5,5′-Indigodisulfonic acid as an efficient catalyst for the synthesis of 2,3-dihydroquinazolinone derivatives

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ABSTRACT
An efficient protocol for the synthesis of 2,3-dihydroquinazolinone derivatives from substituted 2-aminobenzamides and carbonyl analog catalyzed by the natural-based 5,5′-indigodisulfonic acid has been developed with good to excellent yields. Compared to the reaction conditions reported before, the use of eco-friendly and recyclable catalysts and mild reaction conditions are the major advantages of the present method.

GRAPHICAL ABSTRACT

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Introduction

Indigo, as one of the most well-known dyes, has been used worldwide for thousands of years.[1] Natural indigo can be obtained from varied species,[2] and in ancient China and India, indigo was obtained mainly from Polygonum tinctorium Ait. and Indigofera tinctoria Linn, respectively. In the current market, indigo is used mainly for dyeing cotton yarn to produce blue jeans. In addition, indigo and derivatives have also received considerable attention for their electron-accepting properties and good performance in organic semiconductors recently.[3] 5,5'-Indigodisulfonic acid (Scheme 1), the sulfonated product of indigo, is one of the most important derivatives of this dye due to good solubility in an aqueous solution. The sodium salt of 5,5'-indigodisulfonic acid, also known as indigo carmine, is widely used as a coloring substance and redox indicator in analytical chemical technology, biological detection, pharmaceutical and food industries.[4] However, the application of indigo and its derivatives as catalysts in organic synthesis are rarely studied as we know.

2,3-Dihydroquinazolinone derivatives are an important class of fused heterocyclic compounds with impressive biological and pharmaceutical activities such as diuretic, antihypertensive, antibacterial, antifungal and anticancer.[5–7] Some examples of biologically active molecules and commercial drugs bearing 2,3-dihydroquinazolinone moieties are shown in Figure 1. Numerous synthetic methods have been developed for the preparation of 2,3-dihydroquinazolinone moiety, and one of the most widely-used methods is the cyclocondensation reaction between 2-aminobenzamide and carbonyl analog in the presence of various catalysts[8,9] such as NH$_4$Cl,[10] InBr$_3$,[11] ZrCl$_4$,[12] TsOH,[13] Mukaiyama’s reagent,[14] B(C$_6$F$_5$)$_3$,[15] KOH[16] and Cp$_2$TiCl$_2$.[17] Nowadays, to reduce reagents that are harmful to the environment, using natural-based organic compounds as eco-friendly catalysts for organic synthesis is considered by many researchers. It was reported that the synthesis of 2,3-dihydroquinazolinone derivatives could be promoted by using β-cyclodextrin,[18] lactic acid,[19] glycerol[20] or ascorbic acid[21] as catalysts. In addition, natural-based sulfuric acids such as β-cyclodextrin-SO$_3$H,[22] choline sulfonic acid,[23] and cellulose sulfonic acid[24] were also suitable catalysts for this transformation.[25] However, these mentioned conditions suffer from limitations such as high reaction temperature, long reaction time, high catalyst loading, poor product purity or toxic reagents. Therefore, it is still necessary to develop efficient, convenient, and sustainable methods for the synthesis of 2,3-dihydroquinazolinone derivatives (See Table S2 in Supplemental Material).

Our group has a strong interest in the application of renewable and eco-friendly plant dyes in organic synthesis. Indigo, which could be obtained from various sources of plants, however, has rarely been studied in organic synthesis up to now. To show the possibility of using natural-based plant dyes as catalysts in organic synthesis, herein, we

Scheme 1. Preparation of 5,5'-indigodisulfonic acid.
reported our experimental results involving the synthesis of 2,3-dihydroquinazoline derivatives using 5,5'-indigodisulfonic acid as a catalyst for the first time.

