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LEMON JUICE: GREENER APPROACH FOR ONE POT, THREE COMPONENTS SYNTHESIS OF BENZTHIAZOLE-BASED BETTI BASES

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ABSTRACT: A highly efficient eco-friendly route for three-component one-pot synthesis of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol derivatives using lemon juice as a catalyst has been developed. This approach manifests several green chemistry features like use of a biocatalyst, operational simplicity, a short reaction time, remarkable yields of the desired product, and an eco-friendly medium. The current approach could be a beneficial green alternative for the synthesis of targeted molecules as an alternative to existing methods.

KEYWORDS: Green synthesis, lemon juice, one pot, Betti base

INTRODUCTION:

In recent years, the notion of green chemistry has received considerable attention in order to develop synthetic techniques that are not detrimental to the environment by reducing pollution associated with chemical synthesis[i]. Multicomponent reactions have piqued interest as the most promising strategy for incorporating molecular diversity and generating complex molecules[ii]. They offer distinct advantages over conventional linear-step synthesis in terms of simplicity of a one-pot approach, ease of purification, short reaction times, mild reaction conditions, atom-efficient and high-throughput synthesis of organic molecules, and thereby saving energy and raw material consumption. As a result, multicomponent reactions benefit both the economy and the environment and are utilised in the synthesis of pharmacologically essential compounds[iii].

Betti reaction is a three-component, one-pot reaction that involves the condensation of 2-naphthol, aldehyde, and amine to give Betti bases, also known as 1-(-aminoalkyl)-2-naphthols. Betti bases are important scaffolds because they possess various biological activities like antimicrobial[iv], antioxidant[v], antibacterial[vi], anti-Alzhiemer[vii]. Benzthiazole core scaffold is significant for drug development since it has showed a broad range of therapeutic potential[viii]. According to prevailing research, this class of chemicals has anti-Alzheimer's[viii], antimicrobial[ix], antiparasitic[x], anticancer[xi] activities. The Betti bases bearing 2-aminobenzthiazole moiety were reported to have a wide range of biological activities, such as antimicrobial[xii], antiviral[xiii] activities.

Catalysis has shaped a self-evident transformation in the realm of synthetic chemistry. Owing to the advantages of catalysis enticed chemists to explore new catalysts. Natural catalysts are a distinct class of biocatalysts that are environmentally friendly, inexpensive, non-hazardous and have a wide range of applications in organic transformations[xiv]. Lemon is species of the citrus family. The presence of citric and ascorbic acids (vitamin C) in lemon juice causes acidity, which acts as an acid catalyst in organic transformation[xv]. The aqueous extract of lemon juices has gained sustained attention from synthetic organic chemists as a biocatalyst in various organic reactions. Aside from their harmless and eco-friendly behaviour, these fruit juices are also safe, cheap, and easily accessible[xvi].

There are several reported methods for synthesizing 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol with a variety of catalysts. However, current methods have drawbacks such as time-consuming catalyst preparation, prolonged reaction times, expensive reagents, the use of toxic solvents, lower yields, and complicated workup procedures. As a result, it is highly desirable to synthesise 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol derivatives using natural catalysts and eco-friendly solvents under mild reaction conditions. Glycerol was chosen as the reaction medium for condensation of 2-naphthol, 2-aminobenzthiazole with a variety of aryl aldehydes using natural catalyst because it is significantly safer, eco-friendly, non-toxic, and less expensive than other organic solvents[xvii].

In furtherance to develop sustainable methodologies for the synthesis of bio-active heterocyclic scaffolds, the synthesis of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol is presented here using lemon juice as a natural, bio-degradable catalyst under mild reaction conditions.

EXPERIMENTAL

Materials

All the chemicals were purchased from Sigma Aldrich (India) and were used without further purification. Reactions were monitored by using Merck silica gel 60 F254 plates. Melting points were measured in open capillary tubes and are uncorrected. FTIR was recorded on Perkin Elmer, Frontier equipment with ATR. ^{1}H NMR (300 MHz) was recorded on Bruker AVANCE II using TMS as internal standard in DMSO-d6.

General procedure for the synthesis of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol (4a-i)

In a 25-ml-round bottom flask, a mixture of 2-naphthol (1 mmol), substituted aldehyde (1 mmol), 2-aminobenzthiazole (1 mmol) and lemon juice (2 ml) in 5 ml glycerol, was stirred at 80 °C for the appropriate time and monitored by TLC. After completion of the reaction (after 5 h), the solid obtained was filtered. Further, it was recrystallized using ethanol to afford desire product in high purity.

