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Original article

Rapid and mild synthesis of quinazolinones and chromeno[*d*]pyrimidinones using nanocrystalline copper(I) iodide under solvent-free conditions

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ABSTRACT

This paper describes a very simple, efficient synthesis of quinazolinones and chromeno[*d*]pyrimidinones from the reaction of aryl aldehydes, urea/thiourea and active methylene compounds (dimedone/4-hydroxycoumarin) using nano-sized CuI particles under solvent-free conditions. The highlights of this new method are based on using an effective and recyclable catalyst, affording high yields of products, mild reaction conditions, facile work-up and purification.

1. Introduction

The quinazolinone moiety is found, as alterative in a wide variety of biologically active compounds which can be used as hypnotic/sedative drugs for treatment of cancer [1]. Furthermore, quinazolinone derivatives are of interest because they exhibit a broad spectrum of biological properties, such as analgesic [2], anti-inflammatory [3], antimicrobial and anti-tubercular [4,5], anti-HIV [6], antimalarial and antihistamine [7]. Chromenes are also an important class of nitrogen containing heterocycles, which possess a range of diverse pharmacological properties, such as antioxidant [8,9], anticancer [10–13], antimicrobial [14–17], hypotensive [18], and local anesthetic [19]. In addition, they have been used as cognitive enhancers [20,21] in the treatment of neurodegenerative diseases, including Alzheimer's disease [22] and schizophrenia [23].

Recent reports reveal that the utility of nanostructured metal salts, as efficient heterogeneous catalysts, have emerged as a powerful synthetic tools for the synthesis of many organic compounds [24–30]. Copper(I) iodide nanoparticles (CuI NPs) as a Lewis acid catalyst, has attracted intense interest due to its unique and improved properties [31–38].

Thus, the synthesis of heterocycles with a quinazolinone or chromene framework is of particular importance to chemists and, hence, various methods have been developed for the synthesis of these compounds as described in the literature [39–43]. Although, most of these procedures offer several advantages, there are also related disadvantages, such as longer reaction times, unsatisfactory yields, harsh reaction conditions and use of high cost or toxic catalysts. To the best of our knowledge, however, no reports to date have revealed the synthesis of these compounds using a CuI NPs catalyst. With this in mind, the central focus of the present article is to investigate the role of nano-sized CuI particles as an inexpensive, readily prepared, recoverable and high yielding heterogeneous catalyst for the synthesis of quinazolinones (**4**) and chromeno[*d*]pyrimidinones (**6**) *via* a condensation reaction of aryl aldehydes **1**, urea/thiourea **2** and active methylene compounds including dimedone (**3**) or 4-hydroxycoumarin (**5**) under solvent-free conditions (Scheme 1).

2. Experimental

2.1. Materials and methods

All of the chemical materials used in this work were purchased from Merck or Sigma–Aldrich and used without further purification. Melting points were determined on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were obtained on an ABB FT-IR (FTLA 2000) spectrometer. The ^{1}H NMR spectra were

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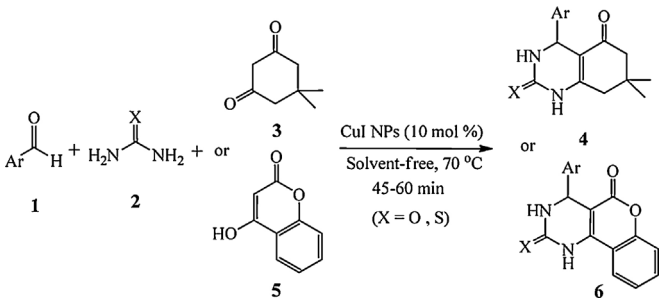

Scheme 1. Synthesis of quinazolinone and chromeno[*d*]pyrimidinone derivatives.

recorded on a Bruker DRX-400 AVANCE at 400 MHz, using TMS as internal standard and DMSO-d_6 as solvent. Elemental analyses were carried out using a Heraeus CHN rapid analyzer.

2.2. General procedure for preparation of compounds **4a–h** and **6a–f**

A mixture of aryl aldehyde (1 mmol), urea/thiourea (1.2 mmol), active methylene compound (dimedone/4-hydroxycoumarin) (1 mmol) and CuI NPs (1.9 mg, 10 mol%) was stirred at 70 °C for appropriate times. After completion of the reaction, indicated by TLC, the reaction mixture was diluted with DMF (5 mL) and centrifuged for five min at 2000–3000 rpm, to separate the catalyst by filtration which then was washed with EtOH, and dried under vacuum for several hours. The filtrate was then poured into ice-cold water (10 mL) to give a solid precipitate, which was filtered, washed with water and recrystallized from EtOH to generate the pure product.

