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Cetrimonium Bromide Promoted Efficient Multi-component Protocol for Synthesis of 1-Amidoalkyl-2-naphthols in Aqueous Medium

A.V. NAKHATE[1], A.P. INGALE[2], and S.V. SHINDE[3]

[1]Department of Chemistry, Marathwada Institute of Technology, Aurangabad-431001, India
[2]Department of Chemistry, Dada Patil College, Karjat, Ahmednagar-414402, India
[3]Department of Chemistry, Pratibha Niketan College, Nanded-431606, India

*Corresponding author: E-mail: arjun108vnakhate@gmail.com

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An efficient and eco-friendly cetrimonium bromide promoted one-pot synthesis of 1-amidoalkyl-2-naphthols by the multicomponent condensation of aldehydes, β-naphthol and amides in water is reported. The wide range of substrate scope, excellent yield, short reaction rate, operational simplicity and environmentally friendly water as medium are the prominent feature of this protocol.

Keywords: Amidoalkyl naphthols, Cetrimonium bromide, Multicomponent, β-Naphthol, Amides, Aldehyde.

INTRODUCTION

Multi-component reactions (MCRs) are the potential and efficient strategies for synthesis of diverse molecules in single synthetic operation from three or more available starting material. These types of reactions (MCRs) is commonly used for the formation of carbon-carbon and carbon-nitrogen bond [1-5]. The high atom economy, operational simplicity, cost and time effective, high selectivity and reduction of number of steps, energy consumption, reduction of waste production are the notable features of multicomponent reactions [6-8]. Therefore, it finds the unique position in organic synthesis and several researchers have made considerable efforts to develop novel MCRs.

The multicomponent reaction between the β-naphthol, aldehydes and amides are used for construction of 1-amidoalkyl-2-naphthols compounds. The 1,3-amino oxygenated functional group bearing 1-amidoalkyl-2-naphthols possess the wide spectrum of biological and pharmacological activities [9-16]. It is an essential building blocks for a variety of biologically significant pharmaceuticals, natural products and potent drugs [17,18]. Thus, due to their medicinal significance, the number of methods has been developed for the synthesis of 1-amidoalkyl-2-naphthols scaffolds by using different Lewis or Brønsted acid catalysts, heterogeneous organic and inorganic catalysts, deep eutectic solvents and ionic liquids. The reported methods used for preparation of 1-amidoalkyl-2-naphthols include the catalysts such as *p*-TSA [19], $Fe(HSO_4)_3$ [20], P_2O_5 [21], I_2 [22], VB_1 [23], $HClO_4$-SiO_2 [24], wet-TCT [25], $ZrO(OTf)_2$ [26], $Bi(NO_3)_3$ [27], zwitterionic salts [28], sulphamic acid [29], DPA [30], graphine oxide [31], MSNs-HPZ-SO_3H [32], β-CD-BSA [33], $NiFe_2O_4$@SiO_2-PPA [34], carbon-SO_3H [35], $Bi(OTf)_3$ [36], $InCl_3$ [37], $Cu(OTf)_2$·SiO_2 [38], SiO_2-$NaHSO_4$ [39], MoO_3-ZrO_2 [40], nano-Fe_3O_4-SO_3H [41], cellulose-SO_3H [42], $H_4SiW_{12}O_{40}$ [43], MCM-41-N-propylsufamic acid [44], $FeCl_3$-SiO_2 [45], nano-S_8 [46], $K_5CoW_{12}O_{40}$ [47], montmorillonite K-10 clay [48], trityl chloride [49], cation exchange resin [50], deep eutectic solvents [51] and Brønsted acidic ionic liquids [52-56]. Many of these reported methods have some synthetic advantages individually, but they still suffers from certain limitations, such as low yields, higher reaction temperature, prolonged reaction time, harsh reaction conditions, waste production, formation of byproducts and use of toxic catalyst and solvents. Therefore, to overcome above all these problems, there is need of development of eco-friendly and efficient protocol with high catalytic activity and short reaction times for the synthesis of 1-amidoalkyl-2-naphthols.

Cetrimonium bromide, also known as cetyltrimethyl ammonium bromide (CTAB), is a quaternary ammonium surfactant

and phase transfer catalyst. It has been extensive explored as surface active agents and widely used as a wetting agent, as emulsifier and plasticizer in the industrial process [57-59]. It showed the micellar catalytic effect and has been used as catalyst in numerous organic transformation [60-66]. It is commonly used in water medium. The CTAB/H_2O catalytic system has several advantages such as improvement of reaction rate and various reaction conditions to eliminate the use of expensive anhy-drous or aprotic hazardous organic solvents [67]. Herein, we report a CTAB catalyzed efficient, metal-free and ecofriendly one-pot synthetic strategy for synthesis of 1-amidoalkyl-2-naphthol in water medium.

EXPERIMENTAL

All the reagents were purchased from the commercial suppliers and used without any purification. Melting points were determined in open glass capillary tubes on a Mettler FP 51 melting point apparatus and are uncorrected. The NMR spectra were recorded on a Brucker Avance Digital 500/400 MHz (^{1}H) and 125/100 MHz (^{13}C) in $CDCl_3$ using tetramethylsilane as an internal standard.

General procedure: To a mixture of β-naphthol (1 mmol), aldehyde (1 mmol) and amide (1 mmol) in 10 mL water, CTAB (10%) were added. Then resulting reaction mixture was heated at 100 ºC in oil bath with constant stirring till the reaction was completed. After completion of the reaction as indicated on TLC, the reaction mixture was cooled at room temperature and finally extracted with ethyl acetate. The organic layer was washed with distilled water and dried over sodium sulphate. The organic layer was evaporated under reduced pressure and desired pure product was isolated easily through the crystallization from hot ethanol (**Scheme-I**). The structure and purity of the synthesized compounds were determined by FT-IR, ^{1}H & ^{13}C NMR and HRMS.

