



Alginate acid in water at room temperature: a natural combination for the environmental benign synthesis of 2,3-dihydroquinazolin-4(1H)-ones

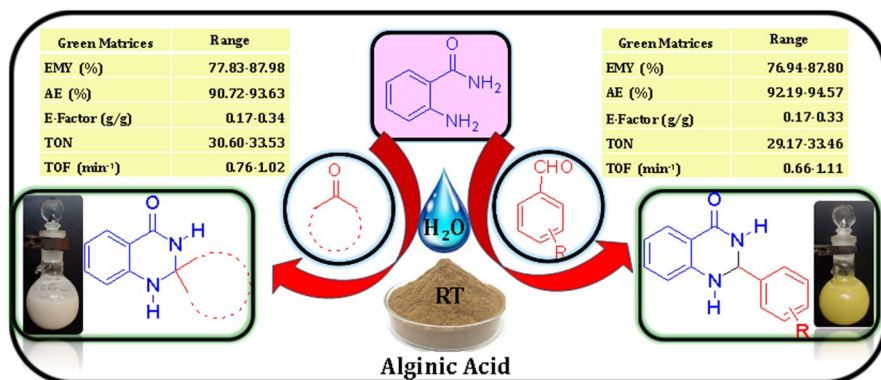
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Abstract

We have successfully synthesized 2,3-dihydroquinazolin-4(1H)-ones (DHQs) using naturally sourced alginate acid as a solid acid catalyst. Our synthetic method was performed in pure water at room temperature and yielded good to excellent results. We achieved this by directly cyclo-condensing anthranilamide with various aldehydes or ketones in the presence of alginate acid in water at room temperature. Furthermore, the alginate acid can be easily separated from the reaction mixture and reused up to five times with consistent yields and reaction time. This method is energy efficient and can be applied on a large scale, with excellent green credentials. Our evaluation of green metrics highlights the sustainable features of the alginate acid-catalyzed DHQ synthesis process.

Graphical abstract



Keywords Alginate acid · Green synthesis · 2,3-Dihydroquinazolin-4(1H)-ones · Aqueous medium · Biopolymers

Extended author information available on the last page of the article

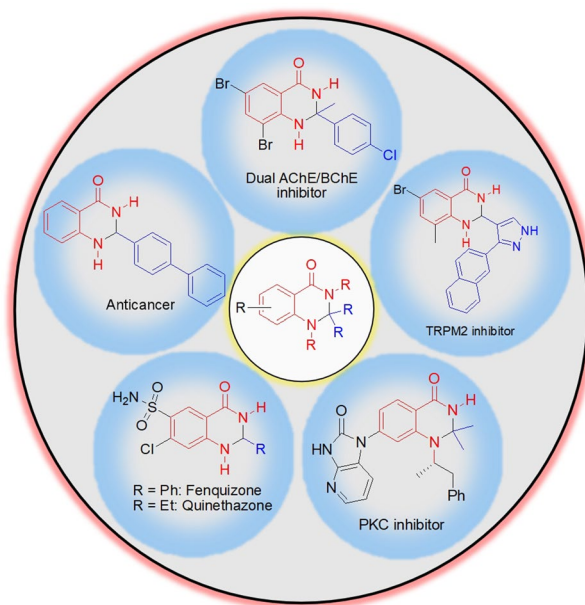


Fig. 1 Crucial pharmaceutical active compounds containing DHQ framework

Introduction

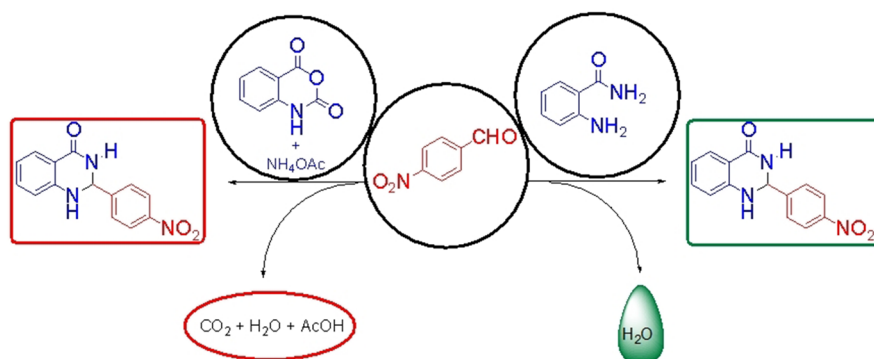
Quinazolinone scaffolds are a type of fused heterocycles that have potential pharmacological and biological activities [1, 2]. They are essential units found in natural products, agrochemicals, pharmaceuticals, and synthetic drugs (Fig. 1) [3]. One particular type of heterocyclic framework, 2,3-dihydroquinazolin-4(1H)-ones (DHQs), plays a crucial role in various medications due to its potent pharmacological and biological effects, such as anticancer, anticonvulsant, analgesic, diuretic, antihistamine, and antihypertensive activities [4–13].

DHQs are essential in many fields, and various methods have been reported for their synthesis, such as two and three-component reactions under different reaction conditions [14] (Fig. 2). The simplest and most direct method is the two-component condensation of anthranilamide with aldehydes or ketones. On the other hand, the three-component condensation of isatoic anhydride and different amines with aldehydes is another method. However, a two-component reaction is more environmentally friendly than a three-component reaction, according to green chemistry matrices. Many of the reported protocols use costly reagents, require extended reaction times and high temperatures, and involve tedious work-up procedures. The atom economy (AE) indicates that a two-component reaction is better than a three-component reaction.