Results and discussion

We commenced our investigation with 2-aminobenzamide (1a) and 4-methoxybenzaldehyde (2a) using 5,5'-indigodisulfonic acid as a catalyst. The catalyst could be easily synthesized from the very cheap reagent indigo in one step (Scheme 1). Our first experiment was conducted by using 2 mol% 5,5'-indigodisulfonic acids as catalyst and water as solvent. To our delight, the desired product 3a could be achieved in 86% yield after being heated at 80°C for 60 min (Table 1, entry 1). Other solvents such as MeOH and EtOH were also suitable for this transformation and good yields were achieved after 10 min (Table 1, entry 2–3). We also tried solvents such as 1,4-dioxane and THF, finding that both solvents gave lower yields, compared to those reactions conducted in alcohol solvents, probably due to the low solubility of the catalyst in 1,4-dioxane and THF (Table 1, entries 4–5).

Next, the effect of catalyst loading and temperature on reaction yields was investigated (Table 1, entries 6–7). Catalyst loading could be reduced to 1 mol% and reaction temperature could be reduced to 60°C, without the loss of yield. A longer reaction time was needed when using 1 mol% catalyst loading. A slightly higher yield could be obtained when increasing the concentration of the reaction system (Table 1, entry 8). In addition, we also found that 5,5'-indigodisulfonic acid was essential for this transformation, while only trace product 3a could be detected in the absence of this catalyst.

With the optimal reaction conditions in hand, we next moved our attention to other aldehydes and 2-aminobenzamide derivatives, and the results were shown in Table 2. We found that the electronic properties of the substitutions in the benzene rings of aromatic aldehydes had little effect on the yields of the target 2,3-dihydroquinazolinone compounds. Both electron-withdrawing and -donating groups gave good yields (Table 2, 3a–h). However, aldehydes with strongly electron-withdrawing groups, such as the -NO₂ group, on aromatic ring gave the products with slightly lower yield in a longer
Table 1. Optimization of conditions for the synthesis of the 2,3-dihydroquinazolinone 3a.

| Entry | Catalyst loading | Solvent | Temperature (°C) | Time (min) | Yield (%) b |
|-------|------------------|---------|------------------|------------|-------------|
| 1     | 2%               | H2O     | 80               | 60         | 86          |
| 2     | 2%               | MeOH    | 80               | 10         | 87          |
| 3     | 2%               | EtOH    | 80               | 10         | 84          |
| 4     | 2%               | 1,4-Dioxane | 80       | 60         | 58          |
| 5     | 2%               | THF     | 80               | 60         | 60          |
| 6     | 2%               | MeOH    | 60               | 10         | 88          |
| 7     | 1%               | MeOH    | 60               | 30         | 87          |
| 8     | 1%               | MeOH    | 60               | 30         | 90          |
| 9     | –                | MeOH    | 60               | 30         | trace       |

The reaction was carried out in 1.0 mmol scale in 6 mL solvent. bIsolated yield.

Table 2. 5,5'-Indigidosulfonic acid-catalyzed synthesis of dihydroquinazolinones derivatives.

Optimal reaction conditions: 1 (1.0 mmol), 2 (1.1 mmol), catalyst (0.01 mmol), MeOH (3 mL), 60 °C. All the yields shown here were isolated yields.

| R1    | R2 | R1-CHO | R2 or R3 | Catalyst loading | MeOH (9 mL) | Temperature (°C) | Time (min) | Yield (%) |
|-------|----|--------|----------|------------------|-------------|------------------|------------|-----------|
| 3a    | Cl |        |          | 5,5'-Indigidosulfonic acid (1 mol%) | 60         | 30 min | 90%       |
| 3b    | R2 |        |          |                  |             | 30 | 90%       |
| 3c    | R3 |        |          |                  |             | 30 | 86%       |
| 3d    | R1 |        |          |                  |             | 30 | 86%       |
| 3e    | R2 |        |          |                  |             | 30 | 89%       |
| 3f    | R3 |        |          |                  |             | 30 | 67%       |
| 3g    | R1 |        |          |                  |             | 30 | 68%       |
| 3h    | R2 |        |          |                  |             | 30 | 85%       |
| 3i    | R3 |        |          |                  |             | 30 | 80%       |
| 3j    | R1 |        |          |                  |             | 30 | 73%       |
| 3k    | R2 |        |          |                  |             | 30 | 90%       |
| 3m    | R1 |        |          |                  |             | 30 | 86%       |
| 3n    | R2 |        |          |                  |             | 30 | 89%       |
| 3o    | R3 |        |          |                  |             | 30 | 82%       |
| 3p    | R1 |        |          |                  |             | 30 | 84%       |
| 3q    | R2 |        |          |                  |             | 30 | 92%       |
| 3s    | R3 |        |          |                  |             | 30 | 78%       |
| 3t    | R1 |        |          |                  |             | 30 | 88%       |
| 3u    | R2 |        |          |                  |             | 30 | 73%       |
| 3v    | R3 |        |          |                  |             | 30 | 90%       |