***N*-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl)-benzamide (Table-2, entry 1):** White, yield 94%, m.p.: 233-235 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3475 (N-H), 3361 (O-H), 2984 (C-H), 1656 (C=O); ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.34 (s, 1H), 9.02 (d, *J* = 8.5 Hz, 1H), 8.09 (d, *J* = 8.5 Hz, 1H), 7.89-7.78 (m, 4H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.47 (dd, *J* = 15.0, 7.5 Hz, 3H), 7.34-7.23 (m, 7H), 7.22-7.17 (m, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.2, 153.7, 142.5, 134.8, 132.8, 131.9, 129.9, 129.1, 129.0, 128.9, 128.7, 127.6, 127.2, 127.0, 126.9, 123.2, 119.2, 118.8, 49.7; HRMS (ESI) *m/z* calcd. for $C_{24}H_{20}NO_2$ [M+H]$^+$ 354.1494, found: 354.1491.

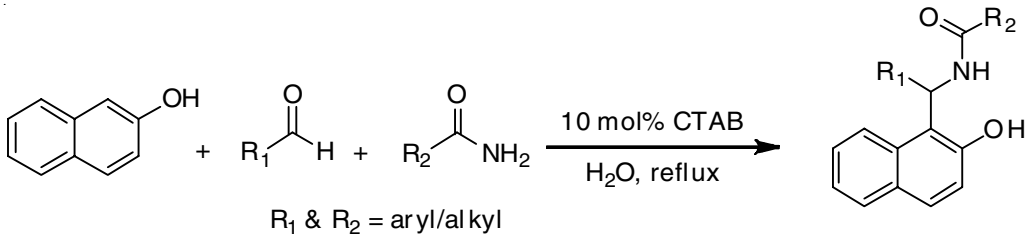
***N*-[(2-Hydroxynaphthalen-1-yl)phenylmethyl]acetamide (Table-2, entry 2):** White, yield 95%, m.p.: 228-230 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3459 (N-H), 3388 (O-H), 3007 (C-H), 1660 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.70 (bs, 1H), 7.94-7.88 (m, 6H), 7.53 (bs, 1H), 7.21-7.07 (m, 5H), 2.01 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 167.6, 152.7, 144.7, 134.6, 128.9, 128.7, 128.1, 127.8, 127.1, 125.5, 124.7, 123.6, 122.3, 122.1, 118.2, 43.6, 21.7; HRMS (ESI) *m/z* calcd. for $C_{19}H_{18}NO_2$ [M+H]$^+$ 291.1337, found: 292.1333.

[(2-Hydroxynaphthalen-1-yl)phenylmethyl]urea (Table-2, entry 3): White, yield 94%, m.p.: 171-177 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3453 (N–H), 3382 (O–H), 3231 (NH_2), 2956 (C–H), 1661 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.99 (bs, 1H), 7.75-7.70 (m, 5H), 7.35 (bs, 1H), 7.17-7.13 (m, 6H), 6.90 (s, 1H), 5.79 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 159.1, 153.3, 144.7, 132.5, 131.1, 129.5, 129.1, 128.3, 126.4, 126.2, 122.9, 121.2,119.7, 48.6; HRMS (ESI) *m/z* calcd. for $C_{18}H_{17}N_2O_2$ [M+H]$^+$ 293.3396, found: 293.3391.

***N*-((2-Hydroxynaphthalen-1-yl)(*p*-tolyl)methyl)-benzamide (Table-2, entry 4):** White, yield 88%, m.p.: 209-211 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3418, 3028, 2951, 1629, 1530, 1381, 1346, 870, 748, 687; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.33 (s, 1H), 9.03 (d, *J* = 8.5 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.84 (t, *J* = 7.5 Hz, 3H), 7.79 (d, *J* = 9.0 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.51-7.43 (m, 3H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.23 (dd, *J* = 19.0, 8.5 Hz, 4H), 6.84 (d, *J* = 9.0 Hz, 2H), 3.68 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.0, 158.5, 153.5, 134.9, 134.4, 132.7, 131.9, 129.7, 129.1, 129.0, 128.9, 128.2, 127.5, 127.2, 123.1, 119.2, 118.9, 114.1, 55.5, 49.4; HRMS (ESI) *m/z* calcd. for $C_{25}H_{22}NO_2$ [M+H]$^+$ 368.1650, found: 368.1646.

***N*-[(2-Hydroxynaphthalen-1-yl)-*o*-tolylmethyl]-acetamide (Table-2, entry 5):** Off white, yield 84%, m.p.: 200-203 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3422 (N–H), 3322 (O–H), 2993 (C–H), 1625 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.98 (br, s, 1H, OH), 8.04-7.89 (m, 6H, Ar–H), 7.30-7.21 (m, 4H, Ar–H), 6.65 (br, s, 1H, NH), 5.77 (s, 1H, CH), 2.27 (s, 3H, CH_3), 1.89 (s, 3H, CH_3); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 167.5, 152.7, 143.5, 139.5, 134.3, 132.4, 129.2, 128.6, 128.1, 126.3, 125.4, 123.4, 122.0, 118.3, 50.1, 22.7, 19.4; HRMS (ESI) *m/z* calcd. for $C_{20}H_{20}NO_2$ [M+H]$^+$ 306.1494, found: 306.1491.

***N*-((4-(*tert*-Butyl)phenyl)(2-hydroxynaphthalen-1-yl)-methyl)benzamide (Table-2, entry 6):** White, yield 90%, m.p.: 207-209 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3420, 3061, 2961, 1630, 1535, 1437, 875, 754, 689; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.33 (s, 1H), 9.04 (d, *J* = 8.5 Hz, 1H), 8.11 (d, *J* = 8.5 Hz, 1H), 7.90-7.82 (m, 3H), 7.79 (d, *J* = 9.0 Hz, 1H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.50-7.42 (m, 3H), 7.35-7.19 (m, 7H), 1.22 (s, 9H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.0, 153.6, 149.4, 139.4, 134.9, 132.8, 131.9, 129.7, 129.1, 129.0,



Scheme-I: Synthesis of CTAB catalyzed 1-amidoalkyl-2-naphthols

128.8, 128.7, 127.9, 127.6, 127.2, 126.8, 125.5, 123.2, 119.2, 118.9, 49.6, 34.6, 31.6; HRMS (ESI) *m/z* calcd. for $C_{28}H_{28}NO_2$ [M+H]+ 410.2120, found: 410.2126.

