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Highly efficient heterogeneous V_2O_5@TiO_2 catalyzed the rapid transformation of boronic acids to phenols

Rahul Upadhyay,[a, b] Deepak Singh,[a] and Sushil K. Maurya*[a, b]

A V_2O_5@TiO_2 catalyzed green and efficient protocol for the hydroxylation of boronic acid into phenol has been developed utilizing environmentally benign oxidant hydrogen peroxide. A wide range of electron-donating and the electron-withdrawing group-containing (hetero)aryl boronic acids were transformed into their corresponding phenol. The methodology was also applied successfully to transform various natural and bioactive molecules like tocopherol, amino acids, cinchonidine, vasicinone, menthol, and pharmaceuticals such as ciprofloxacin, ibuprofen, and paracetamol. The other feature of the methodology includes gram-scale synthetic applicability, recyclability, and short reaction time.

Phenols symbolize a dynamic class of organic compounds present in various natural products,[1] pharmaceuticals,[2] and biopolymers.[3] The phenolic compounds are well known for their antioxidant, anticancer, and antimicrobial activity.[3,4] Due to their vast existence and enormous utility in the living system, cost-effective and greener methods for synthesizing phenolic compounds are highly desired. Generally, the phenols were synthesized by the nucleophilic substitution reactions over aryl halides, diazo compound, the potassium salt of aryl trifluoro borate, and boronic acids. Amongst these, the *ipso*-hydroxylation of boronic acid emerges as the most conventional route for installing the hydroxyl group. In this regard, several methods were reported for the synthesis of phenols which utilized metal catalysts (Pd,[5] Ru,[6] Au,[7] Ag,[8] In,[9] Zn,[10] Cu[11]), photocatalysts (Ru,[12] Ir,[13] Zn,[14] COF,[15] QDs[16] POF[17]), electrochemical[18] and in metal-free conditions (H_2O_2,[19] TBHP,[20] *m*-CPBA,[21] oxone,[22] *N*-oxide,[23] peroxodisulfate[24]) (Figure 1). Previously, Olah and co-workers reported hydroxylation of boronic acid using hydrogen peroxide and water systems. Still, the methodology took a longer reaction time, and a low yield of the desired product was obtained.[19] Sequentially, further advancement in the catalytic system has been progressed, and a wide array of the non-metallic catalytic system has been developed, which utilized WERSA-H_2O_2,[25] ascorbic acid-H_2O_2,[26] TBHP-KOH,[20] and *N*-oxide.[23] Recently, He group reported microwave-assisted aerobic hydroxylation of boronic acid using KOH and DMSO.[27] Several other transition metal-catalyzed reactions were also reported, such as palladium-chitosan-CNT core-shell nanohybrid-H_2O_2 system,[5] Ru@imine-nanoSiO_2,[6] *N*-doped-C-encapsulated ultrafine In_2O_3 nanoparticles,[9] and K-10 supported silver nanoparticles-H_2O_2 system.[8] Apart from this, photoredox catalytic systems were also explored for the synthesis of phenols using $Ru(bpy)_3$,[12] $[Ir(OMe)(COD)]_2$,[13] and other organic photocatalysts (COF, QDs, POF). However, most of these photocatalysts utilized noble transition metal and a tedious preparation process. These methodologies also have several associated shortcomings, such as harsh reaction conditions, prolonged reaction time, a stoichiometric amount of the base, environmentally hazardous solvents, and limited functional group tolerance. In recent times, the utilization of heterogeneous metal catalysts gains considerable attention due to their advantages over homogeneous catalysts, as they can be recycled and reused and avoided metal contamination. Moreover, heterogeneous catalysis also enables easy large-scale production, high selectivity, and high thermal stability. Therefore, to overcome these shortcomings associated with the previous methodology and advantages associated with heterogeneous catalysts, we previously used heterogenous V_2O_5@TiO_2 to oxidize various scaffolds.[28] Here, we report the heterogeneous V_2O_5@TiO_2 catalyzed efficient and rapid hydroxylation of boronic acid to phenol containing a wide array of functionalities.

