Rationally designed Cu(II) Schiff base metal complex anchored on $NiFe_2O_4$@Chitosan: An efficient heterogeneous and magnetically retrievable hybrid nanocatalyst for the one-pot multi-component synthesis of bioactive 1-amidoalkyl-2-naphthols

Sneha Paul[a], Pranjit Barman [a]*

[a]Department of Chemistry, National Institute of Technology, Silchar, Assam 788010, India
*Corresponding author: Prof. Pranjit Barman, Department of Chemistry, National Institute of Technology, Silchar, Assam 788010, India.
Email id:barmanpranjit@yahoo.co.in; pranjit@che.nits.ac.in; Contact no: +91-9435374128

Supplementary Information

Experimental

Materials and methods

$FeCl_3.6H_2O$, $NiCl_2.6H_2O$, 4-(diethylamino)-salicylaldehyde, $Cu(OAc)_2.H_2O$, ethanol, and Chitosan(CS)were acquired from SRL Chemical without undergoing further purification.

With KBr pellets, IR (infrared)spectra were obtained on a Bruker 3000 Hyperion Microscope that was set up with a Vertex 80 FT-IR system. The crystal phases were investigated using a Panalytical Xpert3 X-ray diffractometer that was outfitted with Cu-Kα radiation (λ = 1.54 Å). Jeol 6390LA/OXFORD XMX N SEM instrument was utilized to confirm the surface structure of the composite. With the JEOL Model 2100 F, TEM images are captured.Nova Touch LX_2 gas sorption analyzer from Quantachrome Instruments was used to measure the BET surface area and pore size distribution of materials. Microsense EV7 Vibrating Sample Magnetometer was used to detect the magnetic properties. X-ray photoelectron spectroscopy (XPS) was performed using a PHI 5000 VERSA Probe III (ULVAC PHI, USA). Inductively Coupled Plasma- Atomic Emission Spectroscopy (ICP-AES) was performed using ARCOS, Simultaneous ICP spectrometer.

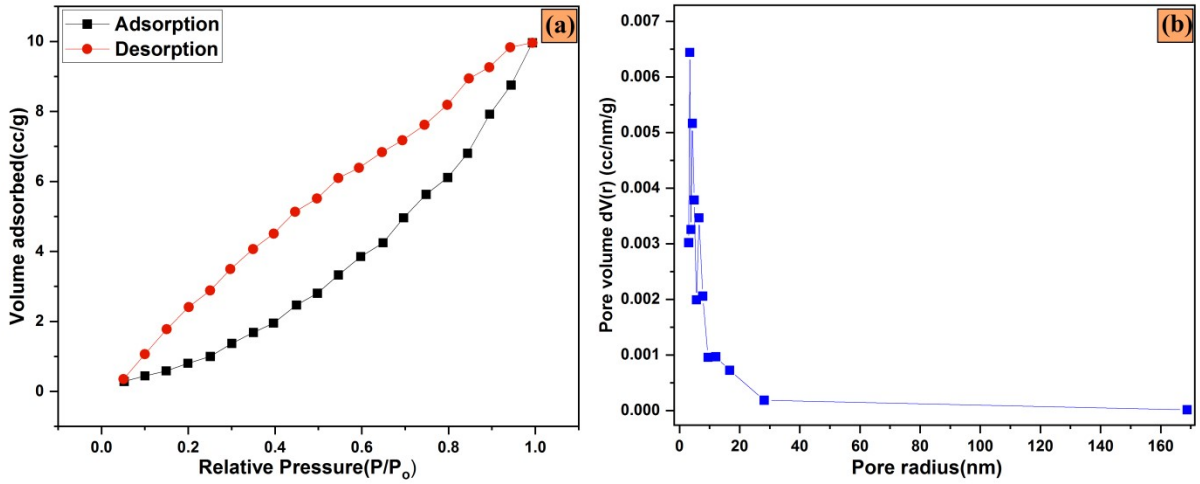

Fig. S1. (a) N_2 adsorption/desorption isotherm and (b) BJH plot for pore-size distribution o $NiFe_2O_4$@CS@CuSB