[(2-Hydroxynaphthalen-1-yl)-(4-methoxyphenyl)-methyl]urea (Table-2, entry 7): White, yield 86%, m.p.: 182-185 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3481 (N-H), 3370 (O-H), 3265 (NH_2), 2977 (C-H), 1637 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 10.03 (bs, 1H, OH), 7.95-7.89 (m, 6H), 7.44 (bs, 1H), 7.28-7.23 (m, 4H), 6.88 (s, 1H), 5.79 (s, 2H), 3.57 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 162.2, 153.4, 142.5, 135.5, 133.6, 129.5, 128.9, 128.2, 126.1, 123.5, 122.1, 118.6, 56.1, 50.1; HRMS (ESI) *m/z* calcd. for $C_{19}H_{19}N_2O_3$ $[M+H]^+$ 323.1395, found: 323.1389.

***N*-[(2-Hydroxynaphthalen-1-yl)-(4-hydroxyphenyl)-methyl]benzamide (Table-2, entry 8):** Light brown, yield 89%, m.p.: 191-193 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3488 (N-H), 3366 (N-H), 2955 (C-H), 1655; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.18 (s, 1H), 9.64 (s, 1H), 8.92 (s, 1H), 8.24 (d, *J* = 9.0 Hz, 1H), 7.84 (d, *J* = 7.0 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.53 (t, *J* = 7.0 Hz, 1H), 7.50-7.37 (m, 4H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 9.0 Hz, 1H), 7.05 (t, *J* = 7.0 Hz, 1H), 6.80 (d, *J* = 7.5 Hz, 1H), 6.68 (t, *J* = 7.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 165.6, 155.3, 153.6, 135.0, 133.0, 131.7, 129.5, 129.2, 128.9, 128.8, 128.5, 128.2, 127.6, 126.6, 123.8, 123.3, 122.9, 119.3, 119.3, 119.1, 115.8, 46.2; HRMS (ESI) *m/z* calcd. for $C_{24}H_{20}NO_3$ [M+H]+ 370.1443, found: 370.1439.

***N*-[(2-Hydroxynaphthalen-1-yl)-(4-hydroxyphenyl)-methyl]acetamide (Table-2, entry 9):** Light crimson, yield 89%, m.p.: 186-188 °C; FT-IR (KBr, ν_{max}, cm^{-1}): 3480 (N-H), 3365 (O-H), 2992 (C-H), 1675 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 10.07 (bs, 1H, OH), 7.71-7.68 (m, 6H), 7.44 (bs, 1H), 7.22-7.17 (m, 4H), 6.41 (s, 1H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 167.2, 148.1, 140.3, 130.4, 129.7, 129.1, 128.4, 127.7, 127.2, 125.6, 124.1, 122.7, 122.1, 120.5, 119.0, 49.3 22.1; HRMS (ESI) *m/z* calcd. for $C_{19}H_{18}NO_3$ $[M+H]^+$ 308.1286, found: 308.1290.

[(2-Hydroxynaphthalen-1-yl)-(4-hydroxyphenyl)-methyl]urea (Table-2, entry 10): White, yield 86%, m.p.: 147-149 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3458 (N-H), 3340 (O-H), 3287 (NH_2), 2965 (C-H), 1656 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 10.51 (bs, 1H), 8.13-7.95 (m, 6H), 7.57 (bs, 1H), 7.20-6.98 (m, 4H), 6.69 (s, 1H), 5.79 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 160.7, 150.5, 143.5, 132.5, 129.0, 129.6, 128.1, 127.8, 127.3, 125.6, 124.8, 122.6, 122.1, 119.8, 47.5; HRMS (ESI) *m/z* calcd. for $C_{18}H_{17}N_2O_3$ [M+H]+ 309.1239, found: 309.1242.

***N*-((2-Hydroxynaphthalen-1-yl)(2-hydroxyphenyl)-methyl)benzamide (Table-2, entry 11):** White, yield 81%, m.p.: 234-236 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3592, 3205, 2363, 1607, 1565, 1349, 816, 752, 687, 588; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.18 (s, 1H), 9.64 (s, 1H), 8.92 (s, 1H), 8.24 (d, *J* = 9.0 Hz, 1H), 7.84 (d, *J* = 7.0 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.53 (t, *J* = 7.0 Hz, 1H), 7.50-7.37 (m, 4H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28 (t, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 9.0 Hz, 1H), 7.05 (t, *J* = 7.0 Hz, 1H), 6.80 (d, *J* = 7.5 Hz, 1H), 6.68 (t, *J* = 7.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 165.6, 155.3, 153.6, 135.0, 133.0, 131.7, 129.5, 129.2, 128.9, 128.8, 128.5, 128.2, 127.6, 126.6, 123.8, 123.3, 122.9, 119.3, 119.3, 119.1, 115.8, 46.2; HRMS (ESI) *m/z* calcd. for $C_{24}H_{20}NO_3$ $[M+H]^+$ 370.1443, found: 370.1447.

***N*-((4-(Dimethylamino)phenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (Table-2, entry 12):** White, yield 83%, m.p.: 219-221 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3413, 3071, 2803, 1630, 1533, 1346, 1329, 1166, 813, 754, 686, 567; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.32 (s, 1H), 9.03 (d, *J* = 8.5 Hz, 1H), 8.09 (d, *J* = 9.0 Hz, 1H), 7.92-7.77 (m, 3H), 7.79 (d, *J* = 9.0 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.52-7.42 (m, 3H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 9.0 Hz, 1H), 7.21 (d, *J* = 8.5 Hz, 1H), 7.13 (d, *J* = 8.5 Hz, 2H), 6.65 (d, *J* = 8.5 Hz, 2H), 2.82 (s, 6H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 165.8, 153.5, 149.9, 135.0, 132.7, 131.8, 129.9, 129.5, 129.0, 128.8, 127.9, 127.4, 127.1, 123.1, 119.3, 119.2, 112.9, 112.8, 49.6, 40.7; HRMS (ESI) *m/z* calcd. for $C_{26}H_{25}N_2O_2$ $[M+H]^+$ 397.1916, found: 397.1913.