In recent years, various catalysts have been used to catalyze these reactions such as cyanuric chloride [15], morpholinoethanesulfonic acid [16], *p*-TSA [11], 5,5'-indogodisulfonic acid [17], lactic acid [18], oxalic acid [19], citric acid [20], amino acid

[21], $\text{Sc}(\text{OTf})_3$ [22], $\text{Y}(\text{OTf})_3$ [23], $\text{Ga}(\text{OTf})_3$ [24], $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ [25], $\text{H}_3\text{PW}_{12}\text{O}_{40}$ [26], NH_4Cl [27], $\text{H}[\text{Gly}_2\text{B}]$ [28], $[\text{Bmim}]\text{BF}_4$ [22], choline-sulfuric acid ionic liquid [9], basic ionic liquid [29], magnetic supported catalysts like Fe_3O_4 -Schiff base of $\text{Cu}(\text{II})$ [30], $\text{Fe}_3\text{O}_4@/\text{SiO}_2\text{-SO}_3\text{H}$ [31], $\text{IL}@/\text{MNP}$ [32], $\text{Fe}_3\text{O}_4@/\text{PEG-Ni}$ [33], $\text{Fe}_3\text{O}_4@/\text{L-proline-SO}_3\text{H}$ [34] silica-supported catalysts such as MCM-41-dtz-Ni [35], $\text{SBA-16/GPTMS-TSC-CuI}$ [36], $\text{CoFe}_2\text{O}_4@/\text{SiO}_2\text{-CPTES-Guanidine-Cu}(\text{II})$ [37] Hb zeolite [38], zinc oxide micelles [39], mono-ammonium phosphate fertilizer modified by cadmium [40], cerium(IV) ammonium nitrate [41], trifluoroethanol [42], $[\text{PEG-TEA}]\text{OH}$ [43], polyethylene glycol [44], and biocomposites such as β -cyclodextrin- SO_3H [45], $\text{Fe}_3\text{O}_4@/\text{nano-cellulose-OPO}_3\text{H}$ [46], β -cyclodextrin [47], Ag-CNTs [48] has been also used for the synthesis of DHQs.

Over the past 20 years, there has been a growing need for more sustainable organic transformations that reduce waste [49–51]. DHQs are important, so it is highly appreciated when organic synthesis are environmentally friendly, high-yielding, and clean. Biopolymers, including solid support and catalysts, are commonly used in various organic transformations [52, 53]. Alginic acid is a binary, block polysaccharide consisting of (1–4) connected β -D-mannuronic acid (M) and α -L-guluronic acid (G) residues of varying compositions and sequences. It is a structural



| Three component protocol | Parameter* | Two component protocol |
|--------------------------|------------|------------------------|
| 68.79 | EMY | 93.72 |
| 68.80 | AE | 93.72 |
| 68.80 | Aef | 93.72 |
| 82.35 | CE | 100.00 |
| 68.79 | RME | 93.72 |
| 99.98 | OE | 100.00 |
| 1.45 | PMI | 1.06 |
| 1.45 | MI | 1.06 |
| 68.96 | MP | 94.33 |
| 0.45 | E-factor | 0.06 |

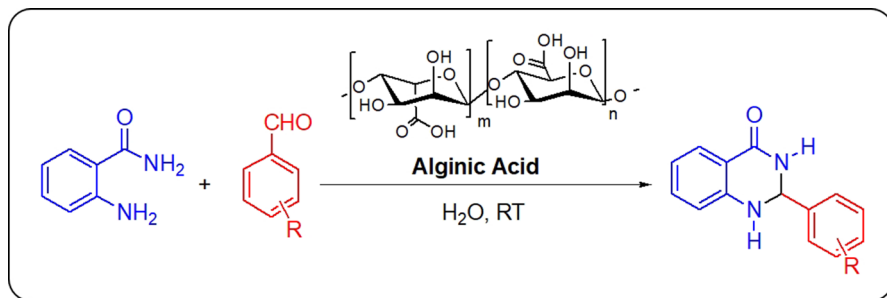
*All the calculated parameters are based on

(a) Solvent and catalyst-free conditions

(b) 100% conversion and yield

(c) 1 mmol reaction scale

Fig. 2 Two protocols and green chemistry matrices for the synthesis of DHQs



Scheme 1. Alginic acid-catalyzed synthesis of DHQ in water at room temperature

component found in marine brown algae and many bacteria [54]. It can be extracted as water-soluble sodium alginate by adding an excess of alkali [55]. The carboxyl and hydroxyl groups of adjacent chains help to stabilize reactants during the reaction [56]. Alginic acids are biodegradable, biocompatible, and easily accessible, making them a popular choice as environmentally friendly catalysts for traditional inorganic and organic solid acids [57–59].

Water is a crucial solvent in many biological processes and is widely recognized for its advantages in organic reactions, such as being safe, cost-effective, and environmentally compatible. Recently, new methodologies including multicomponent reactions have been developed that are highly efficient and compatible [60] with green chemistry principles for the synthesis of various heterocyclic scaffolds and cross-coupling reactions [61–64].

We have recently made an effort to introduce new, more efficient methodologies that adhere to green chemistry principles for synthesizing various heterocyclic scaffolds [65, 66] and cross-coupling reactions [67–69]. Alginic acid, due to its unique properties and ability to act as an excellent Brønsted organic acid catalyst, was used as a solid acid catalyst. This involved the cyclo-condensation of anthranilamides and various aldehydes/ketones in water at room temperature (Scheme 1). The resulting products were obtained in good to excellent yields, and the alginic acid can be easily separated from the reaction mixture and recycled at least five times with consistent yield and reaction time.

Materials and methods

General remarks

All reactions were carried out in a round-bottom flask at RT. Chemical reagents and anhydrous solvents were purchased from commercial suppliers (TCI and Sigma-Aldrich chemical companies) and used as purchased. Thin layer chromatography (TLC) was performed using silica gel pre-coated aluminum plates, which were visualized with UV light at 254 nm or under iodine. ^1H NMR and ^{13}C NMR were recorded with Bruker (600 Mz) spectrometers using CDCl_3 and DMSO solvents

Chemical shifts for ^1H NMR are referred to as internal TMS (0 ppm), and chemical shifts for ^{13}C NMR are referenced to the carbon resonance of the solvent (CDCl_3 : δ 77.0 ppm). Data are reported as follows: chemical shift (δ ppm), multiplicity (s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet), coupling constant (Hz), and integration.