7,7-Dimethyl-4-phenyl-4,6,7,8-tetrahydro-2,5(1*H*,3*H*)-quinazolinedione (**4a**): Yield 254 mg (94%). White powder. M.p. 285–287 °C. IR (KBr, cm^{-1}): ν_{max} 3333, 3254, 2960, 1706, 1678, 1611, 1474, 1364. ^{1}H NMR (400 MHz, DMSO-d_6): δ 0.98 (s, 3H, CH_3), 1.09 (s, 3H, CH_3), 2.21 (q, 2H, J = 16.0 Hz, CH_2), 2.38 (q, 2H, J = 16.7 Hz, CH_2), 5.29 (d, 1H, J = 2.8 Hz, CH), 7.27 (m, 5H, HAr), 7.46 (s, 1H, NH), 9.35 (s, 1H, NH) ppm. Anal. Calcd. for $C_{16}H_{18}N_2O_2$ (270.33): C 71.09, H 6.71, N 10.36; found: C 70.86, H 6.61, N 10.58.

7,7-Dimethyl-4-(2-chlorophenyl)-4,6,7,8-tetrahydro-2,5(1*H*,3*H*)-quinazolinedione (**4b**): Yield 283 mg (93%). White powder. M.p. 269–270 °C. IR (KBr, cm^{-1}): ν_{max} 3427, 3211, 1680, 1655, 1619, 1447, 1370. ^{1}H NMR (400 MHz, DMSO-d_6): δ 0.78 (s, 3H, CH_3), 0.83 (s, 3H, CH_3), 1.85 (m, 2H, CH_2), 2.16 (m, 2H, CH_2), 5.33(s, 1H, CH), 7.12 (m, 4H, HAr), 7.51 (s, 1H, NH), 9.29 (s, 1H, NH) ppm. Anal. Calcd. for $C_{16}H_{17}ClN_2O_2$ (304.78): C 63.05, H 5.62, N 9.19; found: C 63.23, H 5.77, N 9.38.

7,7-Dimethyl-4-phenyl-2-thioxo-2,3,4,6,7,8-hexahydro-5(1*H*)-quinazolinone (**4f**): Yield 263 mg (92%). White powder. M.p. 281–283 °C. IR (KBr, cm^{-1}): ν_{max} 3280, 3204, 1711, 1620, 1572, 1451, 1383. ^{1}H NMR (400 MHz, DMSO-d_6): δ 0.90 (s, 3H, CH_3), 1.02 (s, 3H, CH_3), 2.21 (m, 2H, CH_2), 2.42 (m, 2H, CH_2), 5.23 (s, 1H, CH), 7.26 (m, 3H, HAr), 7.33 (m, 2H, HAr), 9.70 (s, 1H, NH), 10.61 (s, 1H, NH) ppm. Anal. Calcd. for $C_{16}H_{18}N_2OS$ (286.39): C 67.10, H 6.33, N 9.78; found: C 66.91, H 6.41, N 9.56.

7,7-Dimethyl-4-(4-methylphenyl)-2-thioxo-2,3,4,6,7,8-hexahydro-5(1*H*)-quinazolinone (**4h**): Yield 273 mg (91%). White powder. M.p. 279–280 °C. IR (KBr, cm^{-1}): ν_{max} 3307, 3184, 1639, 1565, 1431, 1344. ^{1}H NMR (400 MHz, DMSO-d_6): δ 0.91 (s, 3H, CH_3), 1.04 (s, 3H, CH_3), 2.08 (m, 2H, CH_2), 2.20 (s, 2H, CH_2), 2.26 (s, 3H, CH_3), 5.17 (s, 1H, CH), 7.13 (m, 4H, HAr), 9.63 (s, 1H, NH), 10.56 (s, 1H, NH) ppm. Anal. Calcd. for $C_{17}H_{20}N_2OS$ (300.42): C 67.97, H 6.71, N 9.32; found: C 67.61, H 6.53, N 9.23.