***N*-((4-Fluorophenyl)(2-hydroxynaphthalen-1-yl)-methyl)benzamide (Table-2, entry 13):** White, yield 94%, m.p.: 232-234 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3418, 3063, 1630, 1511, 1345, 1261, 1170, 825, 747, 712, 589; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.37 (s, 1H), 9.05 (d, *J* = 8.5 Hz, 1H), 8.08 (d, *J* = 8.5 Hz, 1H), 7.90-7.83 (m, 3H), 7.81 (d, *J* = 9.0 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.52-7.45 (m, 3H), 7.30 (d, *J* = 14.0, 7.5 Hz, 4H), 7.25 (d, *J* = 9.0 Hz, 1H), 7.11 (t, *J* = 9.0 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.3, 162.4 (d, *J* = 245.7 Hz), 153.7, 138.6, 138.6, 134.7, 132.7, 131.9, 130.1, 130.0, 129.1, 129.0, 128.9 (d, *J* = 8.1 Hz), 127.7, 127.3, 123.2, 119.2, 118.6, 115.5 (d, *J* = 21.1 Hz), 49.3; HRMS (ESI) *m/z* calcd. for $C_{24}H_{19}FNO_2$ $[M+H]^+$ 372.1400, found: 372.1406.

***N*-((4-Chlorophenyl)(2-hydroxynaphthalen-1-yl)-methyl)benzamide (Table-2, entry 14):** White, yield 95%, m.p.: 187-189 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3440 (N-H), 3325 (O-H), 3070 (C-H), 1644 (C=O); ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.35 (s, 1H), 9.02 (d, *J* = 8.0 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.90-7.80 (m, 4H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.48 (dd, *J* = 13.5, 7.5 Hz, 3H), 7.34 (t, *J* = 7.5 Hz, 3H), 7.31-7.28 (m, 3H), 7.25 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.4, 153.7, 141.6, 134.7, 132.7, 132.0, 131.6, 130.1, 129.1, 129.0, 128.9, 128.8, 128.6, 127.7, 127.3, 123.2, 119.1, 118.4, 49.2; HRMS (ESI) *m/z* calcd. for $C_{24}H_{19}ClNO_2$ $[M+H]^+$ 388.1104, found: 388.1107.

[(4-Chlorophenyl)-(2-hydroxynaphthalen-1-yl)-methyl]urea (Table-2, entry 15): White, yield 96%, m.p.: 211-213 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3485 (N-H), 3390 (O-H), 3192 (NH_2), 2955 (C-H), 1650 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.96 (bs, 1H), 7.79-7.71 (m, 6H), 7.36 (bs, 1H), 7.24-7.10 (m, 4H), 6.86 (s, 1H), 5.80 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 159.0, 153.4, 144.0, 132.5, 130.8, 129.7, 129.1, 128.3, 128.1, 127.1, 123.0, 118.9, 49.9; HRMS (ESI) *m/z* calcd. for $C_{18}H_{16}ClN_2O_2$ $[M+H]^+$ 327.0900, found: 327.0905.

***N*-[(2-Chlorophenyl)-(2-hydroxynaphthalen-1-yl)-methyl]acetamide (Table-2, entry 16):** Off white, yield 91%,

m.p.: 213-215 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3440 (N-H), 3251 (O-H), 2990 (C-H), 1645 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.98 (bs, 1H), 7.94-7.88 (m, 6H), 7.47 (bs, 1H), 7.29-7.36 (m, 4H), 6.03 (s, 1H), 1.91 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 165.6, 154.1, 140.4, 133.5, 133.4, 132.8, 132.1, 130.3, 129.8, 129.1, 128.7, 127.6, 127.0, 123.1, 123.6, 119.2, 117.4, 48.6, 23.5; HRMS (ESI) *m/z* calcd. for $C_{19}H_{17}ClNO_2$ [M+H]+ 326.0948, found: 326.942.

***N*-((4-Bromophenyl)(2-hydroxynaphthalen-1-yl)-methyl)benzamide (Table-2, entry 17):** White solid, yield 95%, m.p.: 197-199 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3418, 3182, 1628, 1576, 1341, 810, 724, 585; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.36 (s, 1H), 9.02 (d, *J* = 8.5 Hz, 1H), 8.07 (d, *J* = 8.5 Hz, 1H), 7.89-7.84 (m, 3H), 7.82 (d, *J* = 9.0 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.53-7.44 (m, 5H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.30-7.21 (m, 4H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.4, 153.7, 142.0, 134.7, 132.7, 132.0, 131.5, 130.1, 129.2, 129.1, 129.0, 128.9, 127.7, 127.3, 123.2, 120.1, 119.1, 118.3, 49.3; HRMS (ESI) *m/z* calcd. for $C_{24}H_{19}BrNO_2$ $[M+H]^+$ 432.0599, found: 432.0603.

***N*-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)-methyl)benzamide (Table-2, entry 18):** Light Yellow, yield 96%, m.p.: 230-232 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3434, 3067, 2850, 1638, 1516, 1345, 851, 800, 736; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.40 (s, 1H), 9.08 (d, *J* = 8.0 Hz, 1H), 8.17 (d, *J* = 9.0 Hz, 2H), 8.08 (d, *J* = 8.5 Hz, 1H), 7.92 (d, *J* = 7.5 Hz, 2H), 7.85 (t, *J* = 9.0 Hz, 2H), 7.58-7.54 (m, 3H), 7.50 (t, *J* = 8.0 Hz, 3H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 9.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 166.8, 153.9, 150.7, 146.7, 134.4, 132.7, 132.1, 130.5, 129.2, 128.9, 128.9, 128.0, 127.9, 127.5, 123.9, 123.3, 123.1, 119.0, 117.9, 49.5; HRMS (ESI) *m/z* calcd. for $C_{24}H_{19}N_2O_4$ $[M+H]^+$ 399.1345, found: 399.1339.