General procedure for the synthesis of 2,3-dihydroquinazolin-4(1H)-ones

A mixture of anthranilamide (1 mmol), aldehyde/ketone (1 mmol), and alginic acid (5 mg) in water (5 mL) was stirred at room temperature. Reaction progress was monitored by TLC (ethyl acetate: *n*-hexane, 1:9). After completion of the reaction, 0.1 N NaOH (5 mL) was added to the reaction mixture to separate alginic acid as sodium alginate. The resultant solid product was collected by simple filtration and washed with water (5 mL). This crude product was recrystallized by using ethanol.

Result and discussion

The alginic acid was characterized by FTIR spectroscopy [70]. The broadband IR absorption at 3434 cm^{-1} is for the COOH group which was confirmed by the sharp band at 1744 cm^{-1} (Fig. 3). The weak signal at 2924 and 2854 cm^{-1} is due to C–H stretching vibrations. The asymmetric stretching of carboxylate vibration (O–C–O) is shown at 1628 cm^{-1} . The band at 1402 cm^{-1} may be due to C–OH deformation vibration with the contribution of O–C–O symmetric stretching vibration of the carboxylate group. The band at 1029 cm^{-1} may be assigned to C–O stretching, and C–O and C–C stretching vibrations of pyranose rings.

The porous nature of the alginic acid was evident in scanning electron microscopy (SEM) images. An image shows a relatively fibrous smooth surface about $50\text{ }\mu\text{m}$ thick (Fig. 4a and b) while elemental mapping is highlighted in the EDS micrograph (Fig. 4c).

In order to find the best reaction conditions for synthesizing DHQ, we first reacted anthranilamide with 4-nitrobenzaldehyde under various conditions. Initially,

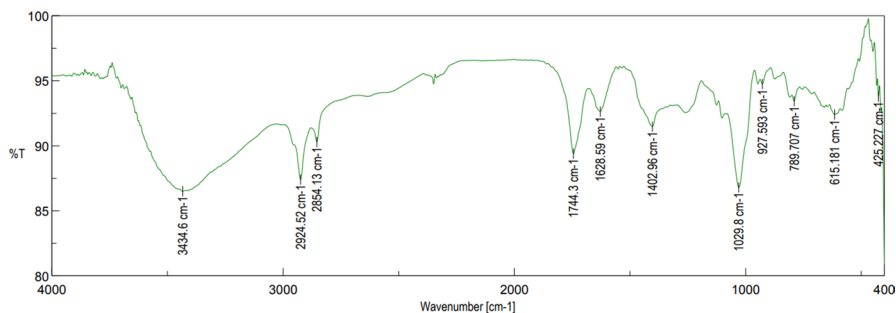


Fig. 3 The FTIR spectrum of alginic acid

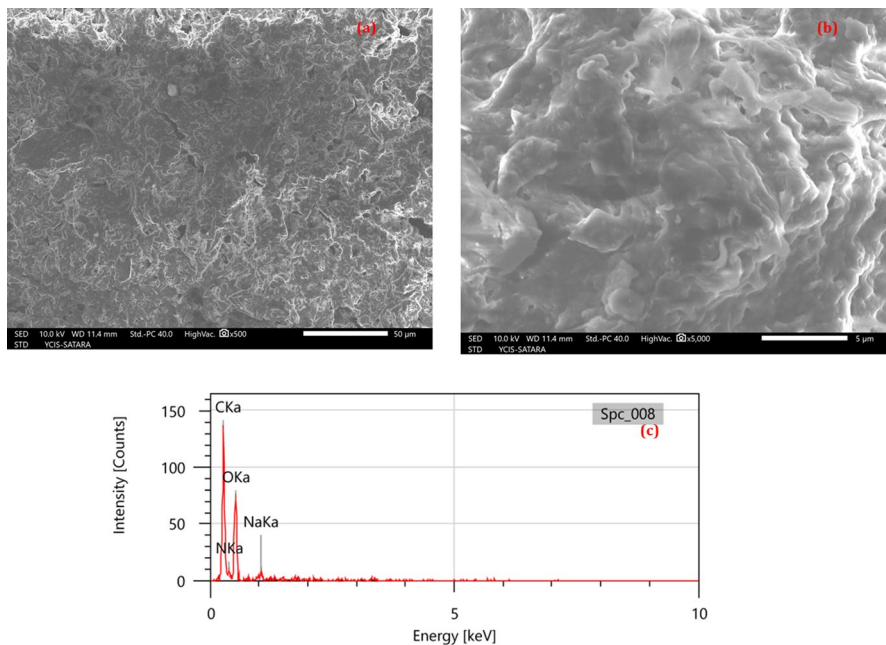
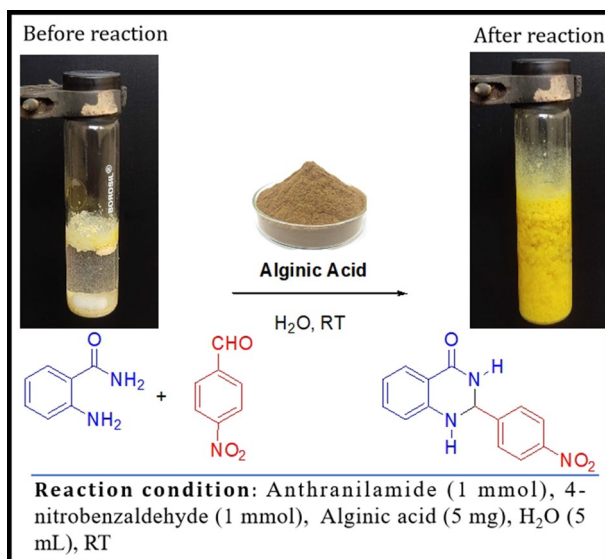