To obtain the optimal reaction condition for boronic acid hydroxylation into phenol, phenylboronic acid was chosen as a model substrate. The finding begins with the screening of active catalysts among five synthesized V_2O_5@TiO_2 catalysts (C-03, C-05, C-10, C-20, C-30)[28] containing different weight % of V_2O_5 using hydrogen peroxide and acetonitrile (Table 1). Initially, the 10 wt. % of each synthesized catalysts were evaluated, and among them, C-30, C-20, and C-10 provided the best results (Table 1, entries 1–5). We choose the C-10 catalyst for further investigation because it has low V_2O_5 content (Table 1, entry 3). A poor yield of the product was obtained when the reaction was performed in the presence of TiO_2 and V_2O_5 (Table 1, entries 6 and 7). Also, V_2O_5 provided a better yield than TiO_2, which confirmed the active participation of vanadium during the reaction. The obtained result also suggested that the vanadium efficiency enhanced on coordination with TiO_2, as they provided excellent yield in their combined form (V_2O_5@TiO_2). A reaction in the absence of a catalyst provided poor conversion into the product (Table 1, entry 8). Next, the catalyst loading was screened and found that the 5.0 wt. % catalyst gave a 99% yield of desired phenol within 5.0 minutes

[a] R. Upadhyay, D. Singh, Dr. S. K. Maurya
Chemical Technology Division,
CSIR-Institute of Himalayan Bioresource Technology Palampur,
Himachal Pradesh, 176 061, India
[b] R. Upadhyay, Dr. S. K. Maurya
Academy of Scientific and Innovative Research (AcSIR),
Ghaziabad 201 002, India
E-mail: skmaurya@ihbt.res.in

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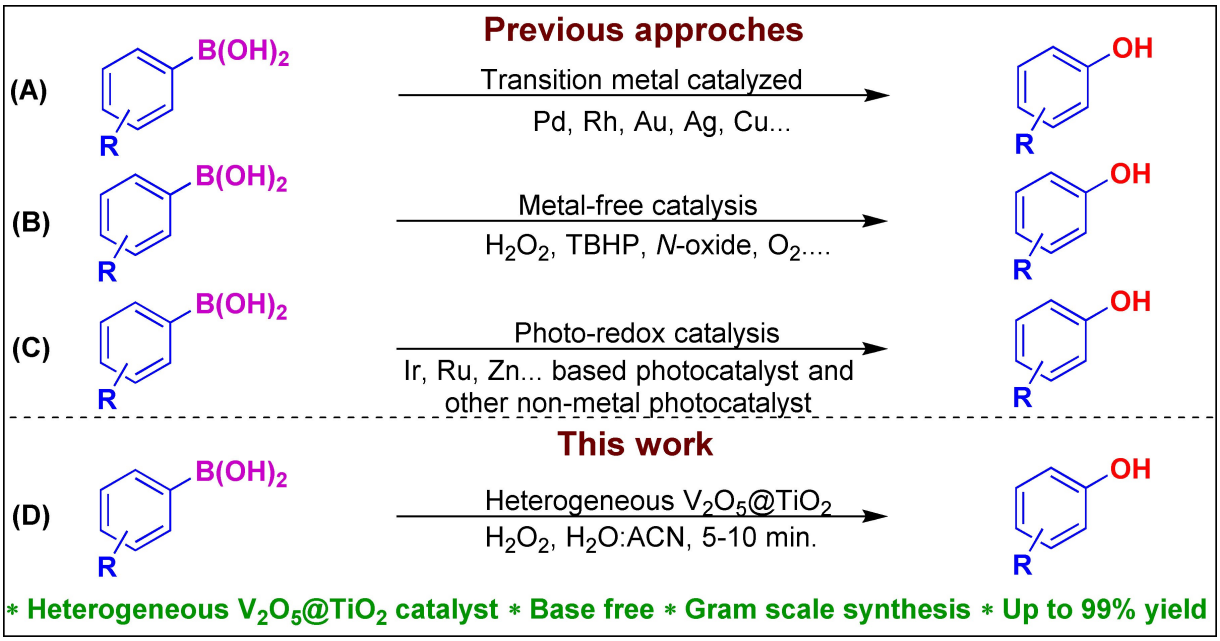
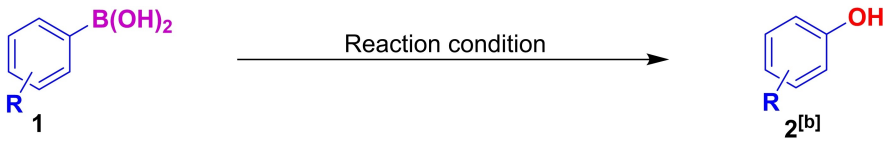


Figure 1. Methods for the hydroxylation of boronic acids into phenols.