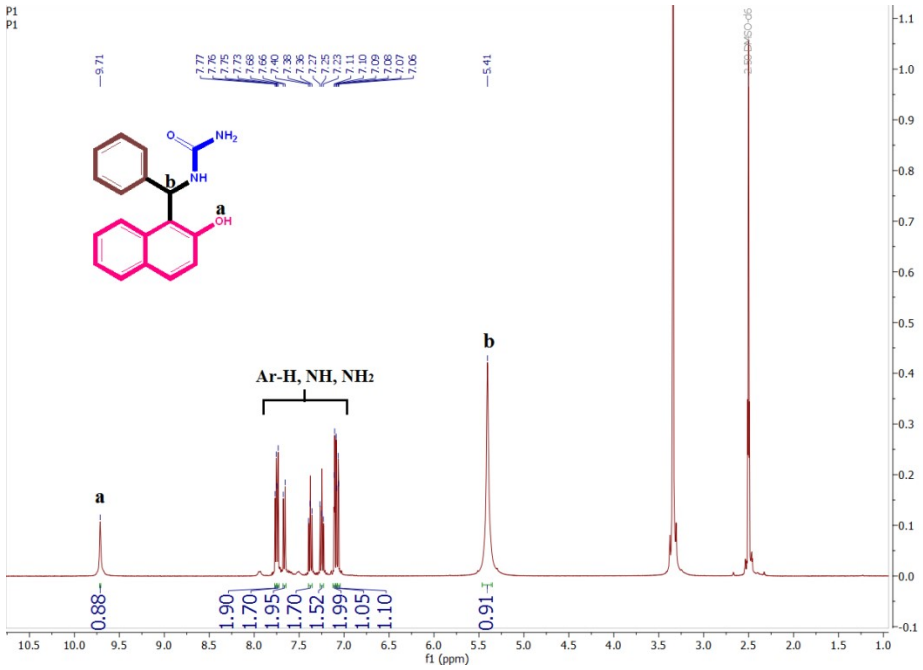

Fig.S2(a).[1]H NMR spectra of 4a

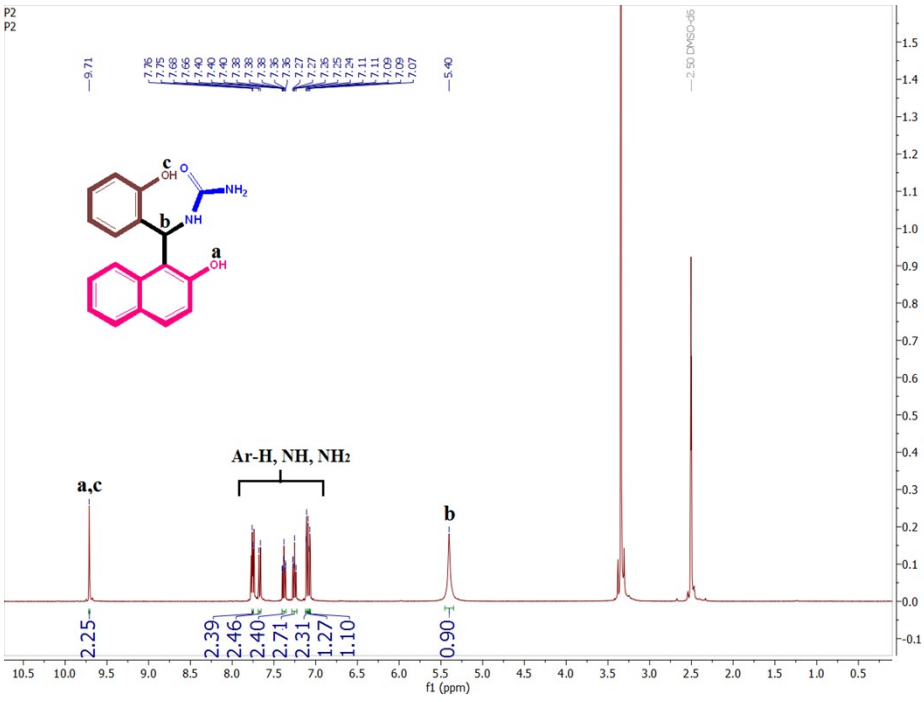

Fig.S2(b). [1]H NMR spectra of 4b

