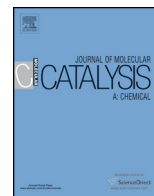




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Efficient synthesis of 2-aryl-2,3-dihydroquinazolin-4(1H)-ones in the presence of nanocomposites under microwave irradiation

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ABSTRACT

The preparation of quinazolinones has been developed by the condensation of aromatic aldehydes and 2-aminobenzamide in the presence of transition metal-CNTs nanocomposites as novel and recyclable catalyst under microwave and solvent-free conditions. This methodology provides a high speed path for the green and mild synthesis of 2-aryl-2,3-dihydroquinazolin-4(1H)-ones with high structural diversity. The present methodology has advantages such as high activity and reusability of catalysts, heterogeneous nature of catalysts, no use of hazardous organic solvents and green conditions.

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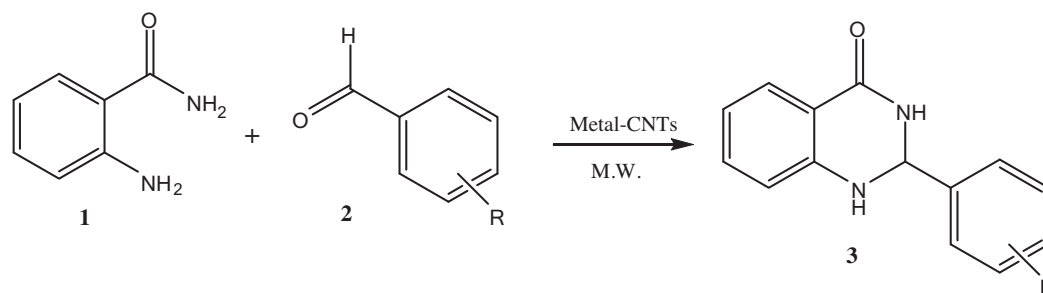
1. Introduction

Natural and synthetic quinazolinone derivatives constitute an important class of fused heterocycles, which influence numerous cellular processes. These scaffolds as a core unit have been extensively studied in a number of biologically active compounds because of their broad range of biological, medicinal, physiological and pharmacological properties and their potential clinical applications [1–14]. Thus, extensive efforts have been exerted to develop methodology for the synthesis of quinazolinone derivatives. A variety of synthetic methods have been extended for the synthesis of quinazolinones and aryl-substituted quinazolinones included amidation of 2-aminobenzonitrile followed by oxidative ring closure [15], catalytic heterocyclization of nitoarenes [16], reduction of the azide functionality [17] and cyclization of oacylaminobenzamides [18]. The other methods are such as solid-phase synthesis of 2-arylamino-substituted quinazolinones [19] and preparation from isatoic anhydrides and Schiff bases [20]. Also, a literature survey showed that many methods were reported for the synthesis of 2,3-dihydro-2-aryl-4(1H)-quinazolinones [21–23]. These methods involve condensation of *o*-aminobenzamide with benzyl [24] and also reductive cyclization of *o*-nitrobenzamide or

o-azidobenzamide with aldehydes and ketones [25]. Among the diverse synthetic methods for the preparation of 2,3-dihydro-2-aryl-4(1H)-quinazolinones, the most popular method includes condensation of derivatives of *o*-aminobenzamide with structurally diverse aldehydes or ketones [21]. The antitumor activities of a number of 2,3-dihydro-2-aryl-4-quinazolinone derivatives were designated for the first time over three decades ago [26,27]. 2-phenyl-4-quinazolinones exhibit a wide range of biological activities such as cytotoxicity as potent tubulin polymerization inhibitors [28]. Therefore, 2,3-dihydro-2-phenylquinazolin-4(1H)-ones are inhibitors of tubulin polymerization with impressive antiproliferative activity against several human cancers with IC50 value in nano molar concentration. They act like colchicine (inhibitors of tubulin polymerization) by binding to α,β -tubulin [28]. Recently, also these analogs have been evaluated as antimitotic agents [29]. Due to the biological and pharmaceutical importance of quinazolinone derivatives, it has a great importance to develop greener process for the synthesis of 2,3-dihydro-2-aryl-4(1H)-quinazolinones.