4-Phenyl-3,4-dihydro-2*H*-chromeno[4,3-d]pyrimidine-2,5(1H)-dione (**6a**): Yield 278 mg (95%). White powder. M.p. 162–163 °C. IR (KBr, cm^{-1}): ν_{max} 3404, 2952, 2677, 2344, 1673, 1439, 1391. ^{1}H NMR (400 MHz, DMSO-d_6): δ 6.31 (s, 1H, CH), 7.38 (m, 9H, HAr), 7.90 (s, 1H, NH), 8.03 (s, 1H, NH) ppm. Anal. Calcd. for $C_{17}H_{12}N_2O_3$ (292.29): C 69.86, H 4.14, N 9.58; found: C 69.64, H 3.96, N 9.69.

4-(Dimethylaminophenyl)-3,4-dihydro-2*H*-chromeno[4,3-d]pyrimidine-2,5(1*H*)-dione (**6c**): Yield 322 mg (96%). Brick-red powder. M.p. 242–244 °C. IR (KBr, cm^{-1}): ν_{max} 3412, 3115, 2904, 2716, 2584, 1685, 1606, 1560, 1536, 1441, 1359. ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.17 (s, 6H, CH_3), 6.50 (s, 1H, CH), 7.30 (m, 8H, HAr), 7.84 (s, 1H, NH), 8.01 (s, 1H, NH) ppm. Anal. Calcd. for $C_{19}H_{17}N_3O_3$ (335.36): C 68.05, H 5.11, N 12.53; found: C 68.21, H 5.24, N 12.60.

4-(4-Methoxyphenyl)-2-thioxo-1,2,3,4-tetrahydro-5*H*-chromeno[4,3-d]pyrimidine-5-one (**6f**): Yield 315 mg (93%). White powder. M.p. 265–267 °C. IR (KBr, cm^{-1}): ν_{max} 3389, 3102, 1953, 1705, 1622, 1605, 1578, 1491, 1450, 1326. ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.77 (s, 3H, OCH_3), 6.44 (s, 1H, CH), 6.94 (m, 8H, HAr), 8.04 (s, 1H, NH), 8.07 (s, 1H, NH) ppm. Anal. Calcd. for $C_{18}H_{14}N_2O_3S$ (338.38): C 63.89, H 4.17, N 8.28; found: C 63.71, H 4.07, N 8.11.

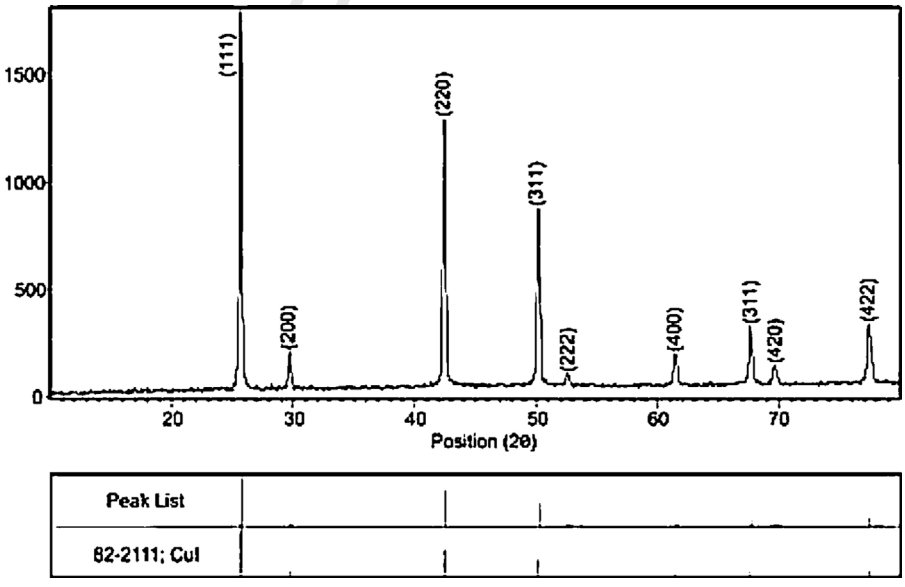

Fig. 1. XRD pattern of the synthesized CuI NPs.

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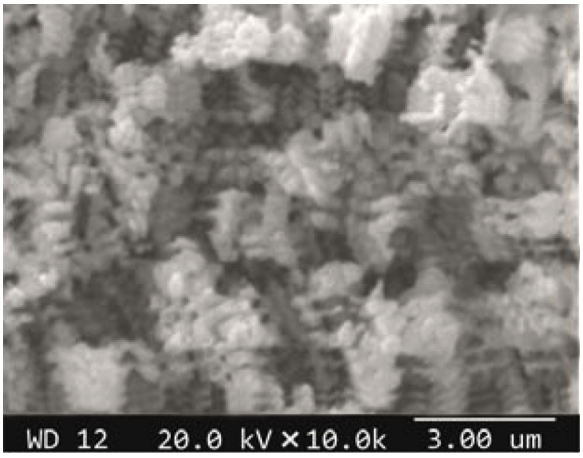

Fig. 2. SEM image of the synthesized CuI NPs.