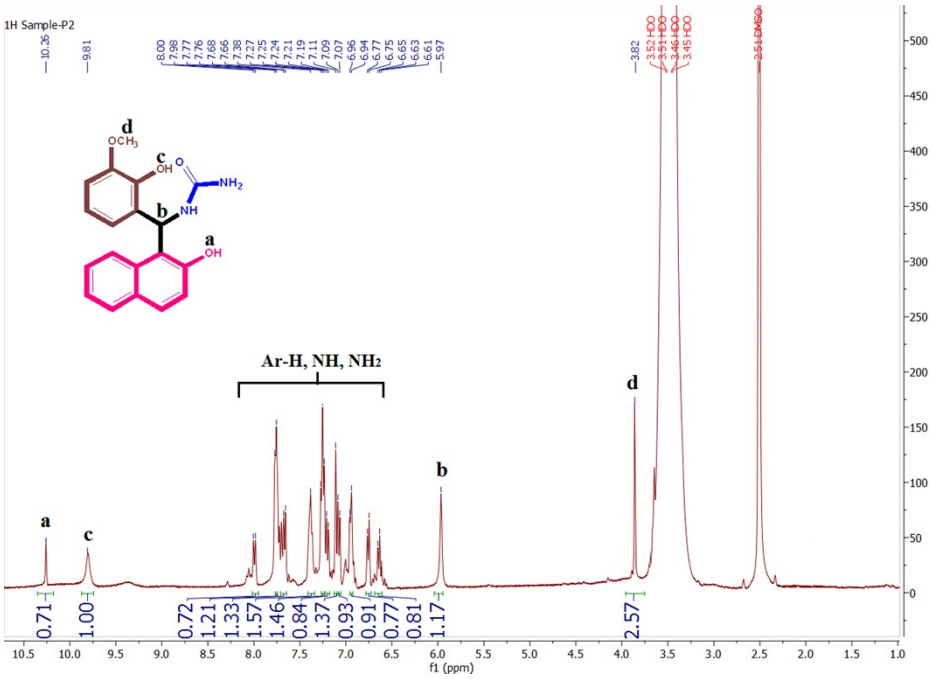

Fig.S2(c). ^{1}H NMR spectra of 4c

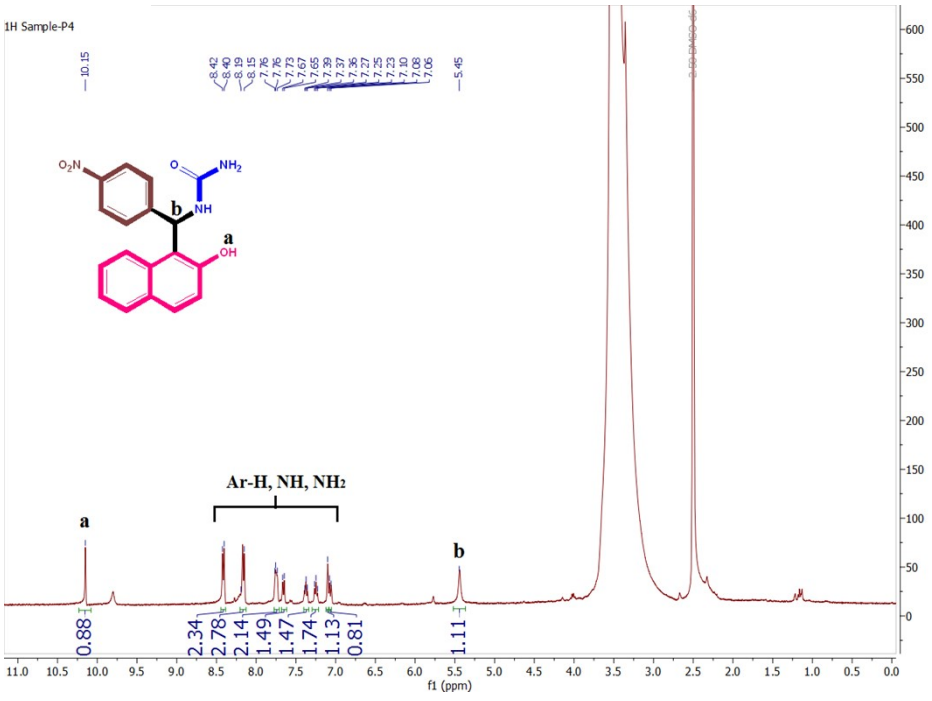

Fig.S2(d). ^{1}H NMR spectra of 4d