Table 1. Reaction optimization.[a]



Entry	Catalyst	Oxidant [2.0 equiv.]	Solvent [1.0 mL]	Temperature	Yield[b]
1	C-30	H_2O_2	ACN	25 °C	99%
2	C-20	H_2O_2	ACN	25 °C	99%
3	C-10	H_2O_2	ACN	25 °C	99%
4	C-05	H_2O_2	ACN	25 °C	80%
5	C-03	H_2O_2	ACN	25 °C	40%
6[c]	TiO_2	H_2O_2	ACN	25 °C	38%
7[c]	V_2O_5	H_2O_2	ACN	25 °C	76%
8	–	H_2O_2	ACN	25 °C	36%
9[d]	C-10	H_2O_2	ACN	25 °C	99%
10[e]	C-10	H_2O_2	ACN	25 °C	99%
11[f]	C-10	H_2O_2	ACN	25 °C	78%
12	C-10	TBHP	ACN	25 °C	54%
13	C-10	$K_2S_2O_8$	ACN	25 °C	NR
14	C-10	H_2O	ACN	25 °C	NR
15	C-10	H_2O_2	DCM	25 °C	48%
16	C-10	H_2O_2	EtOAc	25 °C	62%
17	C-10	H_2O_2	H_2O	25 °C	82%
18[g]	C-10	H_2O_2	H_2O:ACN	25 °C	99%
19[h]	C-10	H_2O_2	H_2O:ACN	25 °C	99%
20[i]	C-10	H_2O_2	H_2O:ACN	25 °C	46%

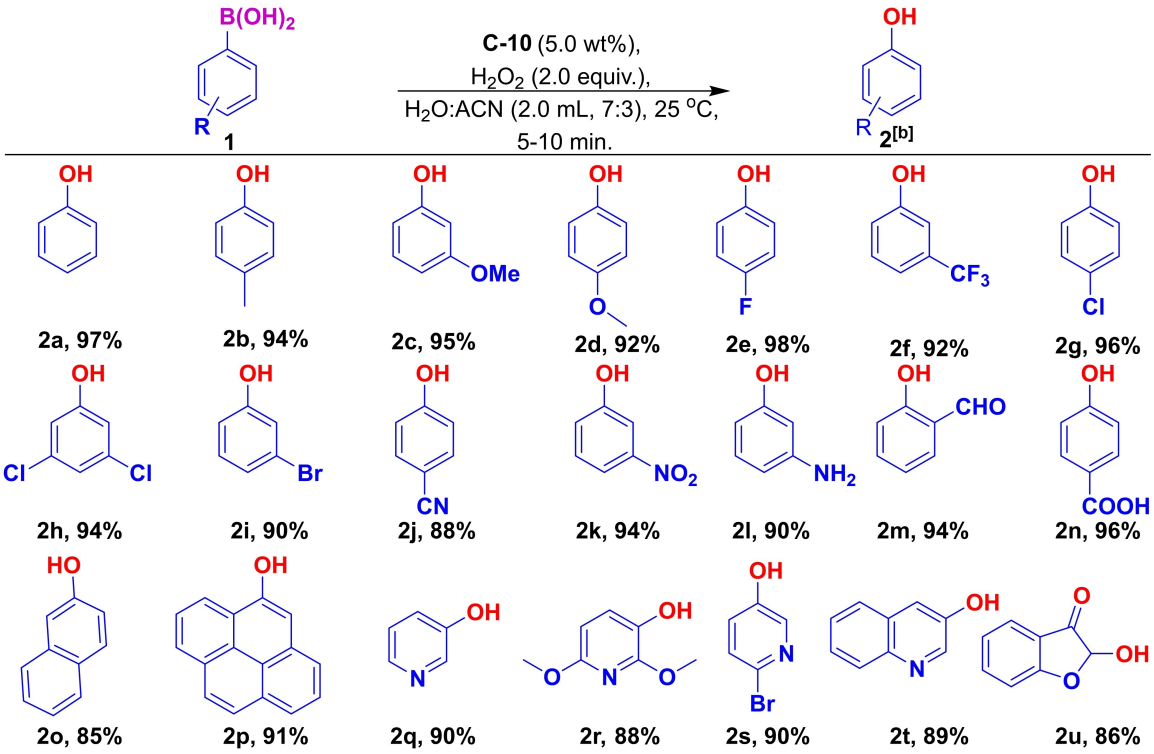
[a] Reaction conditions: **1** (50 mg, 1.0 equiv.), C-10 (10 wt%), H_2O_2 (2.0 equiv.), at 25 °C for 5.0 min. [b] Isolated yield. [c] 5.0 mol%. [d] 7.0 wt%. [e] 5.0 wt%. [f] 3.0 wt%. [g] H_2O:ACN (1:1). [h] H_2O:ACN (7:3). [i] H_2O_2 (1.0 equiv.).