***N*-[(2-Hydroxynaphthalen-1-yl)-(3-nitrophenyl)-methyl]acetamide (Table-2, entry 19):** Pale yellow, yield 94%, m.p.: 240-242 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3436 (N-H), 3258 (O-H), 2997 (C-H), 1644 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 10.03 (bs, 1H), 8.03-7.73 (m, 6H), 7.51 (bs, 1H), 7.11-7.02 (m, 4H), 6.10 (s, 1H), 1.95 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 162.3, 154.2, 132.5, 130.1, 129.4, 128.8, 128.1, 127.3, 126.4, 123.3, 122.9, 122.1, 118.3, 117.0, 115.3, 114.1, 43.9, 22.3; HRMS (ESI) *m/z* calcd. for $C_{19}H_{17}N_2O_4$ $[M+H]^+$ 337.1188, found: 337.1183.

[(2-Hydroxynaphthalen-1-yl)-(3-nitrophenyl)methyl]-urea (Table-2, entry 20): Light yellow, yield 93%, m.p.: 211-213 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3484 (N-H), 3374 (O-H), 3260 (NH_2), 2971 (C-H), 1632 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.91 (bs, 1H), 8.11-8.07 (m, 6H), 7.91-7.85 (m, 4H), 7.34 (bs, 1H), 6.84 (s, 1H), 5.82 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 161.7, 153.4, 147.3, 145.0, 133.4, 132.1, 130.2, 129.2, 128.9, 128.6, 128.1, 127.3, 123.3, 119.1, 49.4; HRMS (ESI) *m/z* calcd. for $C_{18}H_{16}N_3O_4$ $[M+H]^+$ 338.1141, found: 338.1145.

2-(Benzamido(2-hydroxynaphthalen-1-yl)methyl)-benzoic acid (Table-2, entry 21): White, yield 83%, m.p.: 235-237 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3227, 1716, 1628, 1513, 1302, 935, 818, 743, 711; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 12.82 (s, 1H), 9.82 (s, 1H), 8.85 (d, *J* = 7.5 Hz, 1H), 8.14 (d, *J* = 8.5 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 7.5 Hz, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.75 (d, *J* = 9.0 Hz, 1H), 7.67 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.46-7.31 (m, 5H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.15 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, δ ppm): 165.7, 154.2, 141.2, 134.9, 133.7, 131.5, 130.8, 129.7, 129.6, 130.0, 128.8, 128.8, 128.6, 127.9, 127.2, 126.7, 123.7, 122.8, 119.4, 118.5, 48.7, 31.2; HRMS (ESI) *m/z* calcd. for $C_{25}H_{20}NO_4$ $[M+H]^+$ 398.1392, found: 398.1392.

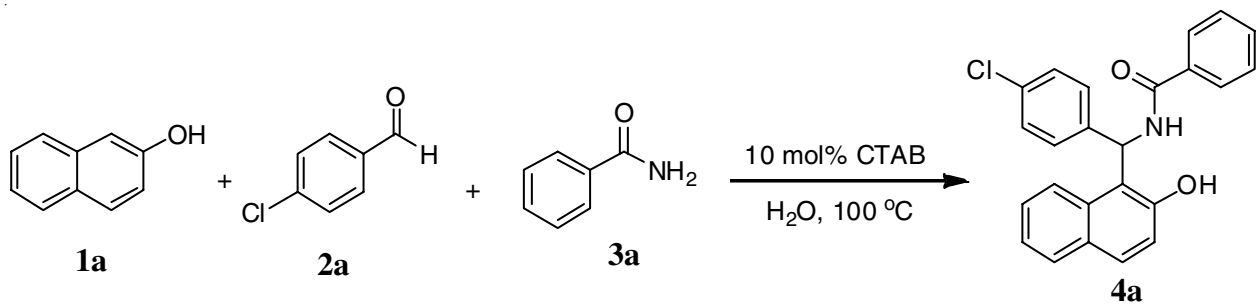
***N*-(Furan-2-yl(2-hydroxynaphthalen-1-yl)methyl)-benzamide (Table-2, entry 22):** Brown, yield 92%, m.p.: 221-223 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3409, 2197, 1632, 1572, 1437, 816, 745, 597; ^{1}H NMR (500 MHz, DMSO-d_6, δ ppm): 10.21 (s, 1H), 9.09 (d, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 8.5 Hz, 1H), 7.89-7.77 (m, 4H), 7.59-7.52 (m, 2H), 7.47 (t, *J* = 7.5 Hz, 3H), 7.36-7.27 (m, 2H), 7.23 (d, *J* = 9.0 Hz, 1H), 6.36 (dd, *J* = 3.0, 2.0 Hz, 1H), 6.13 (d, *J* = 3.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6, ppm) δ 166.1, 154.5, 153.9, 142.5, 142.5, 134.7, 132.7, 131.9, 130.0, 129.0, 128.9, 128.8, 128.7, 127.7, 127.0, 123.4, 123.0, 119.0, 116.8, 110.9, 110.9, 107.2, 107.2, 45.0; HRMS (ESI) *m/z* calcd. for $C_{22}H_{18}NO_3$ $[M+H]^+$ 344.1286, found: 344.1280.

[Furan-2-yl-(2-hydroxynaphthalen-1-yl)methyl]urea (Table-2, entry 23): Off white, yield 93%, m.p.: 162-164 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3471 (N-H), 3392 (O-H), 3254 (NH_2), 2987 (C-H), 1669 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.97 (s, 1H, OH), 7.75-7.69 (m, 6H), 7.39 (s, 1H), 6.86-6.88 (m, 3H), 6.02 (s, 1H), 5.76 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 159.6, 156.8, 153.7, 141.8, 132.8, 129.7, 129.0, 128.7, 126.9, 122.9, 119.0, 118.2, 110.8, 105.6, 50.2; HRMS (ESI) *m/z* calcd. for $C_{16}H_{15}N_2O_3$ $[M+H]^+$ 283.1082, found: 283.1079.

[(2-Hydroxynaphthalen-1-yl)thiophen-2-yl-methyl]-urea (Table-2, entry 24): Light yellow, yield 92%, m.p.: 162-164 ºC; FT-IR (KBr, ν_{max}, cm^{-1}): 3474 (N-H), 3398 (O-H), 3258 (NH_2), 2982 (C-H), 1672 (C=O); ^{1}H NMR (400 MHz, DMSO-d_6, δ ppm): 9.95 (s, 1H), 7.77-7.71 (m, 6H), 7.33 (s, 1H), 6.83-6.88 (m, 3H), 6.22 (s, 1H), 5.80 (s, 2H); ^{13}C NMR (100 MHz, DMSO-d_6, δ ppm): 160.1, 157.3, 154.4, 141.9, 133.4, 129.8, 129.2, 128.8, 126.2, 122.7, 119.2, 118.5, 110.3, 105.9, 49.8; HRMS (ESI) *m/z* calcd. for $C_{16}H_{15}N_2O_2S$ $[M+H]^+$ 299.0854, found: 299.0857.