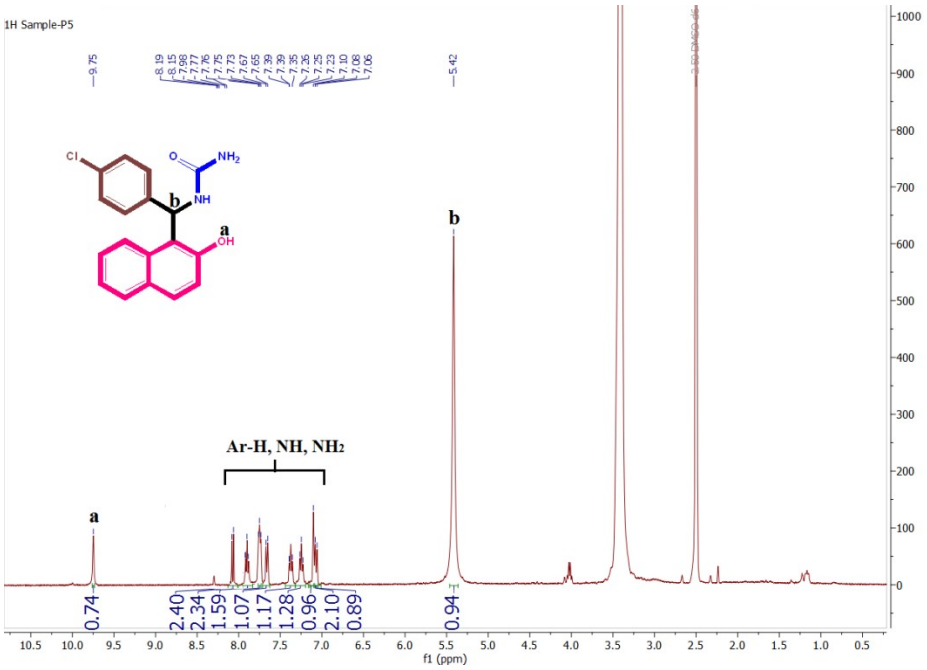

Fig.S2(e). ^{1}H NMR spectra of 4e

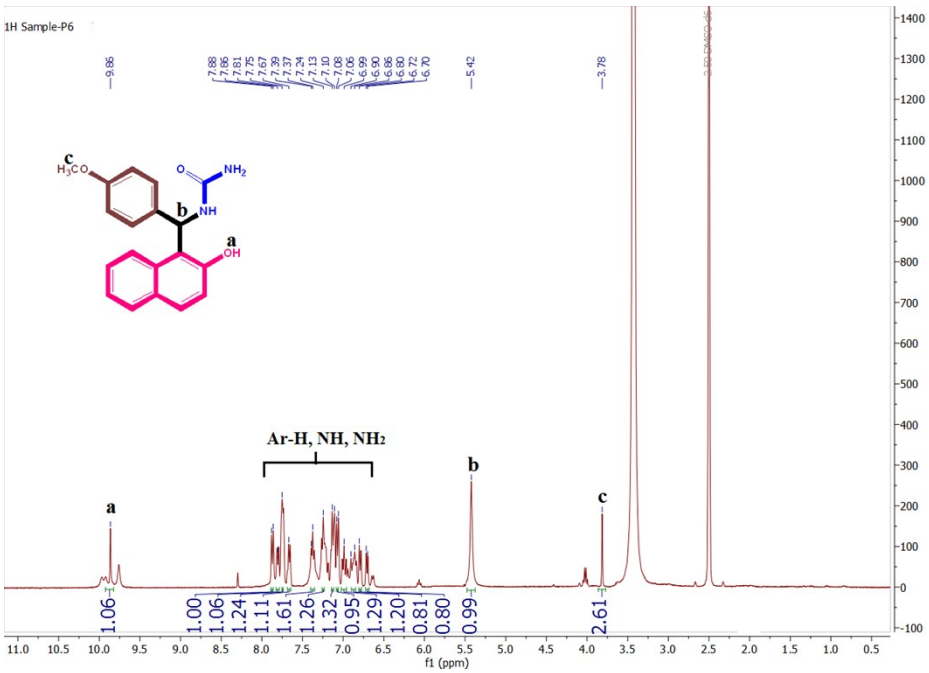

Fig.S2(f). ^{1}H NMR spectra of 4f