(Table 1, entries 9–11). During the catalyst loading optimization, the C-10 catalyst was found better among the various catalyst tested (Table 1, entries 4, 5 and 10–11), even when used in the same mol % (see Supporting information, Table S1). Other oxidants effect was checked and found that no other oxidant provided phenol efficiently than hydrogen peroxide (Table 1, entries 12–14). Furthermore, to screen the solvent other than acetonitrile, the reaction was performed in dichloromethane, ethyl acetate, and water, the reaction in ethyl acetate and dichloromethane resulted in poor conversion, whereas reaction in water gave 82% yield of desired phenol (Table 1, entries 15–17). An increase in the yield was observed when the reaction

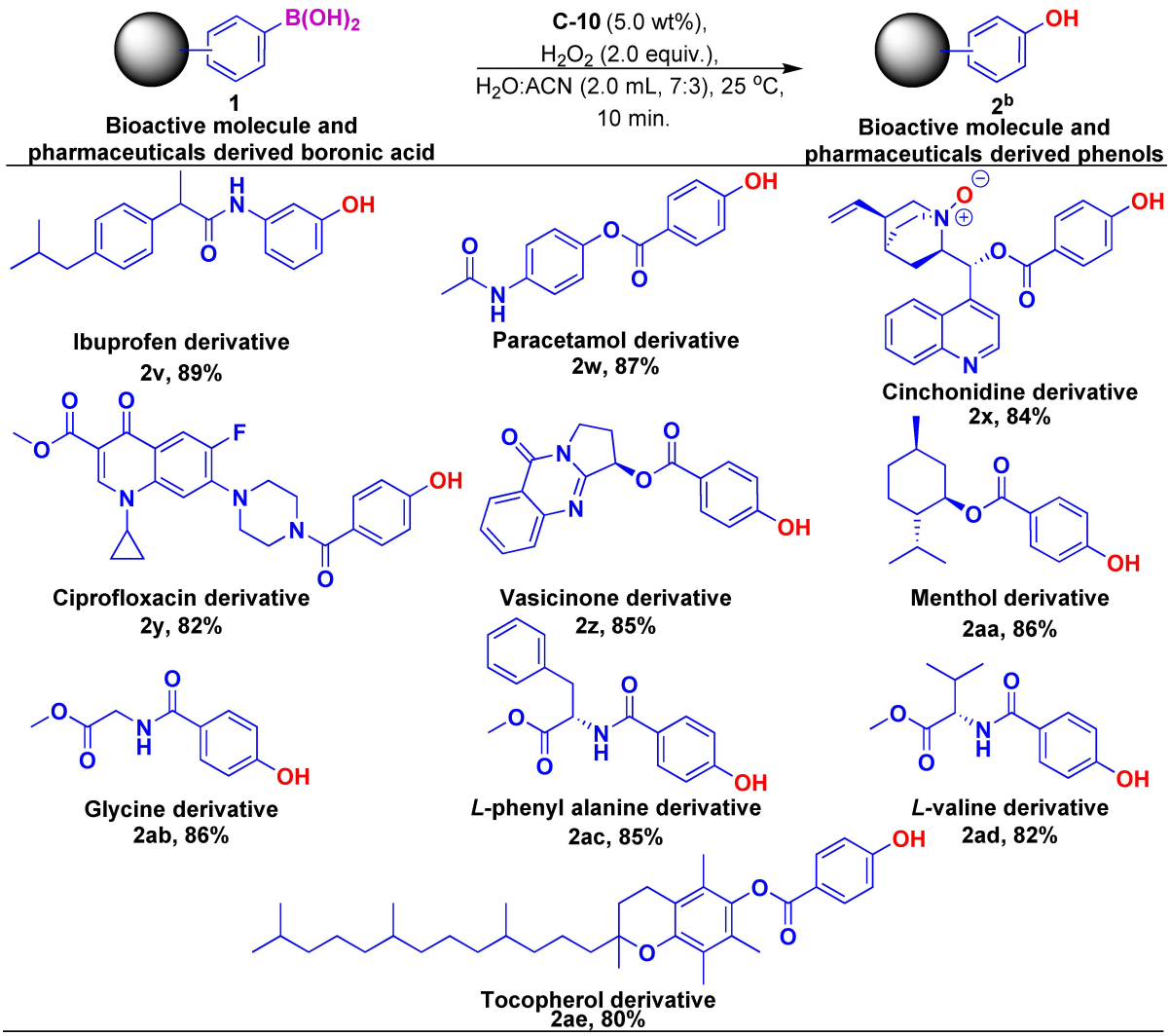
was performed in the mixture of water and acetonitrile (Table 1, entries 18 and 19). As the reaction in the water: acetonitrile mixture gave a similar result, the solvent system was selected for further evaluation based on the green chemistry aspects (Table 1, entries 10 and 19). On decreasing the hydrogen peroxide quantity, a decrease in the product's yield was observed (Table 1, entry 20). Further, based on TON/TOF data, C-10 is the best catalyst for this transformation (see Supporting information, Table S2).