Optimal reaction conditions: 1 (1.0 mmol), 2 (1.1 mmol), catalyst (0.01 mmol), MeOH (3 mL), 60 °C. All the yields shown here were isolated yields.

MeOH was used as the solvent.

3 mol% catalyst loading.

6.0 mmol ketone was used.

10 mol% catalyst loading.

1.5 mmol ketone was used.

1.0 mmol ketone was used.
The position of the substitutions on the aryl ring of the aldehyde also had limited effects on the outcome of the reaction. Ortho-, meta- and para-bromo substituted benzaldehyde gave similar yields (Table 2, 3b–d). Besides substituted benzaldehydes, heteroaromatic aldehyde and cinnamaldehyde could also give the corresponding product in moderate to good yields (Table 2, 3i–j).

We also synthesized substituted 2-aminobenzamide and found that 2-aminobenzamide derivatives with substitution in the aryl ring or the nitrogen atom smoothly transformed to desired products with 73–92% yields (Table 2, 3k–r).

Finally, the above optimal reaction conditions were then applied to the reaction between 2-aminobenzamide and ketones (Table 2, 3s–u). Moderate yields were obtained with a longer reaction time and higher catalyst loading, probably due to the steric effect of ketones.

The dihydroquinazolinone derivatives generated from 2-aminobenzamide can be easily prepared on large scales without the loss of yields (Scheme 2). Products 3a, 3h and 3n were isolated in 90%, 93% and 86% yields, respectively. In addition, although the catalyst loading was rather low, the recyclability of the catalyst was investigated using a model reaction between 2-aminobenzamide and 4-bromobenzaldehyde, in the interests of green chemistry and environmentally benign process (Table 3). After the separation of desired product, the filtrate containing the catalyst was used as a solvent for the next run without adding an excess catalyst (See Supplemental Material for details). As shown in Table 3, the catalyst could be recycled for at least 4 cycles, without a significant decrease in yields. Longer reaction time was needed in the 3rd and 4th cycles, indicating the inadequate recovery of the catalyst due to the residue in the isolated product and attrition during filtration.

Finally, the multi-component reactions between isatoic anhydride, amines and aldehydes were also investigated using 5,5'-indigodisulfonic acid as a catalyst. To our disappointment, higher reaction temperature and catalyst loading was needed and only moderate yields could be obtained. The results are summarized in Table 4.

Based on the relevant literature, a plausible mechanism for this transformation was proposed (Scheme 3). At first, 5,5'-indigodisulfonic acid as a Bronsted acid activates
the carbonyl group, followed by nucleophilic addition and dehydration condensation to give the imine intermediate II. Afterward, the imine intermediate II could be further activated by the Brønsted acid and the target product would be obtained by the intramolecular nucleophilic attack of the amide group on the activated imine group.

**Conclusion**

In summary, an efficient and convenient preparation of 2,3-dihydroquinazolinone derivatives by ring closure of substituted 2-aminobenzamide with carbonyl analog using the natural-based 5,5′-indigodisulfonic acid as a catalyst has been developed. The advantages of this protocol are mild reaction conditions, low catalyst loading, easy separation of products and recyclability of catalyst.