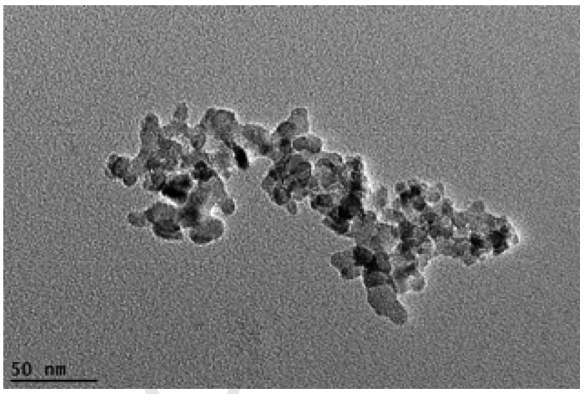

Fig. 3. TEM image of the synthesized CuI NPs.

3. Results and discussion

In continuation of our research program towards highly expedient methodologies for the synthesis of heterocyclic compounds using nanostructured catalysts, we have found that CuI nanoparticles (CuI NPs) are suitable for the preparation of quinazolinones **4**(**a–h**) and chromeno[*d*]pyrimidinones **6**(**a–f**) by the three-component coupling reaction of aryl aldehydes, urea/thiourea and active methylene compounds (dimedone/4-hydroxycoumarin) at 70 °C under solvent-free conditions (Fig. 1).

In a preliminary study, CuI NPs were prepared according to the published procedure of Salavati-Niasari et al. [44]. The crystalline structure and purity of the CuI NPs was confirmed from XRD analyses. The XRD results confirmed the presence of CuI NPs with high crystallinity and a cubic structure with space groupings of *F*-43*m* and cell constants 6.0545 Å (JCPDS 82-2111 of CuI for confirmation). The SEM image of CuI NPs is presented in Fig. 2 which shows triangular shapes with a size range of 30–40 nm of nanoparticles. The TEM image of CuI NPs is also shown in Fig. 3.

We then studied on the optimization of the reaction conditions using a model reaction of 2-chlorobenzaldehyde (**1b**), urea (**2b**) and dimedone (**3**) to afford the corresponding product (**4b**) under various reaction conditions. The results are summarized in Table 1. First, we checked the efficiency of CuI NPs as a heterogeneous catalyst using different amounts of CuI NPs. The results in Table 1 show that using just 10 mol% of catalyst affected the efficiency of the reaction (Table 1, entries 1–4). In investigating the effect of the reaction media, several solvents were also examined. Thus, the best yield of product was obtained under solvent-free conditions (Table 1, entries 3 and 5–7). To determine the optimized reaction temperature, the model reaction was carried out at different temperatures. It was observed that the optimal temperature of 70 °C was best and higher temperatures up to 80 and 100 °C did not improve the yield of product (Table 1, entries 3 and 8–10). During

Table 1
Synthesis of **4b** by the reaction of 2-chlorobenzaldehyde, urea and dimedone under different conditions.

Entry	Mole ratio of the reactants (2-chlorobenzaldehyde: urea:dimedone)	Catalyst (mol%)	Solvent	Temp. (°C)	Time (min)	Yield (%)[a]
1	1:1.2:1	No catalyst	Neat	100	90	43
2	1:1.2:1	CuI NPs (5%)	Neat	80	60	68
3	1:1.2:1	CuI NPs (10%)	Neat	70	60	93
4	1:1.2:1	CuI NPs (15%)	Neat	70	60	93
5	1:1.2:1	CuI NPs (10%)	CH_2Cl_2	70	90	69
6	1:1.2:1	CuI NPs (10%)	DMF	100	80	73
7	1:1.2:1	CuI NPs (10%)	H_2O	90	90	70
8	1:1.2:1	CuI NPs (10%)	Neat	60	90	79
9	1:1.2:1	CuI NPs (10%)	Neat	80	60	94
10	1:1.2:1	CuI NPs (10%)	Neat	100	60	94
11	5:6:5	CuI NPs (10%)	Neat	70	60	95

[a] Isolated yield.