With optimized conditions in hand, we investigate the substrate compatibility and versatility of the developed protocol. The methodology enables the hydroxylation of various boronic acid derivatives containing electron-donating and electron-withdrawing groups (Scheme 1, **2a**–**2p**). Initially, the methyl and methoxy substituted boronic acid were smoothly converted to desired phenols (Scheme 1, **2b**–**2d**). Halogenated phenols were synthesized under the developed conditions, and F, Cl, Br, and trifluoromethyl functional groups were well tolerated (Scheme 1, **2e**–**2i**). The boronic acid derivatives containing nitrile, nitro, and amino functionalities were successfully converted to their corresponding phenols in good to excellent yields (Scheme 1, **2j**–**2l**). Boronic acid bearing carbonyl group (aldehyde and carboxylic acid) were tolerated well and converted to corresponding phenols in excellent yields with high selectivity (Scheme 1, **2m** and **2n**). The boronic acid derivative of bicyclic and polycyclic arenes provided a good to an excellent yield of required phenols (Scheme 1, **2o** and **2p**). Heterocyclic boronic acid compounds containing pyridine and substituted pyridine skeleton were successfully transformed (Scheme 1, **2q**–**2s**). A smooth reaction was observed when the boronic acid derivative of quinoline was subjected to the optimized reaction conditions (Scheme 1, **2t**). An exciting reaction was observed when benzofuran-3-boronic acid was attempted to transform into the corresponding phenol. The process displayed a keto-enol tautomerization to form ketone with an additional hydroxyl group transfer adjacent to ethereal oxygen to give 2-hydroxybenzofuran-3(2*H*)-one (Scheme 1, **2u**).

After successfully implementing the methodology over a diverse range of boronic acid derivatives, the protocol's robustness was evaluated over structurally complex bioactive natural products and pharmaceuticals (Scheme 2). The investigation began with the boronic acid derivative of the widely used generic drugs ibuprofen and paracetamol. These pharmaceutical-derived boronic acids were successfully transformed into their respective phenols (Scheme 2, **2v** and **2w**). Interestingly, the boronic acid derivative of antimalarial agent (−) cinchonidine resulted in the formation of phenol along with the insertion of oxygen atom over aliphatic bridged nitrogen to furnish particular *N*-oxide product in good yield (Scheme 2, **2x**). The boronic acid derivative of the well-known antibacterial drug ciprofloxacin was successfully converted to the corresponding phenol (Scheme 2, **2y**). Vasicinone, a quinazoline alkaloid used to treat allergic and cardiac disease, effectively converted to their corresponding phenol (Scheme 2, **2z**). Menthol, a molecule capable of blocking voltage-sensitive sodium channels, reducing neural activity that may stimulate



Scheme 1. Scope of the synthesis of phenols. [a] Reaction conditions: 1 (100 mg, 1.0 equiv.), C-10 (5.0 wt%), H_2O_2 (2.0 equiv.), H_2O:ACN (2.0 mL, 7:3), at 25 °C for 5–10 min. [b] Isolated yield.