Recently, much attention has been attracted to the usage of carbon nanotubes (CNTs) in composite materials. Carbon nanotubes with unique morphology, good chemical stability and high electrical conductivity are ideal materials to support heterogeneous catalysis [30]. Many studies have been developed to decorate metal nanoparticles on the CNTs, which lead to optimal properties and superior performance [30,31]. The nanosized metallic

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Scheme 1. Direct condensation of anthranilamide with aldehydes catalyzed by metal-CNTs under microwave irradiation.

particles/CNTs have unique properties with respect to the specific particle morphology (size and shape), concentration, metal dispersion and the electronic properties of the metal on the CNTs surface [32]. Additionally, metal NPs/CNTs nanocomposites have presented attractive applications in many fields including sensors, catalysis, fuel cells, electroanalysis and surface-enhanced Raman scattering [33–37]. Several approaches have been developed to prepare CNT-metal NPs assemblies involving electrochemical reduction [38,39], the direct decoration of metal nanoparticles [40], spontaneous reduction [41] and substrate-enhanced electrochemical reduction [42]. However, the investigations have shown that the surface of the CNTs is hydrophobic and highly inert because of high graphitization [43,44]. In order to active the surfaces of the tubes, surface modification of CNTs has been done by using different acid treatments to create carboxylic, carbonyl and hydroxyl groups [45]. The presence of acidic groups makes CNTs surface more hydrophilic [46] and enhances the affinity between the metal and carbon, approving the incorporation of metallic nanoparticles and avoiding agglomeration [47]. Therefore, conjunction of metal NPs with functionalized CNTs increases the catalytic properties of metal, which helps electron–hole pair separation and free migration of electrons from the metal to the electron-accepting CNT surface [48].

We employed microwave energy because of the extensive application of microwave technology in organic synthesis particularly in solid state. Also, it accelerates unique chemical processes with special attributes including reduced polluting, greater selectivity, enhanced reaction rates and ease of manipulation [49]. Current study has been employed as an alternative new methodology for the synthesis of 2,3-dihydro-2-aryl-4(1H)-quinazolinones under microwave irradiation. Herein, we would like to introduce some metals supported on multi-walled carbon nanotubes as heterogeneous catalysts for the synthesis of quinazolinones. Therefore, we have introduced the reaction of substituted aldehydes with anthranilamide (2-aminobenzamide) as the source of nitrogen in the presence of these catalytic systems (Scheme 1). The catalysts are efficient, recyclable and easy to be separated from the reaction mixture via simple filtration. The green and neat approach, easiness and variability in derivatives in the present procedure makes it an attractive method.

2. Experimental

All materials and reagents were purchased from Merck Company. Melting points were recorded by Electrothermal MK3 apparatus using open capillaries and they are uncorrected. MWCNTs were purchased from Nanotech Port Co. (Taiwan). These MWCNTs were produced via the chemical vapor deposition (CVD, or sometimes called catalytic pyrolysis) method. The outer diameter of CNTs was between 10 and 20 nm. The progress of the reactions and the purity of the products were monitored by thin-layer chromatography (TLC). FT-IR spectra were obtained by a Perkin Elmer FT-IR 550 spectrophotometer (KBr pellets). NMR

spectra were obtained in DMSO- d_6 solution by a Bruker DRX 400 MHz spectrometer and chemical shifts are expressed in δ ppm downfield from TMS as an internal standard. X-ray diffraction (XRD) pattern of the as-synthesized material was carried out on a Holland Philips Xpert X-ray diffraction (XRD) diffractometer (CuK α radiation, $\lambda = 0.154056$ nm and 40 kV voltage), at a scanning speed of 2° min^{-1} from 10° to 100° (2θ). The nanocomposites were characterized using a KYKY scanning electron microscope (SEM) model EM3200 operated at a 25 kV accelerating voltage. The TEM images were recorded on a Zeiss EM10C Transmission electron microscope (TEM) operated at 80 kV accelerating voltage. The Raman spectra were measured by a Bruker SENTERRA spectrometer with spectral range: $200\text{--}3500 \text{ cm}^{-1}$ and Laser wavenumber 785 nm.