1-[(Benzothiazol-2-ylamino)-phenyl-methyl]-naphthalen-2-ol (4a)

Yield 96% (White solid); M.P.: 201-203˚C. IR (υ_{max}/cm^{-1}): 3379 (N-H), 746 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.19 (s, 1H, O<u>H</u>), 8.84 (d, *J* = 7.4 Hz, 1H, N<u>H</u>), 7.88 (d, *J* = 6.6 Hz, 1H, Ar-<u>H</u>), 7.80 (dd, *J* = 8.2, 4.4 Hz, 2H, Ar-<u>H</u>), 7.68 (d, *J* = 7.7 Hz, 1H, Ar-<u>H</u>), 7.37 (t, *J* = 8.7 Hz, 3H, Ar-<u>H</u>), 7.30 – 7.23 (m, 6H, Ar-<u>H</u>), 7.19 (dd, *J* = 8.1, 4.3 Hz, 2H, Ar-<u>H</u>), 7.02 (t, *J* = 7.4 Hz, 1H, C<u>H</u>).

1-[(Benzothiazol-2-ylamino)-p-tolyl-methyl]-naphthalen-2-ol (4b)

Yield 97% (White solid); M.P.: 184-186˚C. IR (υ_{max}/cm^{-1}): 3371 (N-H), 750 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.17 (s, 1H, O<u>H</u>), 8.79 (d, *J* = 7.5 Hz, 1H, N<u>H</u>), 7.87 (d, *J* = 6.8 Hz,

1H, Ar-H), 7.83 – 7.75 (m, 2H, Ar-H), 7.67 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.35 (t, *J* = 9.0 Hz, 2H, Ar-H), 7.30 – 7.11 (m, 6H, Ar-H), 7.09 – 6.97 (m, 3H, CH, Ar-H), 2.23 (s, 3H, CH_3).

1-[(Benzothiazol-2-ylamino)-(2-methoxy-phenyl)-methyl]-naphthalen-2-ol (4c)

Yield 95% (White solid); M.P.: 185-187˚C. IR (υ_{max}/cm^{-1}): 3346 (N-H), 748 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 9.93 (s, 1H, OH), 8.63 (d, *J* = 7.8 Hz, 1H, NH), 8.22 (d, *J* = 8.7 Hz, 1H, Ar-H), 7.74 (dd, *J* = 16.1, 8.3 Hz, 2H, Ar -H), 7.62 (d, *J* = 7.3 Hz, 1H, Ar -H), 7.51 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.42 (t, *J* = 7.5 Hz, 1H, Ar- H), 7.36 – 7.12 (m, 6H, Ar -H), 6.96- 6.85 (m, *J* = 22.5, 11.2, 4.1 Hz, 3H, CH, Ar-H), 3.58 (s, 3H, OCH_3).

1-[(Benzothiazol-2-ylamino)-(4-chloro-phenyl)-methyl]-naphthalen-2-ol (4d)

Yield 97% (White solid); M.P.: 208-210˚C. IR (υ_{max}/cm^{-1}): 3373 (N-H), 750 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.22 (s, 1H, OH), 8.84 (d, *J* = 6.0 Hz, 1H, NH), 7.81 (d, *J* = 5.7 Hz, 3H, Ar-H), 7.68 (d, *J* = 7.4 Hz, 1H, Ar-H), 7.40 (d, *J* = 7.5 Hz, 2H, Ar-H), 7.28 (dt, *J* = 17.4, 9.1 Hz, 8H, Ar-H), 7.02 (t, *J* = 7.2 Hz, 1H, CH).

1-[(Benzothiazol-2-ylamino)-(3-fluoro-phenyl)-methyl]-naphthalen-2-ol (4e)

Yield 95% (White solid); M.P.: 180-182˚C. IR (υ_{max}/cm^{-1}): 3387 (N-H), 746 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.24 (s, 1H, OH), 8.86 (d, *J* = 7.4 Hz, 1H, NH), 7.96 – 7.75 (m, 3H, Ar-H), 7.69 (d, *J* = 7.2 Hz, 1H, Ar-H), 7.45 – 7.17 (m, 7H, Ar-H), 7.10 – 6.96 (m, 4H, CH, Ar-H).