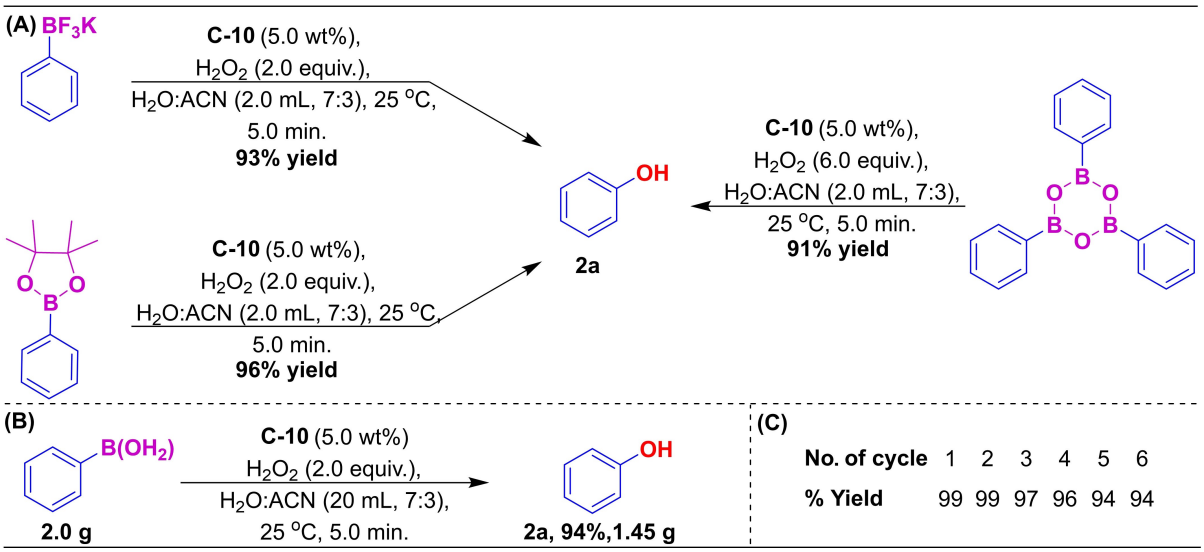


Scheme 2. Scope of phenol synthesis from natural products and pharmaceutical-derived boronic acids. [a] Reaction conditions: Boronic acid (50 mg, 1.0 equiv.), C-10 (5 wt%), H_2O_2 (2.0 equiv.), H_2O:ACN (2.0 mL, 7:3), at 25 °C for 10 min. [b] Isolated yields.

muscles, and increasing GABAergic transmission in PAG neurons, provided respective phenol utilizing their corresponding boronic acid derivative (Scheme 2, **2aa**). Amino acids are an integral part of our living system and play an important role in various biological processes. Boronic acid derivatives derived from the different amino acids such as glycine, *L*-phenyl alanine, and *L*-valine were tested for their compatibility with the developed protocol and resulted in corresponding phenols in good yield (Scheme 2, **2ab**–**2ad**). The boronic acid derivative of α-tocopherol, a form of vitamin E, efficiently yielded the desired product (Scheme 2, **2ae**).

Furthermore, phenol synthesis was successfully achieved in good to excellent yield utilizing other organoborane compounds rather than boronic acids. In this regard, the organoborane compounds such as potassium phenyltrifluoroborate, phenylboronic acid pinacol ester, 2,4,6-triphenylboroxin provided phenol under optimized reaction conditions (Scheme 3A). To demonstrate the synthetic applicability at the multi-gram scale, we performed the gram-scale synthesis of phenol and obtained excellent yield (Scheme 3B). The catalyst used for the transformation was recycled up to 6.0 cycles without any significant loss in catalytic activity. After each cycle, the catalyst was recycled by centrifugation or filtration, and then the residual catalyst was washed with acetone, dried in an oven for 2.0 h at 100 °C, and again used for the next cycle (Scheme 3C).[28] These obtained results further extended the robustness and versatility of the developed protocol.

The methodology was applied to synthesize the potential therapeutic candidate and the key intermediate for drug molecules to demonstrate the applicability of the developed protocol in medicinal chemistry. The synthesis of 3-hydroxy-2,5-dimethoxy pyridine (**2r**) was done in 88% yield, which is used to prepare an active antitubercular agent. The previous methodology utilized multiple reagents, required a long reaction time, and hazardous chemicals such as peracetic acid (Figure 2A).[29] The benzofuranone (**2u**), an essential antimicrobial agent used in various active pharmaceuticals, was synthesized rapidly from the boronic acid derivative of benzofurane in



Scheme 3. (A) Scope of the synthesis of phenol from potassium phenyltrifluoroborate, phenylboronic acid pinacol ester, and 2,4,6-triphenylboroxin (B) Gram-scale synthesis (C) Recyclability experiment.