2.1. General procedure for the preparation of quinazolinones under microwave irradiations

Co-CNTs (0.025 g) were added to the mixture of benzaldehyde (1 mmol) and 2-aminobenzamide (1 mmol) in a 25 mL pyrex flask and microwave energy were irradiated under solvent free condition. The reactions were followed by thin layer chromatography (TLC) using petroleum ether/ethyl acetate (7:3) as a mobile phase. After completion, hot ethanol was added to the resulting mixture and the heterogeneous catalyst was separated by filtration. Then, the filtrate precipitated onto crushed ice while was being stirred for several minutes. Insoluble products were filtered and recrystallized from ethanol to afford the pure product.

3. Results and discussion

Interest in the environmental control of chemical processes to provide an eco-friendly system has increased significantly in recent years. The most important principles of the green chemistry are the elimination of toxic and hazardous solvents in chemical synthesis, avoidance from the generation of waste and use of environmentally friendly and recyclable catalyst. A solvent-free or solid state reaction reduces pollution and cost and the formed products are pure enough without the need to chromatography. Therefore, it was interesting to investigate the catalytic effect of a number of transition metals supported on CNTs in the synthesis of the quinazolinone compounds under microwave irradiation in solvent free conditions as a green media. To exploit environmental friendly, facile and suitable conditions for the generation of 2-substituted 2,3-dihydroquinazolin-4(1H)-ones, the solvent free treatment of substituted aldehydes and anthranilamide under microwave irradiation was selected as a model reaction. From a practical perspective, these catalysts can be easily prepared from available reagents according to literature procedures [50–53] and they can also be recycled. In addition, the functionalization process can be carried out by pretreatment of CNTs in mixture of HNO_3 and H_2SO_4 for surface modification of CNTs. The acidic sites using improvement of the surface hydrophilicity can act as nucleation

Table 1
Condensation of anthranilamide and benzaldehyde in presence of different catalysts.

Entry	Catalyst	Time (min)	Yield (%)
1	Ag-CNTs	25	75
2	Co-CNTs	10	98
3	Cu-CNTs	20	80
4	Pt-CNTs	15	89

Table 2
Effects of different amounts of Co-CNTs nanocomposites on the model reaction.

Entry	Catalyst (g)	Time (min)	Yield (%)
1	0	49	10
2	0.01	35	61
3	0.015	20	76
4	0.02	15	90
5	0.025	10	98
6	0.03	10	94

center for the deposition of metal NPs on the surface of CNTs. Our initial study was started by employing a number of nanocomposites such as Ag-CNTs, Co-CNTs, Cu-CNTs and Pt-CNTs to evaluate the capability and efficiency of their catalytic activity and to promote the model condensation reaction. As Table 1 shows, initial screening studies identified that all the above-mentioned catalysts show a good catalytic activity for the model reaction but Co-CNTs exhibited the highest catalytic activity as compared with other catalysts in terms of time, yield and reaction conditions. Catalytic activity of these nanocomposites decreases in the following order: Co-CNTs > Pt-CNTs > Cu-CNTs > Ag-CNTs. The advantage of the Co-CNTs for this reaction is the more number of unfilled d-orbitals of the metal particles, its better Lewis acid character, greater selectivity and more stability. With notice to these results, Co-CNTs were chosen as the best catalyst for further work.