1-[(Benzothiazol-2-ylamino)-(4-trifluoromethyl-phenyl)-methyl]-naphthalen-2-ol (4f)

Yield 96% (White solid); M.P.: 197-199˚C. IR (υ_{max}/cm^{-1}): 3375 (N-H), 748 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.27 (s, 1H, OH), 8.91 (d, *J* = 6.8 Hz, 1H, NH), 7.83 (d, *J* = 8.5 Hz, 3H, Ar-H), 7.68 (dd, *J* = 12.1, 8.2 Hz, 3H, Ar-H), 7.44 (dd, *J* = 19.1, 7.7 Hz, 5H, Ar-H), 7.32 – 7.19 (m, 3H, Ar-H), 7.04 (t, *J* = 7.4 Hz, 1H, CH).

4-[(Benzothiazol-2-ylamino)-(2-hydroxy-naphthalen-1-yl)-methyl]-benzonitrile (4g)

Yield 94% (White solid); M.P.: 202-204˚C. IR (υ_{max}/cm^{-1}): 3377 (N-H), 2227 (CN), 1579 (C=N), 750 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.29 (s, 1H, OH), 8.90 (d, *J* = 7.0 Hz, 1H, NH), 7.83 (d, *J* = 8.7 Hz, 3H, Ar-H), 7.72 (dd, *J* = 15.0, 8.0 Hz, 3H, Ar-H), 7.41 (dd, *J* = 14.2, 7.1 Hz, 5H, Ar-H), 7.31 – 7.19 (m, 3H, Ar-H), 7.04 (t, *J* = 7.3 Hz, 1H, CH).

1-[(Benzothiazol-2-ylamino)-(3-nitro-phenyl)-methyl]-naphthalen-2-ol (4h)

Yield 96% (White solid); M.P.: 201-203˚C. IR (υ_{max}/cm^{-1}): 3385 (N-H), 1532 (NO_2), 742 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.31 (s, 1H, OH), 8.98 (d, *J* = 7.1 Hz, 1H, NH), 8.13 (s, 1H, Ar-H), 8.07 (d, *J* = 8.0 Hz, 1H, Ar-H), 7.86 (t, *J* = 10.9 Hz, 3H, Ar-H), 7.75 – 7.64 (m, 2H, Ar-H), 7.57 (t, *J* = 7.9 Hz, 1H, Ar-H), 7.48 – 7.36 (m, 3H, Ar-H), 7.34 – 7.17 (m, 3H, Ar-H), 7.08 – 6.99 (m, 1H, CH).

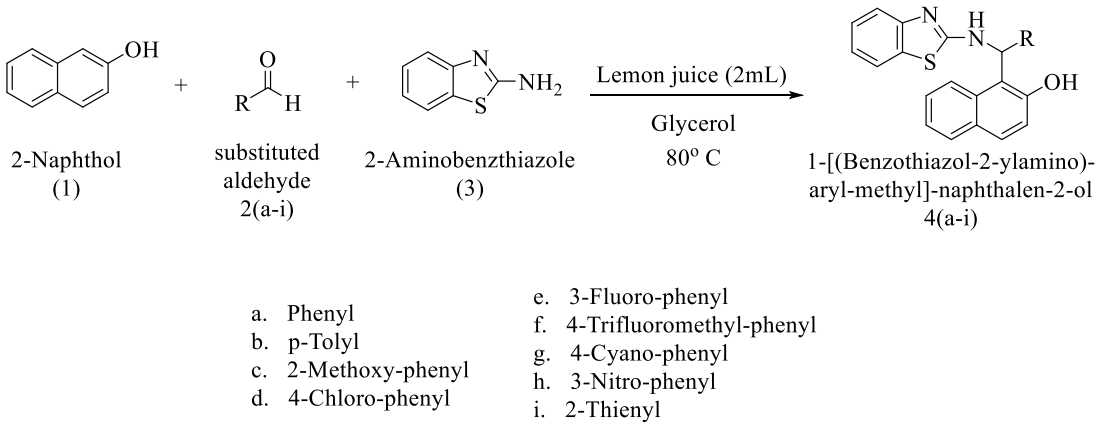
1-[(Benzothiazol-2-ylamino)-thiophen-2-yl-methyl]-naphthalen-2-ol (4i)