Fig. 4 SEM images (a and b) and EDS micrograph c of alginate acid



Scheme 2. Alginic acid-catalyzed synthesis of DHQ in water at room temperature

Table 1 Influence of quantity of alginic acid on the yield and reaction time of DHQ in water at room temperature^{a,b}

| Entry | Amount of alginic acid (mg) | Time (min) | Yield ^b (%) |
|-------|-----------------------------|------------|------------------------|
| 1 | 0 | 60 | Trace |
| 2 | 2 | 60 | 72 |
| 3 | 5 | 30 | 94 |
| 4 | 10 | 30 | 95 |
| 5 | 20 | 30 | 96 |
| 6 | 30 | 25 | 95 |
| 7 | 40 | 25 | 96 |
| 8 | 50 | 25 | 96 |

^aReaction conditions: Anthranilamide (1 mmol), 4-nitrobenzaldehyde (1 mmol), alginic acid (2–50 mg), H₂O (5 mL), RT

^bIsolated yields

Table 2 Effect of temperature and screening of various solvents for the synthesis of DHQ^{a,b}

| Entry | Solvent | T (°C) | Time (min) | Yield ^b (%) |
|-------|------------------|--------|------------|------------------------|
| 1 | H ₂ O | RT | 30 | 94 |
| 2 | H ₂ O | 40 | 20 | 96 |
| 3 | H ₂ O | 60 | 20 | 95 |
| 4 | H ₂ O | 80 | 15 | 95 |
| 5 | EtOH | RT | 25 | 96 |
| 6 | 50% Aq. EtOH | RT | 30 | 95 |
| 7 | DMF | RT | 40 | 95 |
| 8 | DCM | RT | 55 | 54 |
| 9 | Toluene | RT | 40 | 76 |
| 10 | Acetonitrile | RT | 45 | 81 |

^aReaction conditions: anthranilamide (1 mmol), 4-nitrobenzaldehyde (1 mmol), alginic acid (5 mg, 2.84 mol%), solvent (5 mL), RT–80 °C

^bIsolated yields

we tested all reactions using water as a safe and environmentally friendly solvent [70] at room temperature (27–30 °C) (Scheme 2).

To determine the proper amount of catalyst, we conducted the model reaction using different concentrations of alginic acid (Table 1). Our findings showed that product formation ranged from 72 to 96% for all concentrations tested. Although the yield was similar across all concentrations, the reaction time for 5 mg of catalyst was 30 min compared to only 25–30 min for the other amounts. This suggests that 5 mg of alginic acid is sufficient for a smooth reaction.

To assess how solvents impact the model reaction, we tested a variety of solvents, including EtOH, methanol, toluene, DMF, acetonitrile, and aqueous ethanol (50%), using 5 mg of alginic acid at room temperature (Table 2). Aqueous ethanol proved to be the most effective in terms of product yield and reaction time, while organic

solvents like DCM, toluene, and acetonitrile were less effective. Water is considered the preferred solvent for reactions from a green chemistry perspective, and solvent selection guides often prioritize SH&E criteria (Table S6).

In an effort to reduce the reaction time, we tested the model reaction at various temperatures (Table 2). Increasing the temperature resulted in a decrease in reaction time from 30 to 15 min, but the higher temperature was ultimately more effective. For economic reasons, we carried out further reactions at ambient temperature.

We tested the effectiveness of our optimized conditions by synthesizing different DHQ derivatives (Table 3). This process is important as it shows how well the method can tolerate various functional groups and handle diverse substrates with different properties. We found that a wide range of aromatic aldehydes worked well in this reaction, indicating that our method is highly versatile. All reactions were performed on a 1 mmol scale in water with 5 mg of alginic acid at room temperature, unless otherwise noted. The yields were excellent, ranging from 84–94%. Even aromatic heterocyclic aldehydes were successfully reacted, yielding the desired product with a 84–94% yield.

As we were successful in using different aromatic aldehydes as electrophiles we then used ketones as another electrophiles for the synthesis of different DHQs. By reacting different ketones with anthranilamide, we obtained the desired products with good to excellent yields (86–94%) as shown in Table 4. The reaction also worked well with cyclic ketones such as cyclopentanone, cyclohexanone, and isatin.

We then expanded the scope of our work by creating bis-quinazolinone through the reaction of anthranilamide with terephthalaldehyde under optimized conditions (Scheme 3). The reaction was able to produce the desired products with a yield of 92% in just 35 min.

Based on our results, we have a possible mechanism for the formation of DHQ (3a) shown in Scheme 4. In the first step, alginic acid acts as a powerful Brønsted acid and activates aldehyde (2a) through protonation of the carbonyl group to form an intermediate I. The activated aldehyde I then undergoes a nucleophilic attack by anthranilamide (2) to form imine adduct II. In the presence of alginic acid, imine II is then converted into an iminium intermediate III through a protonation-cyclization process. Finally, DHQ (3a) is generated from III via deprotonation and the catalyst is released for the next cycle.

The efficient recycling of catalysts with consistent activity after a catalytic reaction is very important for green and sustainable development. In addition, recycling the catalyst is an important requirement for the industry's practical application of a catalyst.

After completion of reaction, the alginic acid was extracted using 0.1 N NaOH. The resulting product was separated through simple filtration. The aqueous extract containing sodium alginate was re-precipitated by 0.1N HCl. The solid alginic acid obtained was separated through centrifugation, dried in a vacuum, and reused for the next reaction cycle. The catalyst was observed to be recyclable at least five times, with a slight loss in activity with increased reaction time from 30–55 min. The catalyst also showed good TON and TOF values (Fig. 5).