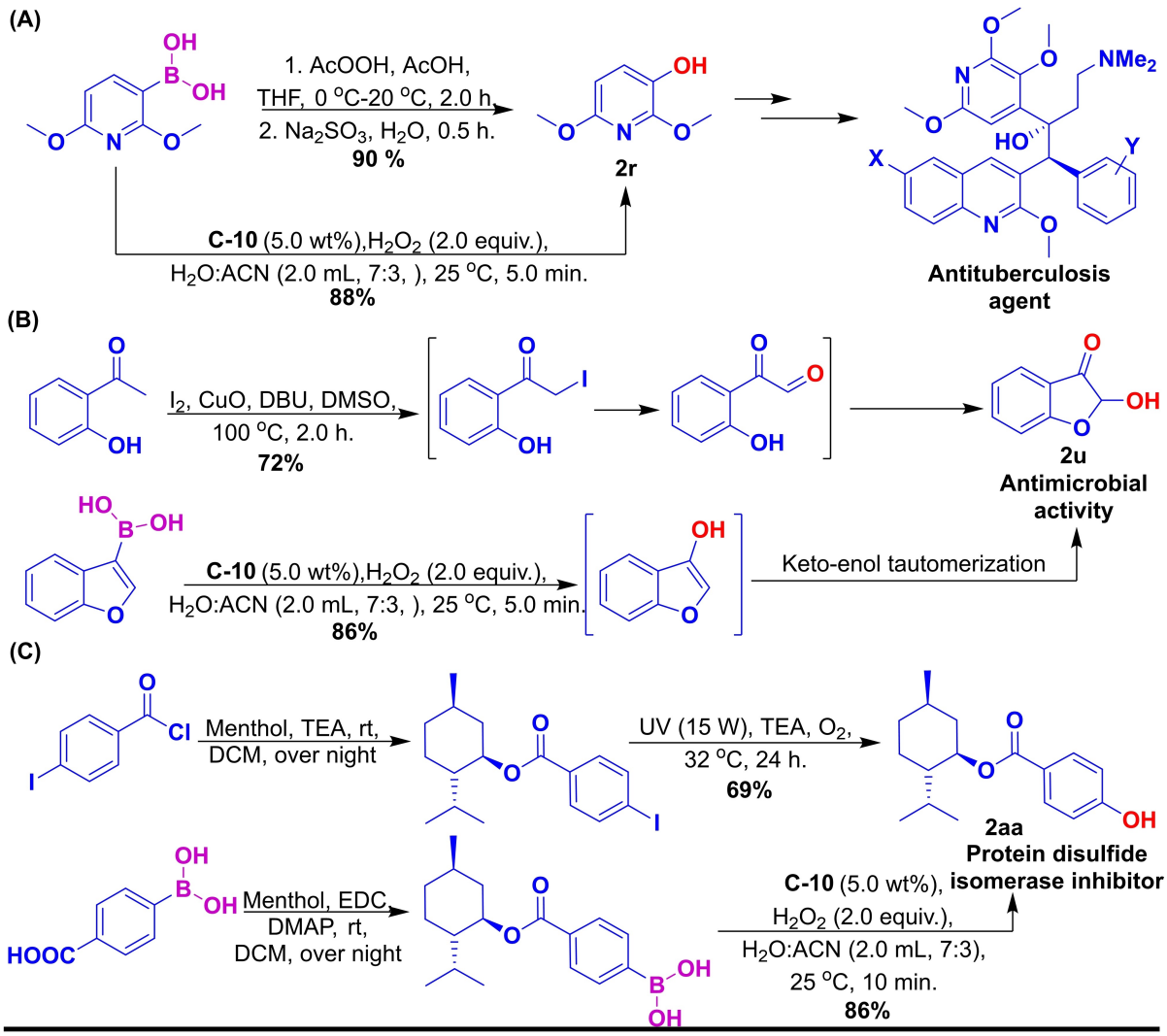


Figure 2. Application in medicinal chemistry and comparison studies.

Table 2. Comparison with the recent catalytic system for *ipso*-hydroxylation of boronic acids.

Entry	Catalyst	Oxidant	Oxidant load	Solvent	Time	Temp. [°C]	Yield [%]	Ref.
1	–	H_2O_2	1.0 eq.	H_2O	Several hours	RT	85	19
2	COF–P-3Ph/Visible light	O_2/TEA (additive)	–	ACN	4.0 h	RT	99	15
3	–	$(NH_4)_2S_2O_8$	2.5 eq.	MeOH:H_2O	3.0 h	80	98	24
4	–	Air	–	DMSO	5.0 min	100 MW (300 w)	98	27
5	Graphene oxide	H_2O_2	4.0 eq.	H_2O	5.0 min	RT	99	33
6	V_2O_5@TiO_2	H_2O_2	2.0 eq.	H_2O: ACN	5.0 min	RT	99	This work

86% yield. Earlier reported protocol for the synthesis of benzofuranone (**9u**) utilizes a stoichiometric amount of catalyst, required elevated temperature, and resulted in a relatively lower yield (Figure 2B).[30] The synthesis of a protein disulfide isomerase inhibitor derivative of menthol (**2aa**) was accomplished in 86% yield by using the developed protocol, whereas the previously developed protocol utilized UV light irradiation over 24 h in the presence of molecular O_2 and provided comparatively lower yield (69%) of the desired product (Figure 2C).[31,32]