Yield 94% (White solid); M.P.: 187-189˚C. IR (υ_{max}/cm^{-1}): 3375 (N-H), 748 (C-S-C). ^{1}H NMR (300 MHz, DMSO) δ= 10.29 (s, 1H, OH), 8.97 (d, *J* = 7.2 Hz, 1H, NH), 8.02 (d, *J* = 7.7 Hz, 1H, Ar-H), 7.81 (dd, *J* = 8.2, 5.1 Hz, 2H, Ar-H), 7.68 (d, *J* = 7.5 Hz, 1H, Ar-H), 7.49 (d, *J* = 7.6 Hz, 1H, Ar-H), 7.41 (d, *J* = 7.8 Hz, 2H, Ar-H), 7.34 – 7.19 (m, 4H, Ar-H), 7.02 (t, *J* = 7.1 Hz, 1H, Ar-H), 6.91 (dd, *J* = 4.9, 3.6 Hz, 1H, Ar-H), 6.82 (d, *J* = 3.3 Hz, 1H, CH).

RESULTS AND DISCUSSION:

The general path for the synthesis of Betti bases with 2-aminobenzthiazole scaffold **4(a–i)** is depicted in Scheme 1. The compounds were synthesized by condensation of 2-naphthol (1), substituted aldehyde (2) and 2-aminobenzthiazole (3) at 80 °C using lemon juice as a catalyst. The synthesized compounds were characterized using IR, ^{1}H NMR and the characterization data of synthesized compounds **4(a–i)** are presented in experimental part. In IR spectrum, a band for NH-stretching was detected in the range of 3340–3390 cm^{-1}. In ^{1}H NMR spectrum, a peak around δ = 6–8 ppm corresponds to NH proton while a peak around δ = 9.9–10.3 ppm

denotes OH proton. A peak around δ = 6.8–7.4 ppm corresponds to methine proton (CH) which is attached next to Ar groups. The physical and spectral results were in agreement with those reported in the literature.



a. Phenyl
b. p-Tolyl
c. 2-Methoxy-phenyl
d. 4-Chloro-phenyl
e. 3-Fluoro-phenyl
f. 4-Trifluoromethyl-phenyl
g. 4-Cyano-phenyl
h. 3-Nitro-phenyl
i. 2-Thienyl

Scheme 1. Synthesis of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol using Lemon juice

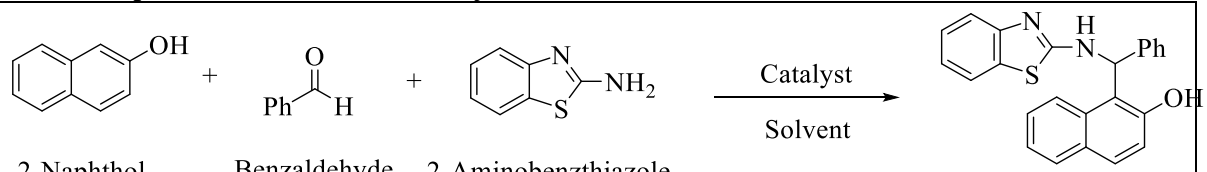
Optimization of the reaction condition

In order to find appropriate parameters for reaction, a model reaction of 2-naphthol (1 mmol), 2-aminobenzthiazole (1 mmol) and benzaldehyde (1 mmol), was chosen (Table 1). The amount of catalyst required for reaction, effect of solvent, effect of temperature and time of the model reaction were studied in order to optimize the reaction condition.

To begin, the necessary amount of lemon juice required to catalyse the reaction was determined. It was observed that 2ml (Table 1, entry 3) lemon juice was effective to carrying out the reaction.

Further investigation into the influence of solvent revealed that yields were lower in neat conditions (Table 1, entries 1, 2, 3, 4). The reaction was performed in water, with only a trace amount of product (Table 1, entry 5). In comparison, the yield of the product from reactions carried out in polar protic solvents like ethanol and methanol was minimal (Table1, entries 6 and 7) and when the reaction was performed in glycerol, good yields were achieved (Table 1, entry 8), demonstrating that glycerol is an appropriate reaction solvent. Further, varying the temperature for the model reaction. It was found that increasing the temperature from 25 to 80° C, the yield of the product increased till 80°C. As a result, the optimal temperature for the reaction was determined to be 80°C (Table 1, entry 8). Later the time for the reaction was evaluated, reaction carried out for 3 h gave lower yield (Table 1, entry 11), while carrying out reaction for 5 h gave good yield (Table 1, entry 8) and further increasing time for 7 h didn't gave significant increase in the yield (Table 1, entry 12). Hence, 5 h was considered optimal time for the reaction to complete.