To test the effectiveness of this protocol, a gram-scale (10 mmol scale) experiment was conducted for the reaction of anthranilamide with 4-nitrobenzaldehyde

Table 3 Exploration of the substrate scope of the developed synthetic method^{a,b}

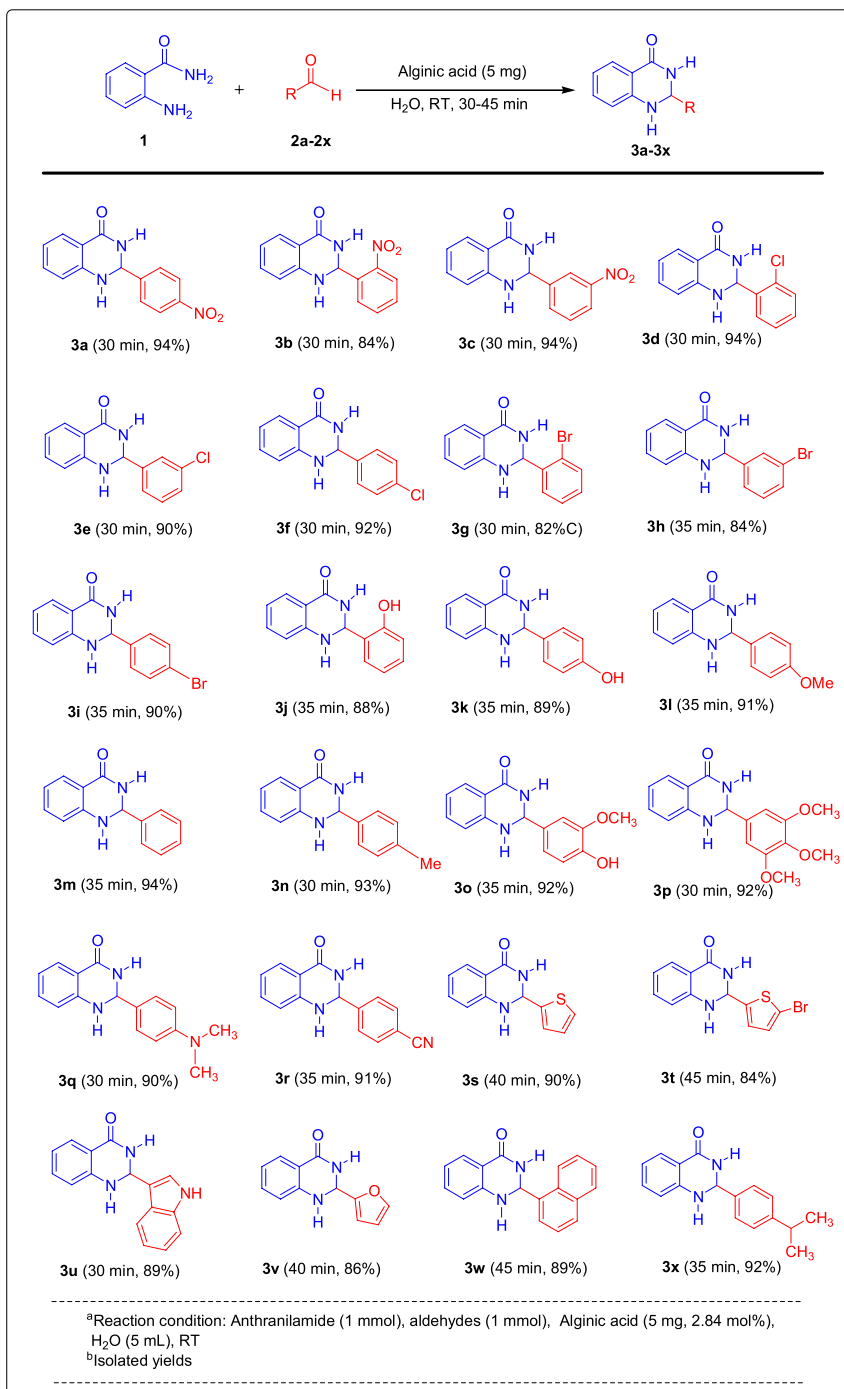
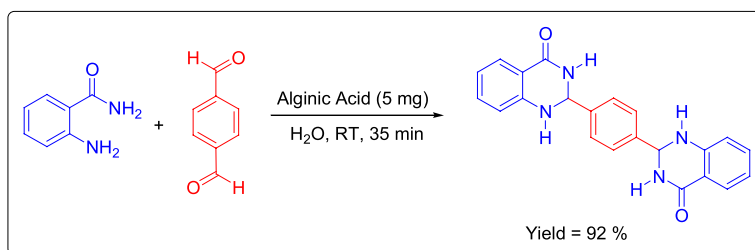
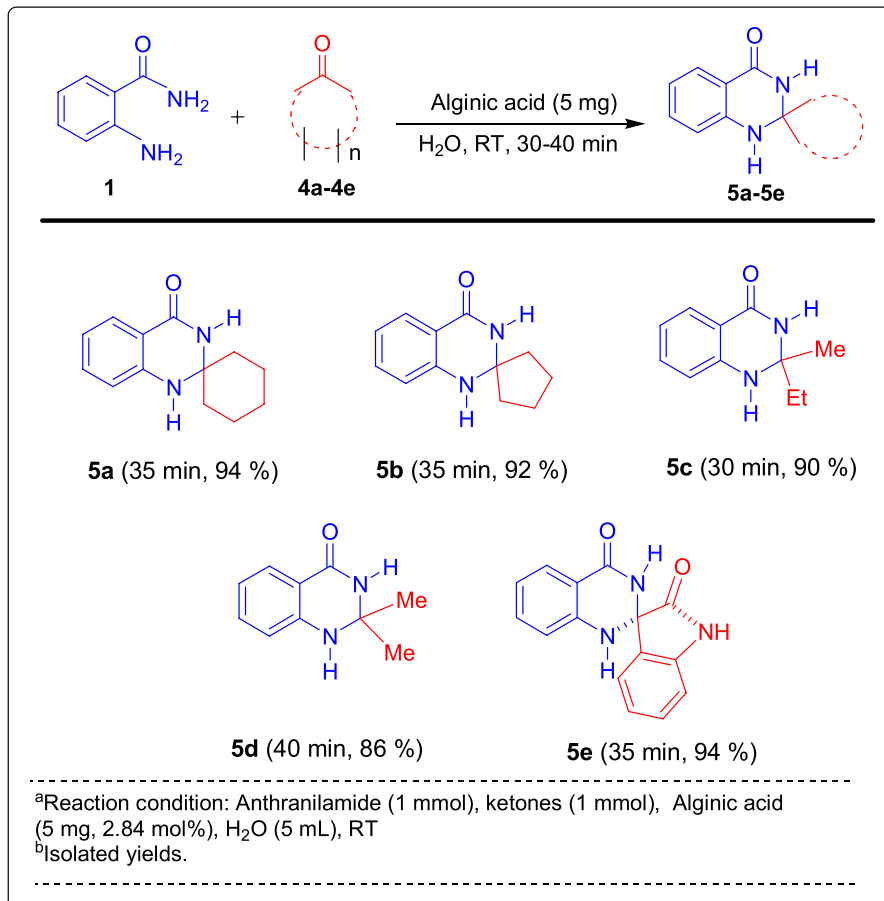
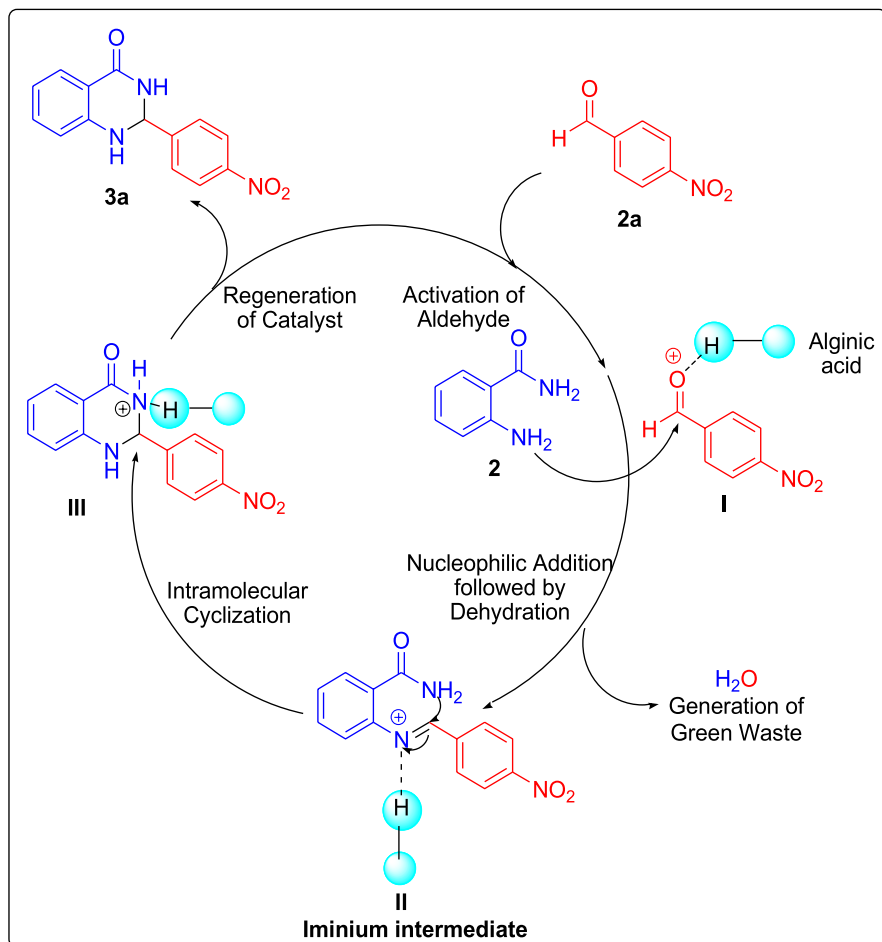