RESULTS AND DISCUSSION

In order to optimize condition of the title multicomponent synthesis of 1-amidoalkyl-2-naphthols, reaction of β-naphthol (**1a**), 4-chlorobenzaldehyde (**2a**) and benzamide (**3a**) was selected as the model reaction (**Scheme-II**). The optimized conditions for different surfactants, solvents, amount of catalyst and temperature are summarized in Table-1. At first, to determine the effect of temperature, the model reaction studied at different temperatures in water solvent. The reaction did not proceed in the absence of catalyst at room temperature under water and solvent-free condition (Table-1, entries 3-5). Stirring the three component mixture at 50 ºC, 70 ºC and 100 ºC also did not result in formation of product **4a** even after prolonging



Scheme-II: Model reaction for synthesis of 1-amidoalkyl-2-naphthols

TABLE-1
OPTIMIZATION OF REACTION CONDITIONS[a]

Entry	Catalyst	Solvent	Temp. (°C)	Time (h)	Yield (%)[b]
1	None	None	rt	12	NR[c]
2	None	Water	rt	12	NR[c]
3	None	Water	50	16	NR[c]
4	None	Water	70	16	NR[c]
5	None	Water	100	16	NR[c]
6	CTAB (10 mol%)	Water	rt	12	27
7	CTAB (10 mol%)	Water	50	6	53
8	CTAB (10 mol%)	Water	70	3	73
9	CTAB (10 mol%)	Water	100	1	95
10	CTAB (10 mol%)	Water	120	1	94
11	CTAB (10 mol%)	DMF	100	4	62
12	CTAB (10 mol%)	DMSO	100	4	55
13	CTAB (10 mol%)	THF	100	6	39
14	CTAB (10 mol%)	DCM	100	5	34
15	CTAB (10 mol%)	ACN	100	7	57
16	CTAB (10 mol%)	1,4-Dioxane	100	6	36
17	CTAB (10 mol%)	MeOH	100	5	45
18	CTAB (10 mol%)	EtOH	100	6	52
19	TTAB (10 mol%)	Water	100	2	73
20	TEAB (10 mol%)	Water	100	2.5	64
21	TBAB (10 mol%)	Water	100	2.5	53
22	TMAB (10 mol%)	Water	100	3	57
23	TBAF (10 mol%)	Water	100	3	37
24	CPC (10 mol%)	Water	100	3	43
25	SDS (10 mol%)	Water	100	3	26
26	CTAB (2.5 mol%)	Water	100	2	47
27	CTAB (5 mol%)	Water	100	2	73
28	CTAB (7.5 mol%)	Water	100	2	81
29	CTAB (15 mol%)	Water	100	1	94

[a]Reaction conditions: β-naphthol (**1a**, 1 mmol) 4-chlorobenzaldehyde (**2a**, 1 mmol) and benzamide (**3a**, 1 mmol), solvent (10 mL), stirring. [b]Isolated yield. [c]No reaction.

the reaction time (Table-1, entry 1). These negative results showed that the necessity of a catalyst. Next, we examined the model reaction by using CTAB (10 mol%) in water solvent. To our delight, the model multicomponent condensation in the presence of CTAB in the water at room temperature has been resulted into the formation of compound **4a** as expected in 27% yield after 12 h (Table-1, entry 6). To our satisfaction, by varying the temperature from room temperature to 100 ºC, the product yield gradually increased and at 100 ºC, the product 1-amidoalkyl-2-naphthols (**4a**) was obtained in excellent yield (Table-1, entry 3). There is no significant improvement in the reaction time and yield of product was observed above that temperature (Table-1, entry10), so 100 ºC was chosen as the optimum reaction temperature.

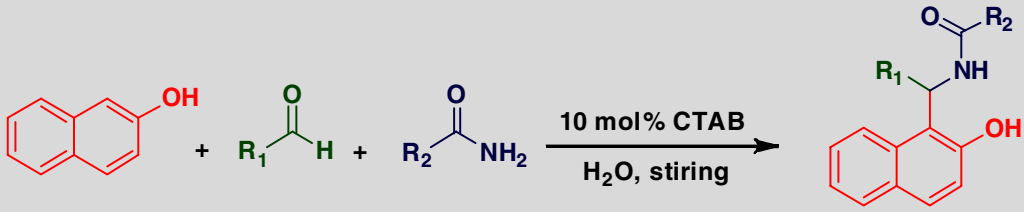
In order to check the effect of solvents on reaction time and yield of product, several solvents were screened. Then the reaction was performed in different common solvents such as DMF, DMSO, THF, DCM, ACN, 1,4-dioxane, CH_3OH and EtOH (Table-1, entries 11-18). The reactions gave the moderate yield in high boiling point organic solvents and comparatively poor yield in in low boiling point organic solvents. No significant improvement in the yield was observed; the reactions were sluggish and gave poor yield with longer reaction time. This solvent effect study demonstrates that water can be used as potential solvent for synthesis of 1-amidoalkyl-2-naphthols by using CTAB catalyst. Then, we investigated the catalytic utility of various types of surfactants such as TTAB, TEAB, TBAB, TMAB, TBAF, CPC and SDS for given transformation under the optimized condition (Table-1, entries 19-25). The surfactant screenings experiments showed that CTAB are more effective and excellent catalyt for this transformation.