Table 2
CuI NPs catalyzed syntheses of quinazolinones **4**(**a–h**) and chromeno[*d*]pyrimidinones **6**(**a–f**) under solvent-free conditions.

Active methylene compounds	Product	Ar	X	Time (min)	Yield (%)[a]	MP (°C)	
						Obs.	Lit.
3	**4a**	C_6H_5	O	50	94	285–287	288–290 [42]
3	**4b**	2-ClC_6H_4	O	60	93	269–270	271–273 [42]
3	**4c**	4-$MeOC_6H_4$	O	50	90	274–276	272–274 [42]
3	**4d**	4-MeC_6H_4	O	45	90	298–300	300–302 [42]
3	**4e**	3-$NO_2C_6H_4$	O	45	93	300–301	297–299 [42]
3	**4f**	C_6H_5	S	60	92	281–283	280–282 [42]
3	**4g**	4-$MeOC_6H_4$	S	60	93	271–273	268–270 [42]
3	**4h**	4-MeC_6H_4	S	50	91	279–280	280–282 [42]
5	**6a**	C_6H_5	O	45	95	162–163	160–162 [43]
5	**6b**	2-ClC_6H_4	O	60	92	205–206	205–207 [43]
5	**6c**	4-$N(CH_3)_2C_6H_4$	O	50	96	242–244	239–241 [43]
5	**6d**	C_6H_5	S	50	92	187–189	188–190 [43]
5	**6e**	4-$N(CH_3)_2C_6H_4$	S	60	90	233–234	232–234 [43]
5	**6f**	4-$MeOC_6H_4$	S	45	93	265–267	264–266 [43]

[a] Isolated yield.

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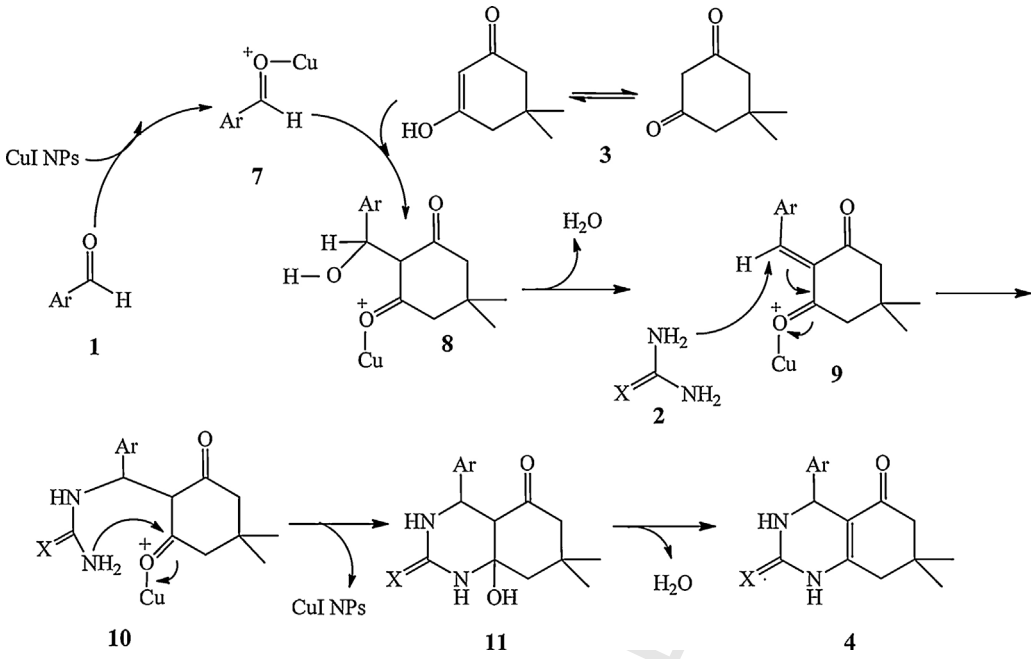

Scheme 2. Proposed mechanism for the reaction of aryl aldehyde, urea/thiourea and dimedone as active methylene compound, catalyzed by CuI NPs.

optimization, we then explored the role of mole ratio of the reactants and determined, that the present method is effective for large-scale synthesis as well as routine scale (Table 1, entries 3 and 11).