Table 4 Exploration of the substrate scope of the developed synthetic method by using various ketones^{a,b}**Scheme 3.** Synthesis of bis-quinazolinone

or cyclohexanone under optimized reaction conditions (Scheme 5). The large-scale process delivered the target products (**3a**), with 92% and (**5a**) 91% yields within 65 and 70 min, respectively. It was observed that the gram-scale synthesis



Scheme 4. A plausible mechanism for the synthesis of DHQ catalyzed by alginic acid

is almost similar to mmol scale entry in terms of yield but requires more time to complete. The TON and TOF were also high for the gram scale.

To avoid the consumption of solvents during the isolation process, different methods (Method I and Method II) were attempted and their green metric values were calculated with a catalyst (Fig. 6). Method I involved the extraction of products with $CHCl_3$ (2×5 mL), while Method II involved the treatment of the reaction mixture with NaOH (5 mL, 0.1 N NaOH), followed by filtration. The use of aqueous NaOH treatment dramatically reduced the green metrics.

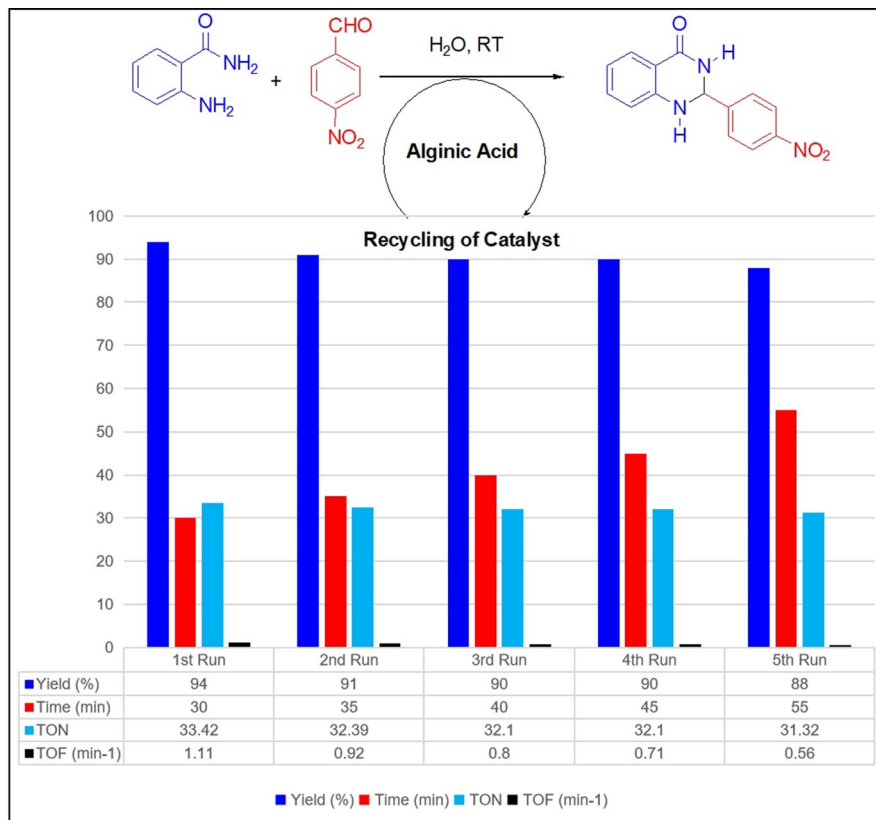
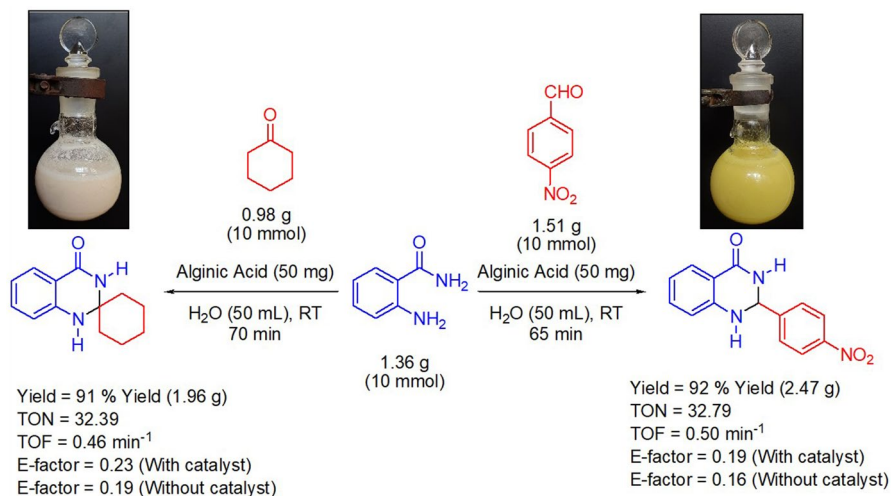
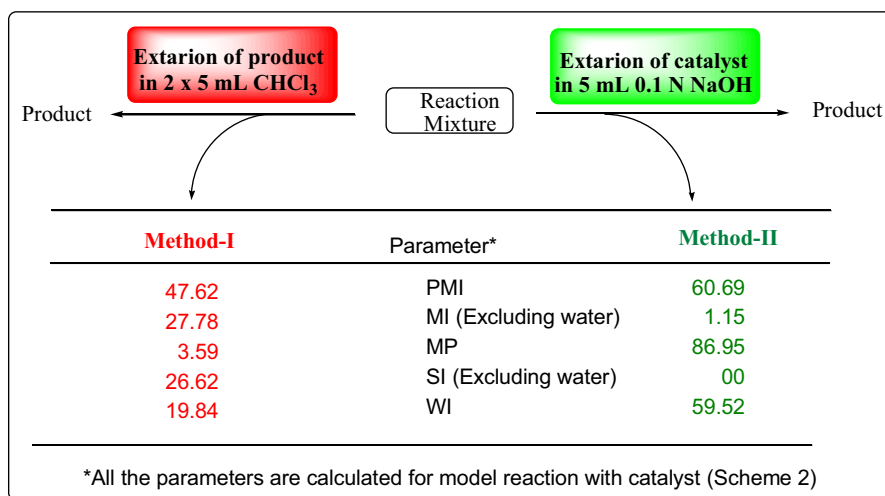


Fig. 5 Recyclability of the alginic acids for the synthesis of DHQ

General reflections on the evaluation of green metrics (estimation of greenness of present method)

Various sets of metrics were analyzed to estimate the "greenness" of syntheses [71, 72]. Green metrics measure the efficiency and environmental concert of chemical processes and can be better measured by environmental factor (E factor), turnover number (TON), turnover frequency (TOF), atom economy (AE), atom efficiency (AEf), carbon efficiency (CE), optimum efficiency (OE), effective mass yield (EMY), reaction mass efficiency (RME), mass productivity (MP), mass intensity (MI), and process mass intensity (PMI), as well as solvent and water intensity (SI and WI). These green chemistry metrics were measured for all reactions and depicted in Table S5.

The representative results of the model reaction under optimized reaction conditions are depicted in Fig. 7. The matrices were calculated by considering both the with and without catalyst. The results demonstrated that the values of green chemistry metrics are nearly close to their ideal values.