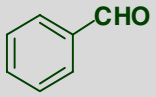
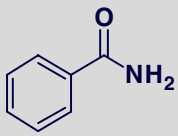
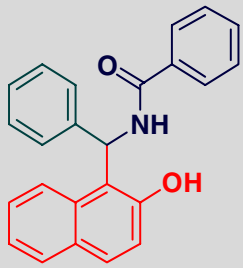
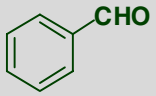
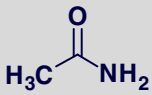
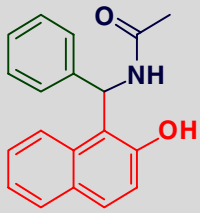
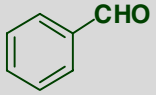
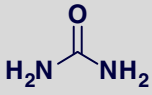
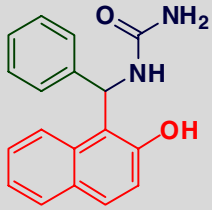
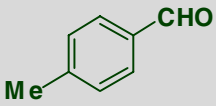
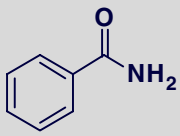
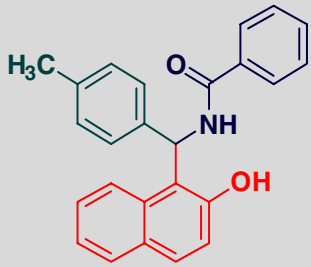
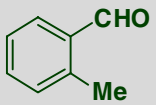
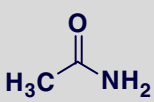
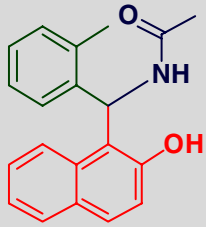
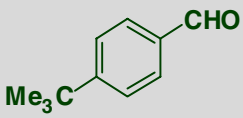
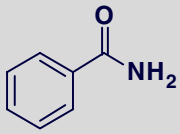
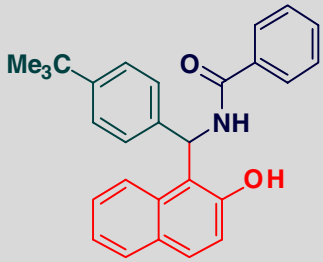
The effect of amount of catalyst loading in the model reaction was also examined. The study of catalyst loading indicated that the catalytic efficiency of CTAB increased from 2.5 to 15 mol% and showed the maximum efficiency at 10 mol% loading. The best result was attained in the presence of 10 mol% (Table-1, entries 9, 26-27). Further increasing the quantity of CTAB, keeping the reaction time constant, did not increase the yield remarkably (Table-1, entry 29).

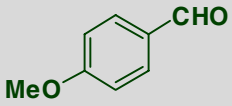
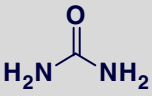
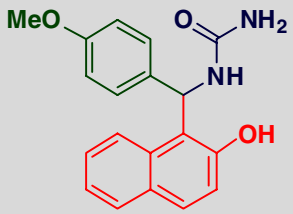
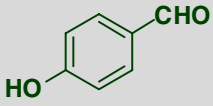
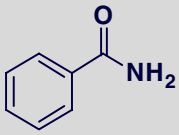
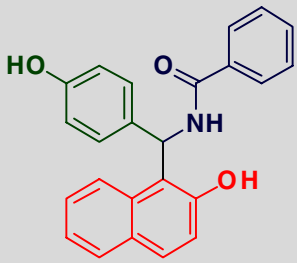
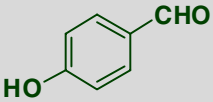
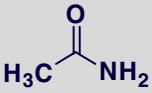
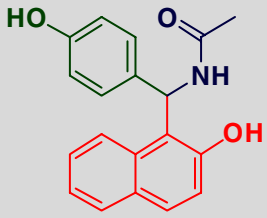
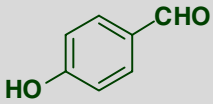
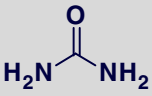
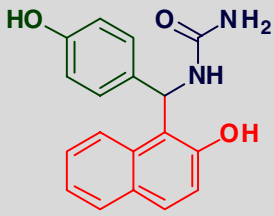
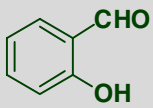
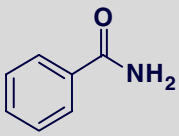
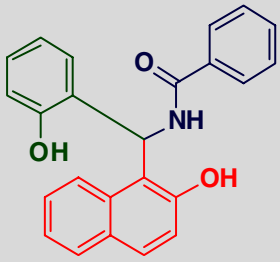
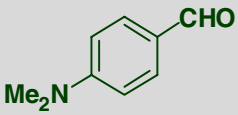
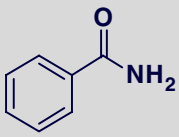
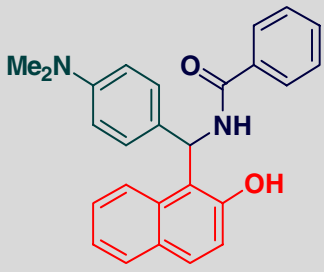
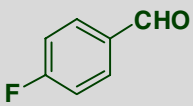
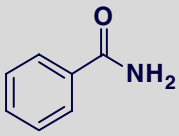
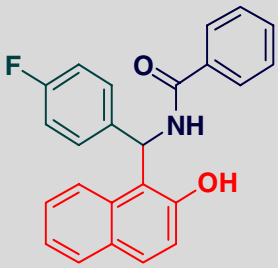
After optimization of the reaction conditions, to explore the scope and efficiency of the described protocol, the wide range of functionalized 1-amidoalkyl-2-naphthols derivatives were synthesized under optimized conditions. The reaction of wide range of aryl, heteroaryl, aliphatic aldehydes with β-naphthol and various amides such benzamide, acetamide, urea and 2-chloroacetamide under optimized conditions were also performed. The scope of this novel approch is illustrated in Table-2.

