by the coordination with carbonyl groups followed by nucleophilic attack of amine on the carbonyl unit to form intermediate **I**. Then decarboxylation resulting intermediate **I** into the formation of intermediate **II** followed by proton transfer yields 2-amino-*N*-substituted-amide **III**. Condensation of the promoted aldehyde with the amino group of 2-amino-*N*-substituted-amide **III** then produces an imine intermediate **IV**. The part of amide in intermediate **III** could be converted into its tautomer in the presence of metal. Meanwhile, the part of imine in intermediate **IV** could be activated by catalyst. Thus, intermediate **V** could be prepared by intermolecular nucleophilic attack of the amide nitrogen on activated imine carbon, followed by a 1,5-proton transfer to produce the final products.

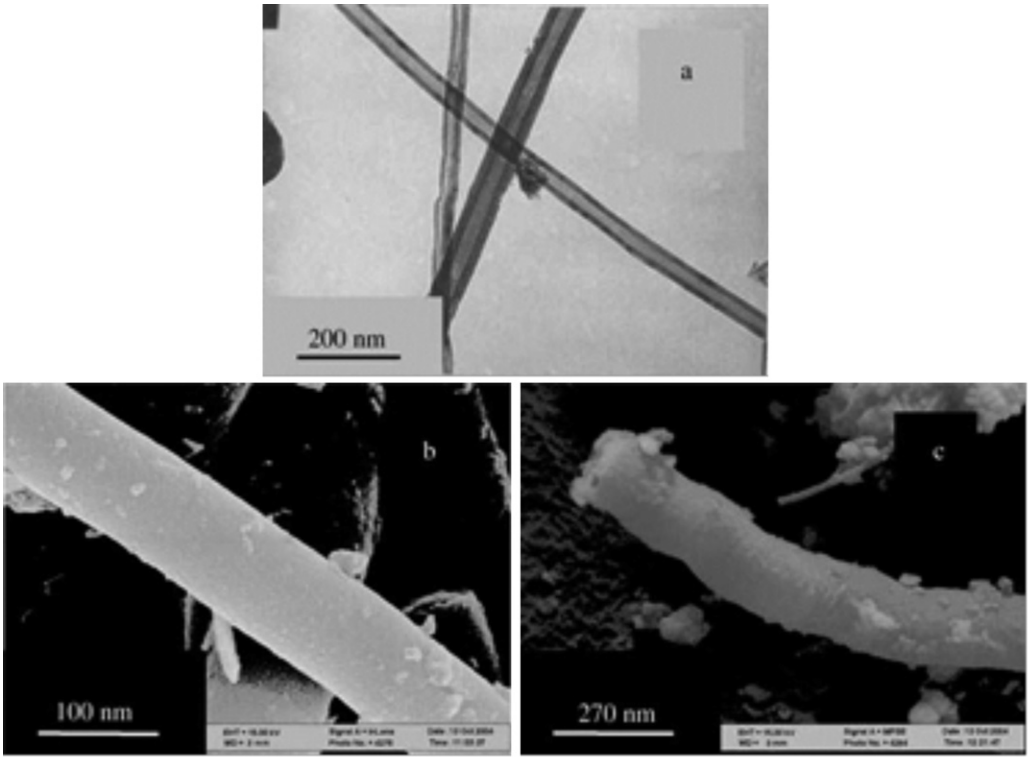
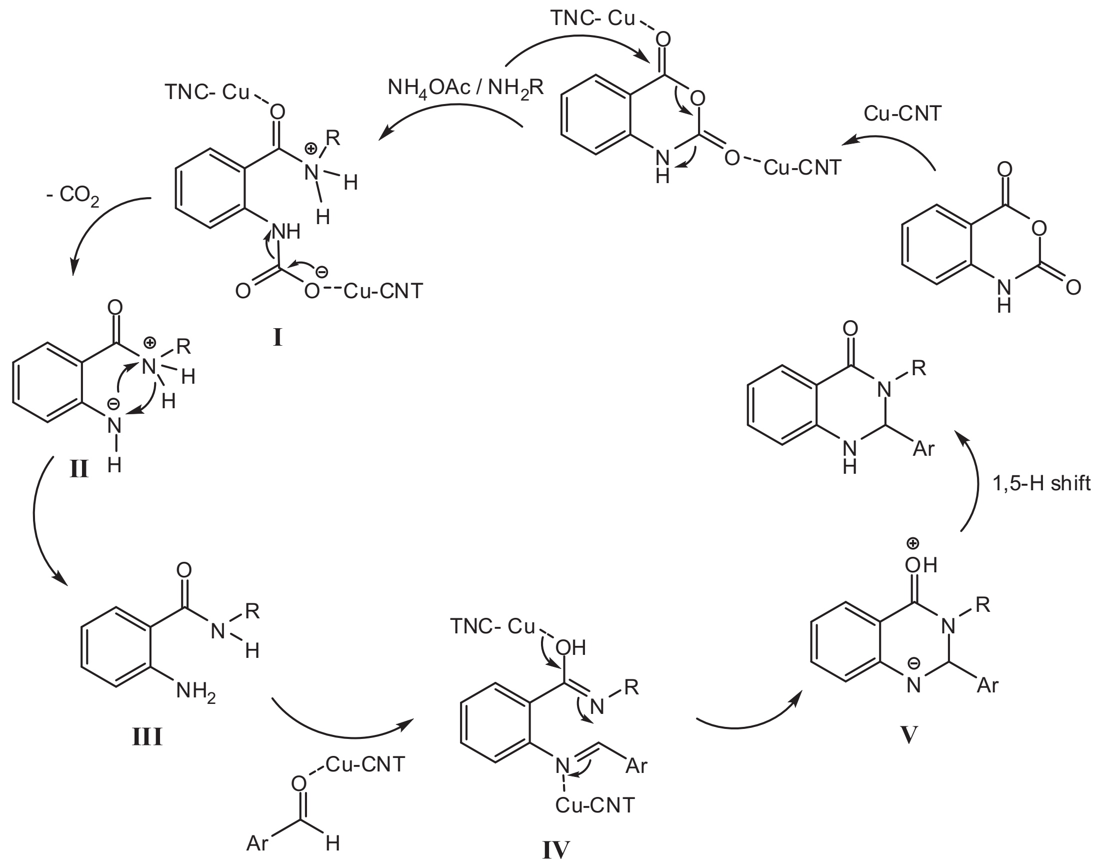


**Fig. 5.** Images of the samples (a) TEM of unpurified CNTs; (b) SEM of purified CNTs; (c) SEM of copper-coated CNTs.



**Scheme 3.** Plausible reaction pathway in the synthesis of mono- and disubstituted quinazolinones.

## 4. Conclusions

In summary, we have successfully demonstrated for the first time that Cu-CNTs nanocomposite could be used as an excellent and efficient catalyst for convenient synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives under solvent free conditions and microwave irradiation. The novelty and synthetic valuability of this methodology developed the mild reaction conditions, avoiding the use of organic solvents, easy experimental procedure, ease of product isolation, and recovery of the catalyst. These conditions may be ideally suited for an effective synthesis on a larger scale.

## Acknowledgement

The authors are grateful from university of Kashan for the financial support of this work, grant no. 159198/15.

## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.molcata.2013.01.031.

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