Scheme 5. Gram scale synthesis of products **3a** and **5a**

Fig. 6 The green metrics analysis for the isolation of DHQ

- The green credentials such as safety, health, and environmental scores, and the overall ranking of solvents assessed in the present protocol by using the CHEM21 solvent selection guide [73] and COSMO-RS [74]. Thus, we used water as a solvent for the reaction, which is a highly recommended solvent for chemical processes (Table S6). Though we used EtOH for the recrystallization of crude products, its use is recommended. This was also supported by solvent intensity (SI=0) and water intensity (WI) parameters of the present protocol, which are found to be 52.08 – 81.52 g/g.

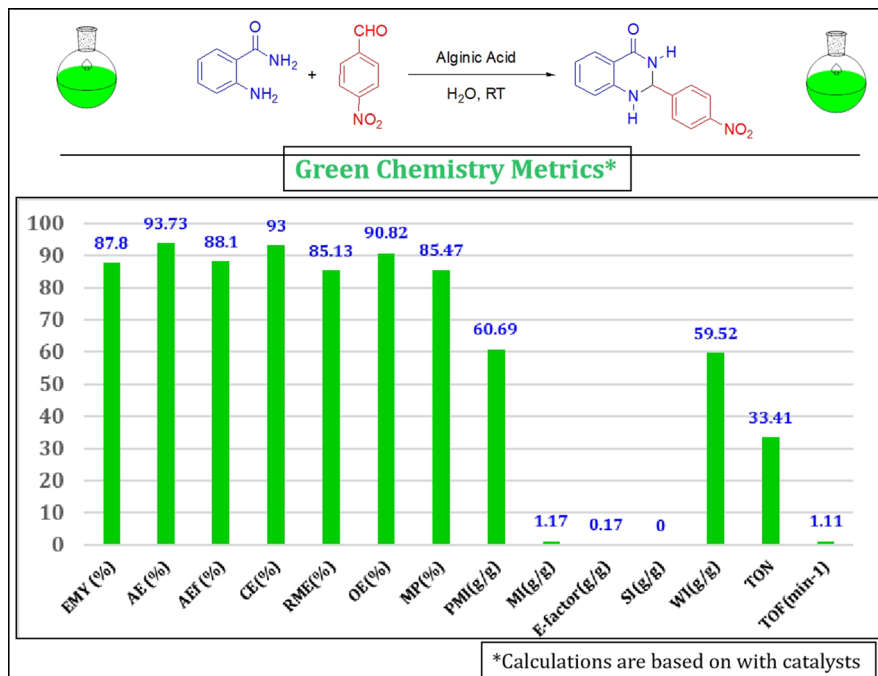


Fig. 7 The green metrics analysis for the synthesis of DHQ

- In current years the efficiency of the catalytic process has been evaluated by turnover number (TON) and turnover frequency (TOF) values [75]. The present catalyst attained TON = 29.17–33.46 and TOF = 0.66–1.11 min⁻¹.
- Effective mass yield (EMY) measures the environmental acceptability of a process. This metric defines yield in terms of the product made from non-toxic materials [76]. In the present method, EMY is 76.94–87.80% indicating the considerable greenness of the method.
- The DHQ can be synthesized straightforwardly in high yields. There is only the elimination of water as green waste. As all the products are obtained with good to excellent yields, the atom efficiency (AEf) for the present method is also excellent at 92.19–94.57% atom economy (AE) and 77.10–88.10% AEf.
- Carbon Efficiency (CE) and Mass productivity (MP) are used to elucidate reaction process consumption. The CE and MP for the present work are 81–100% and 75.18–85.47%, much better than the reported catalysts.
- Reaction mass efficiency (RME) is a realistic metric for describing the greenness of a process [77]. It accounts for yield, stoichiometry, and atom economy. In the present protocol, up to 85.13% RME is attained for 4-nitrobenzaldehyde, while 74.84% for 2-bromobenzaldehyde.
- Process mass intensity (PMI) is the total mass of materials used to produce a specified product. This is the key, high-level matrix for evaluating and benchmarking progress toward more sustainable manufacturing [71]. PMI evaluation (53.26–82.83 g/g) supports the above fact.

- Mass intensity (MI) accounts for the yield, stoichiometry, solvent, and reagent used in the reaction. The present method has MI ranges from 1.17 to 1.33 (g/g), which is near the ideal value of MI (MI = 1 g/g).
- Environmental Factor (E-factor) is a simple and fast metric for the evaluation of the environmental impact of industrial processes. It accounts for chemical yield and includes reagents; solvent loss and all process aids. The lower the E-factor, the lesser the waste generated in the process. The E-factor of the present method is between 0.17 and 0.33 (g/g) indicating the considerable greenness of the present protocol (the ideal E-factor is 0).
- In chemical reactions, a large amount of solvent used can be accounted for and assessed for the green matrix in terms of solvent intensity (SI) because of the significant environmental impact of solvents [78, 79]. Because SI excludes water from the calculations [71], the water intensity (WI) of a process should be calculated separately [80].

Conclusion

To summarize, a natural and eco-friendly process for creating 2,3-dihydroquinazolin-4(1H)-one analog has been developed using alginic acid as a catalyst. This method is advantageous because it has a low environmental impact and operates under mild conditions. The reaction occurs at room temperature through a simple stirring process, resulting in high yields. Additionally, this approach has a wide range of applications, can be easily scaled up, and allows for hassle-free product recovery. Its sustainability credentials have also been verified through green metrics evaluation. Overall, this alginic acid-catalyzed DHQ synthesis represents a promising greener alternative.

Supplementary Information The online version contains supplementary material available at <https://doi.org/10.1007/s11164-023-05147-8>.

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Author's contributions The author confirms sole responsibility for the following: Principal author collected the data, carried out experimental work, drafted the manuscript preparation, and designed the schemes and figures. Second author: All second authors partly helped to characterize and calculate green matrices. Corresponding author designed the manuscript, formatted figures and schemes, and contributed to analysis and interpretation of data.

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Availability of data and materials The data will be available upon reasonable request.

Declarations

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical approval There are no human subjects in this research article, and informed consent is not applicable.

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