To determine the utility of this new procedure, we examined some substituted benzaldehydes for the reaction with urea/thiourea and active methylene compounds, including dimedone or 4-hydroxycoumarin, to obtain desired products under optimized conditions (Table 2). The isolated compounds **4**(**a–h**) and **6**(**a–f**) were characterized by IR and ^{1}H NMR spectroscopic data and also by elemental analyses. The analytical, spectroscopic and physical data are in good agreement with those described in the literature. Selected spectroscopic data are reported in Section 2.2.

A plausible mechanism for the formation of product **4** is given in Scheme 2. It is feasible that CuI NPs participates in two following catalytic cycles. The initial event is the formation of copper oxonium salt **7** which then undergoes a Knoevenagel condensation with dimedone **3**, to generate alkene **9** *via* intermediate **8**. The urea/thiourea **2** then adds to alkene **9** to produce the Michael adduct **10**. Further cyclization of **10** gives product **4**, after dehydration. The formation of product **6** is likely to occur *via* a tandem Knoevenagel–Michael condensation as that was explained in Scheme 2, for the formation of product **4**.

Moreover, we evaluated separation, isolation and recycling of the CuI NPs catalyst, which was successfully recycled as mentioned in Section 2.2 and reused several times in the model reaction with no significant loss of activity (Fig. 4). From the XRD patterns of the second and third recycled catalyst reuse, the character of CuI in the cubic phase is not changed and could be compared to the original one (Fig. 5).

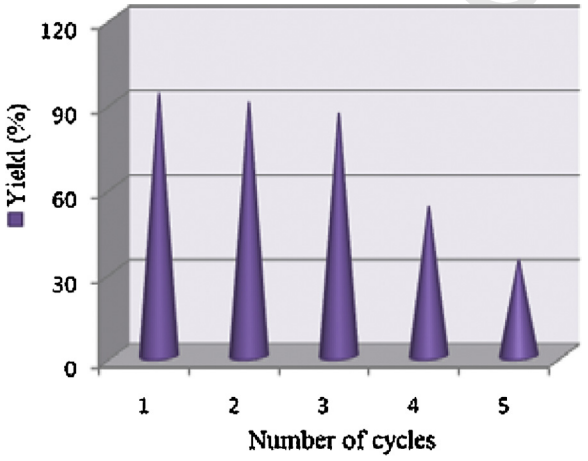

Fig. 4. Reuse of CuI NPs for the synthesis of **4b**.

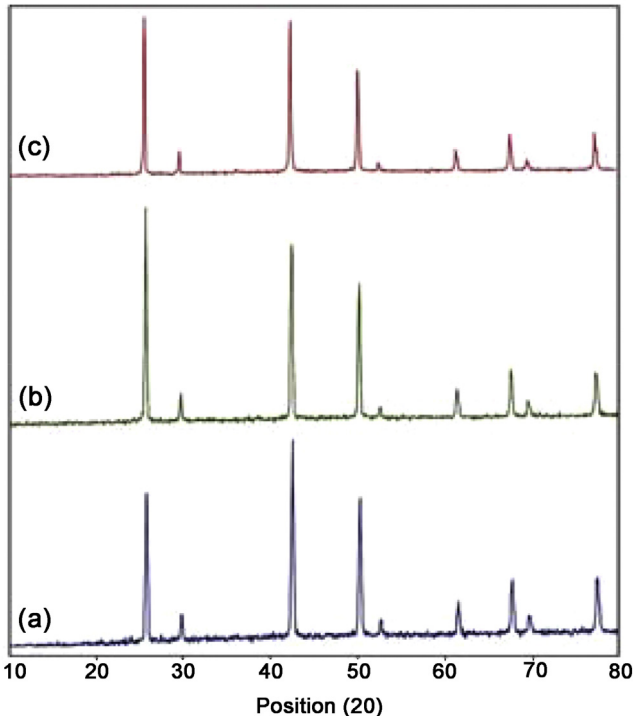

Fig. 5. XRD patterns of (a) original catalyst, (b) 2nd recycled catalyst use, and (c) 3rd recycled catalyst use.

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4. Conclusion

In summary, we have developed an efficient and less wasteful manufacturing method for the synthesis of some of quinazolinone and chromeno[*d*]pyrimidinone derivatives from aryl aldehydes, urea/thiourea and active methylene compounds, including dimedone or 4-hydroxycoumarin, using CuI NPs as reusable and inexpensive heterogeneous catalysts under solvent-free conditions. This new catalytic method has several advantages including high yields of products, short reaction time, simple operation and use of reusable, non-toxic and inexpensive catalyst.

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