During the present synthesis, the effect of amount of catalyst in yield and reaction time was evaluated. Table 2 shows that the yields of 2-aryl-2,3-dihydroquinazolin-4(1H)-one were improved initially with increase in the amount of catalyst at short reaction times. The optimum amount of catalyst was found to be 0.025 g with respect to product yield, reaction time and concentration of catalyst. The use of excessive amounts of the catalyst can increase

Table 3
Effect of irradiation power on the yield of dihydroquinazolinones.

Entry	Power (W)	Time (min)	Yield (%)
1	100	59	50
2	200	37	65
3	300	25	70
4	400	20	87
5	500	10	98
6	600	10	98

Table 4
Reusability of Co-CNTs in the synthesis of 4a.

Run	1	2	3	4	5
Yield (%)	98	97	97	95	95

adsorption of the corresponding product in the excess active sites and restricted dispersion of the adsorbed product within the active sites of the catalytic system.

The rapid microwave-assisted organic synthesis (MAOS) of quinazolinones under solvent-free conditions results shorten reaction times, improved reaction yields and easier work up in agreement with green chemistry protocols. It is noticed that the synergetic effects of MW/nanocomposites couple have promoted this solvent free condensation. Also, the effect of irradiation power on the reaction progress was studied. As shown in Table 3, it was observed that the yield and reaction time were significantly influenced by irradiation power. Increasing the power to 500 W led to the reduction of reaction time and improvement of the yield of products. A further increasing in power did not increase the product yield.

The use of heterogeneous and reusable catalysts represents one of the greenest procedures in chemical technology. Moreover, heterogeneous catalysts should not only be stable and active, but also require easy separating and reusing for useful applications. For this purpose, hot ethanol was added to the mixture reaction and the solid catalyst separated by filtration, was washed in ethyl acetate and dried at 100 °C for 1 h, and finally was reused for the same reaction process. It is noteworthy to highlight that catalyst could be recovered efficiently and could be recycled while no decrease in

Table 5
Cobalt supported on CNTs promoted synthesis of substituted dihydroquinazolinones under microwave irradiation.

Entry	R	Product	Time (min)	Yield (%)	M.p (°C)	
					Found	Reported
1	H	3a	10	98	216–218	217–219 [54]
2	4-OH	3b	35	77	279–280	278–280 [55]
3	3-NO ₂	3c	10	95	196–198	195–196 [54]
4	2,4-(OMe) ₂	3d	25	81	184–186	186–187 [55]
5	4-OMe	3e	20	82	181–183	182–184 [56]
6	4-Cl	3f	15	89	205–206	206–207 [54]
7	4-Me	3g	30	86	225–227	224–226 [54]
8	4-CN	3h	10	93	250–252	249–251 [57]
9	3-F	3i	10	96	264–266	266–267 [55]
10	2-Naphthyl	3j	20	84	225–226	225–227 [58]
11	2-Br	3k	15	88	172–173	173–175 [59]
12	2-Cl	3l	10	90	204–205	203–205 [56]
13	2-OH	3m	35	75	222–224	221–223 [60]
14	2,4-Cl ₂	3n	15	85	167–169	165–167 [56]
15	4-F	3o	10	89	203–204	203–205 [61]
16	4-(NMe ₂)	3p	30	78	208–210	210–212 [54]
17	4-Br	3q	10	86	195–197	197–199 [62]
18	2-NO ₂	3r	10	91	193–194	190–192 [60]
19	2,3-Cl ₂	3s	20	84	223–225	225–227 [63]
20	3-Cl	3t	15	87	185–187	186–188 [64]
21	2-OMe	3u	20	80	173–174	173–175 [64]
22	2,3-(OMe) ₂	3v	30	78	239–241	238–240 [65]
23	2-F	3w	10	90	265–267	266–267 [66]