Table 1. Optimization condition for synthesis of benzothiazole based Betti base



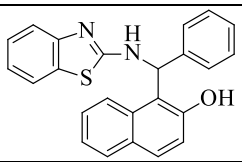
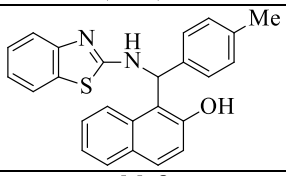
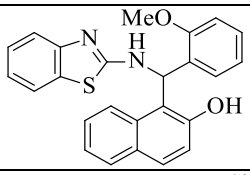
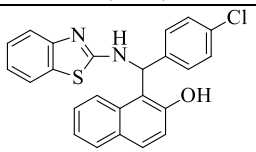
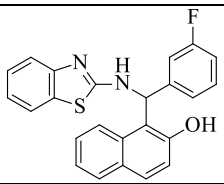
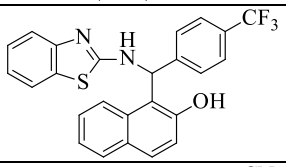
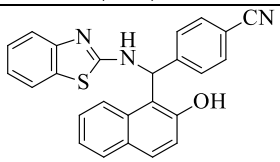
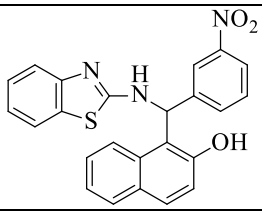
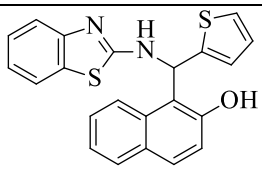
2-Naphthol (1) | Benzaldehyde (2a) | 2-Aminobenzthiazole (3) | (4a)

Entry	Amount of juice (mL)	Solvent	Temperature (ºC)	Time (h)	Yield (%)[a]
1	1.0	-	80	5	Trace
2	1.5	-	80	5	24
3	2.0	-	80	5	38
4	2.5	-	80	5	40
5	2.0	H_2O	80	5	Trace
6	2.0	Methanol	80	5	46
7	2.0	Ethanol	80	5	61
8	2.0	Glycerol	80	5	95
9	2.0	Glycerol	25	5	48
10	2.0	Glycerol	50	5	81
11	2.0	Glycerol	80	3	63
12	2.0	Glycerol	80	7	97

Reaction conditions: 2-naphthol (1 mmol), benzaldehyde (1 mmol), 2-aminobenzthiazole (1 mmol), catalyst (2 mL) and solvent (5 ml)
[a]Isolated yields

A series of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol were synthesised using various aromatic aldehydes to validate the scope and feasibility of this green catalytic procedure. The findings are summarised in Table 2. The results showed that this method is applicable to various aldehydes with electron-withdrawing groups, electron-donating groups, and heteroaryl aldehydes with yields ranging from 94–97%. The results indicated that lemon juice produced a comparable yield of the desired product as other reported methods.

Table 2. Synthesis of Betti base derivatives using Lemon juice

Entry	Aldehyde	Product	Yield (%)[a]	MP (˚C)	
				Observed	Literature
1	C_6H_5		96	201-203	201-203[xviii]
2	4-MeC_6H_4		97	184-186	183-185 [xix]
3	2-$OMeC_6H_4$		95	185-187	170-172 [xviii]
4	4-ClC_6H_4		97	208-210	208-210 [xviii]
5	3-FC_6H_4		95	180-182	179−181[xx]
6	4-$CF_3C_6H_4$		96	197-199	196-198[xxi]
7	4-CNC_6H_4		94	202-204	214-215[xxii]
8	3-$NO_2C_6H_4$		96	201-203	197-199[xxiii]
9	2-Thienyl		94	187-189	190-192[xix]

Reaction conditions: 2-naphthol (1 mmol), aldehyde (1 mmol), 2-aminobenzthiazole (1 mmol), Lemon juice (2 mL) and 5 ml glycerol; stirring at 80 °C for 5h

[a]Isolated yields

CONCLUSION:

In conclusion, a highly efficient eco-friendly route for three-component one-pot synthesis of 1-[(Benzothiazol-2-ylamino)-aryl-methyl]-naphthalen-2-ol derivatives using lemon juice as a natural catalyst has been developed. This approach provided various advantages like operational simplicity, short reaction time, good yields of the desired product, and an eco-friendly medium.

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