**Experimental**

**Synthesis of 5,5′-indigodisulfonic acid**

Indigo (10.00 g, 38 mmol) was added into concentrated H₂SO₄ (38.00 g, 380 mmol) at 100 °C and the mixture was further maintained at 100 °C for 2 h. Then the reaction
mixture was cooled to room temperature and 150 mL AcOH was added. The precipitated solid was collected by centrifugation and further washed with 10 mL AcOH and 120 mL EA. The title compound was obtained as a dark blue solid (12.36 g, 77%). m.p. = $>300^\circ$C. $^1$H NMR (400 MHz, D$_2$O) $\delta$ 7.74 (s, 2H), 7.40 (d, $J$ = 8.3 Hz, 2H), 6.32 (d, $J$ = 8.3 Hz, 2H). $^{13}$C NMR (101 MHz, D$_2$O) $\delta$ 190.23, 154.83, 137.75, 136.05, 123.93, 123.75, 120.34, 115.12.

**Procedure for the synthesis of 2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one (3a) from 2-aminobenzamide**

A mixture of the 2-aminobenzamide (1.00 mmol), 4-methoxybenzaldehyde (1.10 mmol) and 5,5'-indigodisulfonic acid (0.01 mmol, 1 mol%) in MeOH (3 mL) was continuously stirred at 60 $^\circ$C for 30 min. When TLC (PE/EA 1:3) indicated the disappearance of the starting material, the reaction mixture was cooled to room temperature and 15 mL of cold water was added. The precipitated solid was then filtered and washed with cold water (5 mL). The solid was pure enough and no further purification was needed.

3a: Yield: 90%, off-white solid, TLC $R_f$ = 0.74 (PE/EA = 1:3). m.p. = 187–188 $^\circ$C. $^1$H NMR (400 MHz, DMSO) $\delta$ 8.19 (s, 1H), 7.61 (dd, $J$ = 7.7, 1.3 Hz, 1H), 7.42 (d, $J$ = 8.7 Hz, 2H), 7.27 – 7.20 (m, 1H), 7.01 (s, 1H), 6.95 (d, $J$ = 8.7 Hz, 2H), 6.74 (d, $J$ = 8.0 Hz, 1H), 6.70 – 6.62 (m, 1H), 5.70 (s, 1H), 3.75 (s, 3H). $^{13}$C NMR (101 MHz, DMSO) $\delta$ 163.68, 159.42, 148.01, 133.46, 133.22, 128.20, 127.33, 117.07, 114.99, 114.40, 113.62, 66.29, 55.17. The $^1$H NMR & $^{13}$C NMR spectra were consistent with the literature.[24]

**Procedure for the synthesis of 2-(4-methoxyphenyl)-3-phenyl-2,3-dihydroquinazolin-4(1H)-one (3k) from isatoic anhydride**

A mixture of the isatoic anhydride (1.00 mmol), aniline (1.00 mmol) and 5,5'-indigodisulfonic acid (0.05 mmol, 5 mol%) in EtOH/H$_2$O (6 mL, v/v 1:1) was continuously stirred at 100 $^\circ$C for 20 min. Then 4-methoxybenzaldehyde (1.10 mmol) was added and the mixture was further stirred at 100 $^\circ$C for 2 h. When TLC (PE/EA 2:1) indicated the disappearance of the starting material, the reaction mixture was cooled to room temperature and 15 mL of cold water was added. The precipitated solid was then filtered and washed with cold water (5 mL). The solid was pure enough and no further purification was needed.

3k: Yield: 47%, off-white solid, TLC $R_f$ = 0.43 (PE/EA = 2:1). m.p. = 210–212 $^\circ$C. $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.01 (dd, $J$ = 7.8, 1.3 Hz, 1H), 7.32 – 7.24 (m, 5H), 7.20 – 7.11 (m, 3H), 6.91 – 6.85 (m, 1H), 6.79 – 6.72 (m, 2H), 6.62 (d, $J$ = 8.0 Hz, 1H), 6.06 (s, 1H), 4.74 (s, 1H), 3.74 (s, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 163.18, 159.94, 145.44, 140.53, 133.79, 131.88, 129.02, 128.86, 128.19, 127.09, 126.73, 119.49, 116.86, 114.77, 113.94, 74.30, 55.21. The $^1$H NMR & $^{13}$C NMR spectra were consistent with the literature.[11]

Full experimental detail, $^1$H and $^{13}$C NMR spectra. These materials can be found via the “Supplementary Content” section of this article’s webpage.
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Disclosure statement

The authors declare no conflict of interest.

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