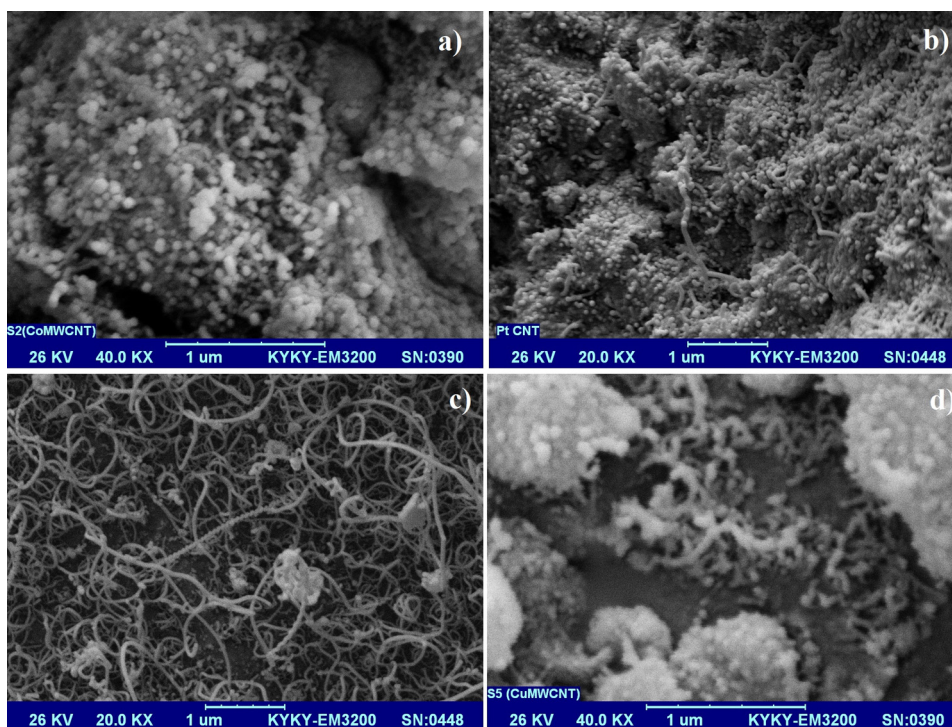


Fig. 1. The scanning electron microscope (SEM) micrographs of the (a) Co-CNTs, (b) Pt-CNTs, (c) Ag-CNTs and (d) Cu-CNTs.

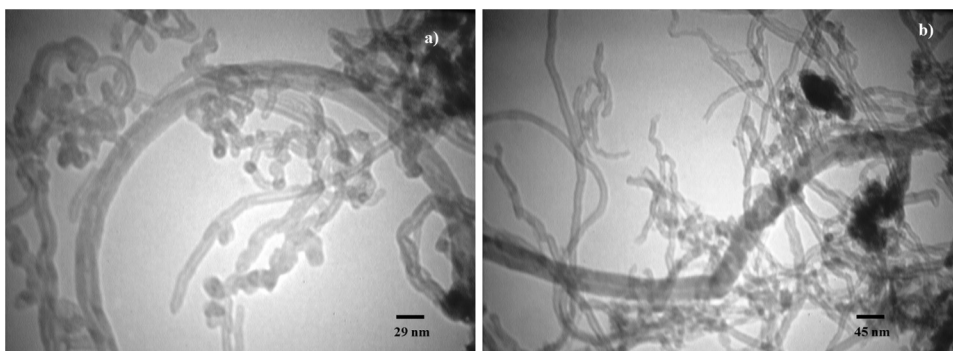


Fig. 2. Images of the TEM of Co nanoparticles supported on carbon nanotubes.