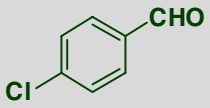
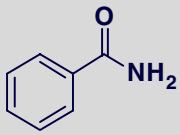
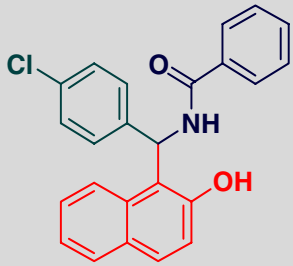
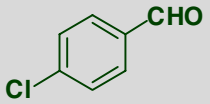
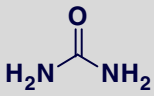
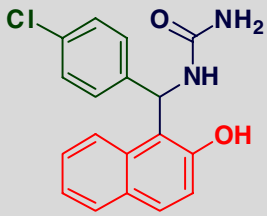
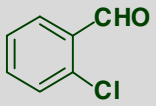
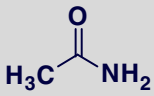
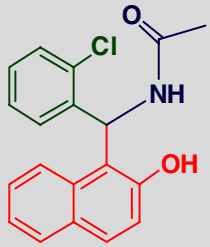
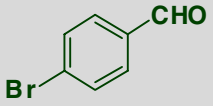
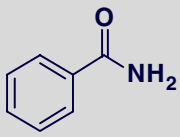
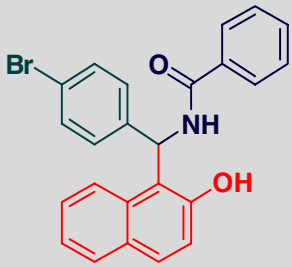
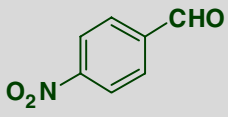
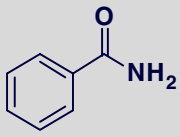
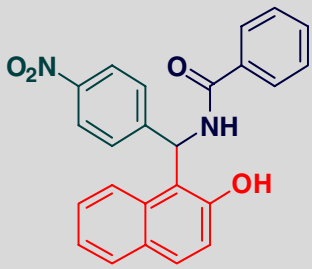
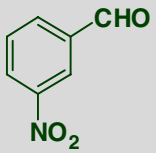
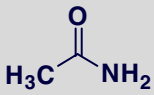
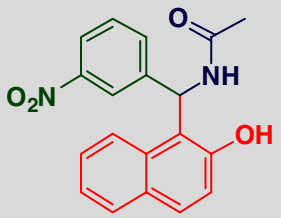
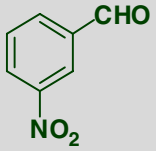
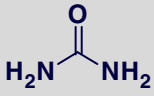
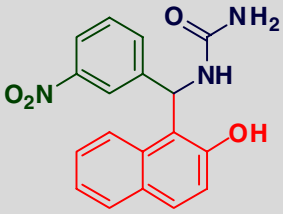
Delightfully, it was found that under optimized reaction condition benzaldehyde and aryl aldehydes bearing the electron-donating groups reacted smoothly and affording the 1-amidoalkyl-2-naphthol products in good yields (Table-2, entries 1-12). Halogen bearing aryl aldehydes reacts effectively with β-naphthol and various amides and afforded the desired products in excellent yields (Table-2, entries 13-17). Pleasingly, aryl aldehydes with electron withdrawing groups also successfully gave the desired products in excellent yields (Table-2,

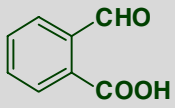
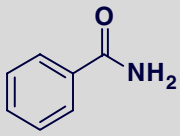
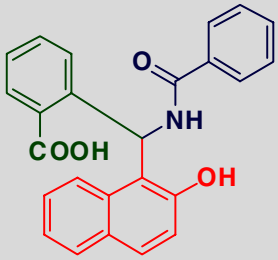
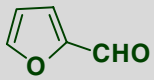
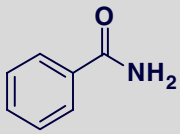
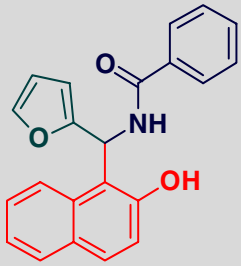
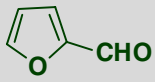
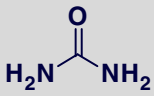
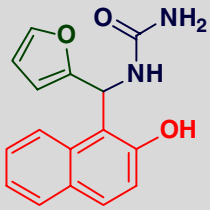
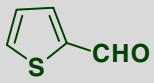
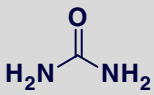
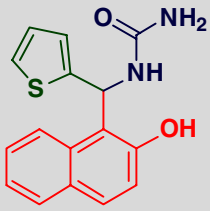
TABLE-2
SYNTHESIS OF 1-AMIDOALKYL-2-NAPHTHOL USING CTAB AS CATALYST[a]



Entry	Aldehyde	Amide	Products	Time (min)	Yield[b] (%)
1				90	94
2				90	95
3				90	94
4				120	88
5				150	84
6				110	90

7				120	86
8				120	85
9				120	89
10				120	86
11				150	81
12				150	83
13				75	94

14				60	95
15				60	96
16				75	91
17				60	95
18				60	96
19				60	94
20				60	95

21				90	83
22				90	92
23				90	93
24				90	92

[a]Reaction conditions: β-Naphthol (**1a**, 1 mmol), aldehydes (**2a**, 1 mmol) and amides (**3a**, 2 mmol), catalyst CTAB (10 mol%), solvent (10 mL), stirring. [b]Isolated yield.

entries 13-21). The reactions of aryl aldehydes bearing electron withdrawing groups were faster than aryl aldehydes containing electron donating groups (Table-2, entries 13-21 *vs*. entries 4-12). Notably, there is significant effect of steric hindrance on yields desired products. The *ortho*-substituted aryl aldehydes provided the desired product in moderate yields (Table-2, entries 5, 11, 21). Additionally, heteroaryl aldehydes also reacted effectively under the optimized reaction condition and gave the desired products in excellent yields (Table-2, entries 22-24).

Conclusion

In conclusion, a potent, ecofriendly and efficient synthesis of 1-amidoalkyl-2-naphthols by the one-pot three-component condensation of aldehydes, 2-naphthol and amides using CTAB as catalyst in water medium is present. The developed method is applicable for synthesis of 1-amidoalkyl-2-naphthols from varieties of functionalized aryl, heteroaryl, aliphatic aldehydes and amides. The inexpensive catalyst, faster reaction, functional group compatibility, excellent yield, no column purification, high catalytic activity, easy handling, low corrosiveness and environmental compatibility are the prominent features of this protocol. The above strategy is feasible, economical and environment friendly.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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