We further calculated the green metrics parameter to determine the developed protocol efficiency and applicability over existing methodologies. These parameters for three medicinally important molecules (Figure 2A–C), provided the efficiency compared to existing methodology in terms of carbon (For **A**, 88 > 57; **B**, 86 > 57; **C**, 87 > 69), reaction mass (For **A**, 54 > 41; **B**, 56 > 50; **C**, 63 > 45), atom efficiency (For **A**, 62 > 53; **B**, 65 > 50; **C**, 70 > 45), and optimum efficiency (For **A**, 76 > 69; **B**, 73 > 71; **C**, 78 > 69). Similarly, the other green parameter such as atom economy, E-factor, mass productivity, productive mass intensity, solvent intensity, and water intensity strengthen the integrity of the developed reaction condition over the existing methodology (see Supporting information, Table S3). Additionally, the comparison of the recently developed methodology and our methodology were also listed to demonstrate the developed protocol's advantages (Table 2). As methodologies listed in Table 2, the previous hydroxylation of boronic acid into sole hydrogen peroxide gave 85% yield and required a longer reaction duration (Table 2, entry 1). The photocatalyzed methodology for the hydroxylation of boronic acid using COF–P-3Ph utilizing O_2 requires 4.0 hours (Table 2, entry 2). The elevated temperature treatment either by heating or microwave resulted in phenol (Table 2, entries 3 and 4). The graphene oxide catalyzed hydroxylation of boronic acid to phenol using hydrogen peroxide in water was developed (Table 2, entry 5). In comparison, our methodology enables hydroxylation of boronic acid into phenol in good to excellent yield utilizing heterogeneous V_2O_5@TiO_2 and hydrogen peroxide system in water: acetonitrile (7:3) solvent (Table 2, entry 6).

The previously reported mechanistic study of the vanadium oxide complexes in various oxidants demonstrates the vanadium metal ability to form the active species responsible for the catalytic cycles.[33] In this regard, to check the oxidant's modification after contacting the catalyst, the control experiments were performed (Figure 3). The interaction of peroxide

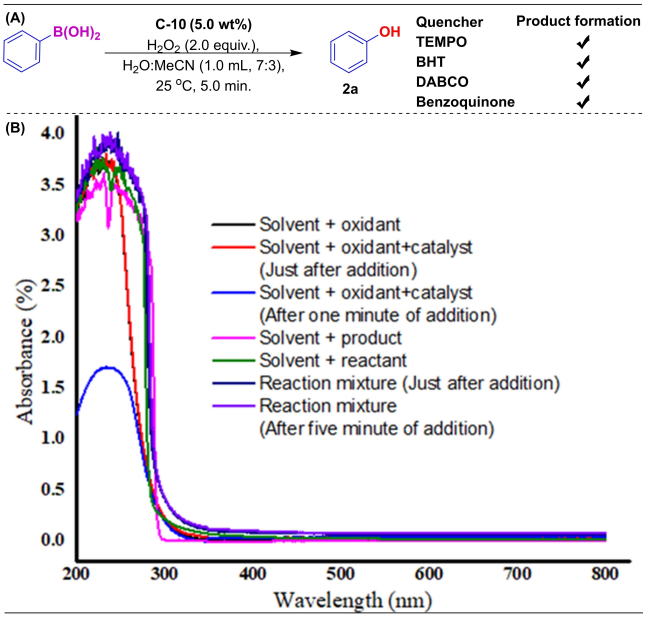


Figure 3. (A) Quenching experiments. (B) The UV-visible spectrum of different components used in the reaction.

with catalyst was studied over UV-visible spectrometry (Figure 3B); the data analysis of peroxide in acetonitrile with and without catalyst indicated the interaction between catalyst and peroxide. The concentration of the peroxide was diminished after 1.0 minutes of the addition of the catalyst. In the mechanistic investigation of boronic acid hydroxylation into phenol, it was observed that the radical scavenger does not affect the yield of the reaction, which vanished the radical pathway. A series of radical scavengers such as TEMPO, benzoquinone, BHT, and DABCO was used in this study and resulted in the respective phenol formation (Figure 3A). The study indicated that the reaction does not involve any possible hydrogen peroxide radicals, such as hydroxyl radical, peroxy hydroxyl radical, and superoxide radical.[34] A plausible mechanism for the reaction has been proposed based on the previously reported literature.[32] The transformation of boronic acid **II** to phenol **V** was accelerated rapidly by forming boronic acid adduct **III** with activated hydrogen peroxide complex **I**, which further undergoes rearrangement to give intermediate compound **IV** and final hydrolysis yielded phenol **V** (see Supporting information, Figure S1).[34]

This work represents the catalytic activity of heterogeneous V_2O_5@TiO_2 catalyst for the *ipso*-hydroxylation of a wide range of hetero(aryl) boronic acids utilizing hydrogen peroxide. A wide variety of functional groups containing compound and bio-active molecules were transformed into corresponding products in good to excellent yield with high selectivity. Recyclability and gram-scale synthetic utility are the other alluring features of the developed protocol.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: Boronic acid · Heterogeneous catalysis · Hydroxylation · Pharmaceuticals · Phenol

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