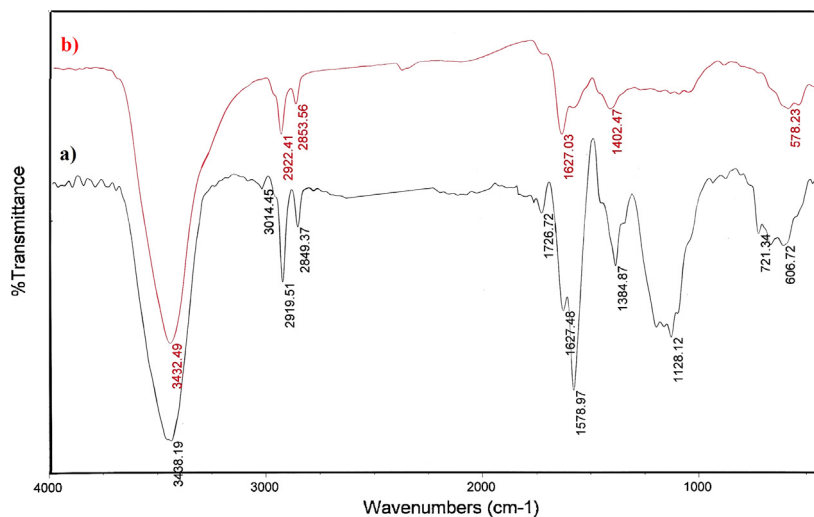


Fig. 3. FT-IR of (a) acid treatment CNTs and (b) Co-CNTs.

the yield of the desired product was observed during the first recycle. Further in the fifth run, it was found to be a slight decrease in the yield of 2-aryl-2,3-dihydroquinazolin-4(1*H*)-one in the standard reaction (Table 4).

To explore the synthetic scope of this model reaction, a wide range of structurally diverse aldehydes were investigated under this protocol to afford the corresponding dihydroquinazolinones in excellent yields. As shown in Table 5, no obvious effects of electrons and nature of substituents on the aromatic ring were observed in this reaction. In all cases, the reactions provided the corresponding products in excellent isolated yields and high purity. All the products were fully characterized by spectroscopic methods (^1H NMR, ^{13}C NMR, and IR spectra) and also by comparison with the reference compounds.

Fig. 1 demonstrates the SEM micrographs of the metal-CNTs nanocomposites which metal particles are well distributed on the surface of carbon nanotubes.

From Fig. 2, it can be seen that the most of carbon nanotube surfaces were decorated with cobalt nanoparticles and there was a catalyst particle on each tip of the CNTs. It indicates that our method has high efficiency. Also, the distribution of Cobalt nanoparticles on the CNTs surface is quite uniform and no detectable local aggregation is observed.

Fig. 3 is the FT-IR spectra of purified CNTs and Co-CNTs catalyst. It shows a broad peak centered at 3438 cm^{-1} , which corresponds to stretching mode of O–H group and peaks 2919 and 2849 cm^{-1} correlated to stretching vibration of C–H bond. The peak at 1627 cm^{-1} is due to the C=C stretching mode and the peak centered at 1726 cm^{-1} is due to C=O. Also, the vibration band at 578 cm^{-1} is assigned to the Co–O absorption band.

4. Conclusions

The present study describes the synthesis of quinazolinones through a novel straightforward procedure by the condensation of 2-aminobenzamide with various aromatic aldehydes in the presence of several metal-CNTs as novel green promoters under microwave irradiation. Therefore, we have investigated the catalytic performances of some CNT-supported transition metal catalysts, and it presented that the supported Cobalt catalyst is unique for this condensation. Co-CNTs have emerged as a highly efficient and effective potential Lewis acid catalyst that represents high selectivity in eco-friendly tandem synthesis of substituted 2,3-dihydroquinazolin-4(1*H*)-ones. The generality and simplicity of this methodology, easy separation of the nanocatalyst owing to its heterogeneous nature, omitting any organic solvent and lastly agreement with the green chemistry protocols are the unique features of the present methodology. All of these advantages make it an effective and attractive process for the synthesis of quinazolinone derivatives. This methodology provides an entry to the development of a facile and green strategy to prepare quinazolinones and it can be extrapolated for similar kinds of heterocycles to generate large libraries in shorter time.

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