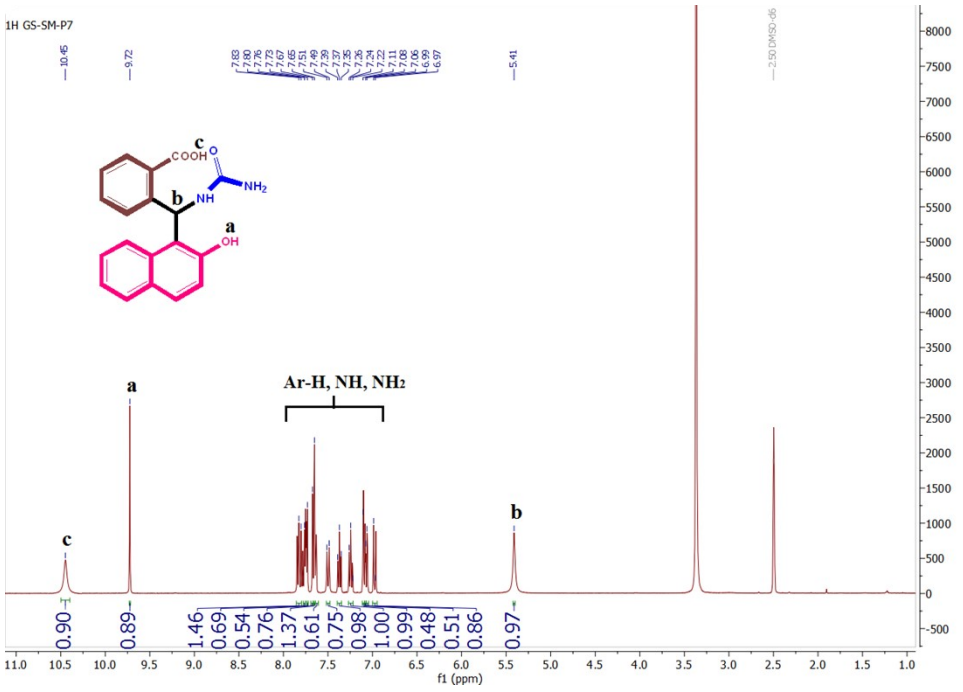

Fig.S2(g). ^{1}H NMR spectra of 4g

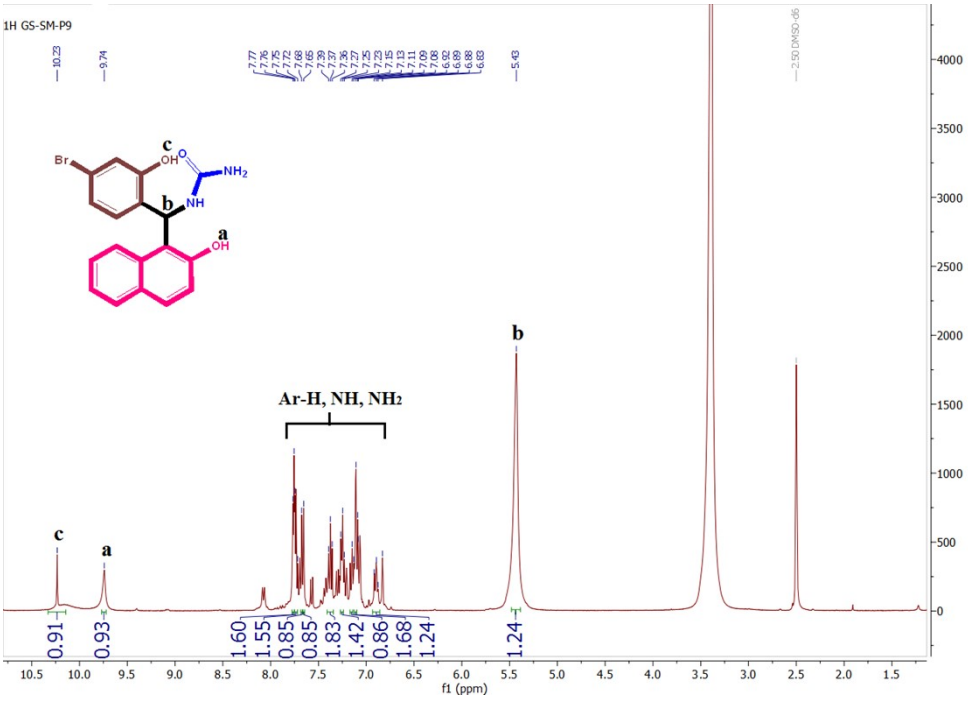

Fig.S2(h). ^{1}H NMR spectra of 4h

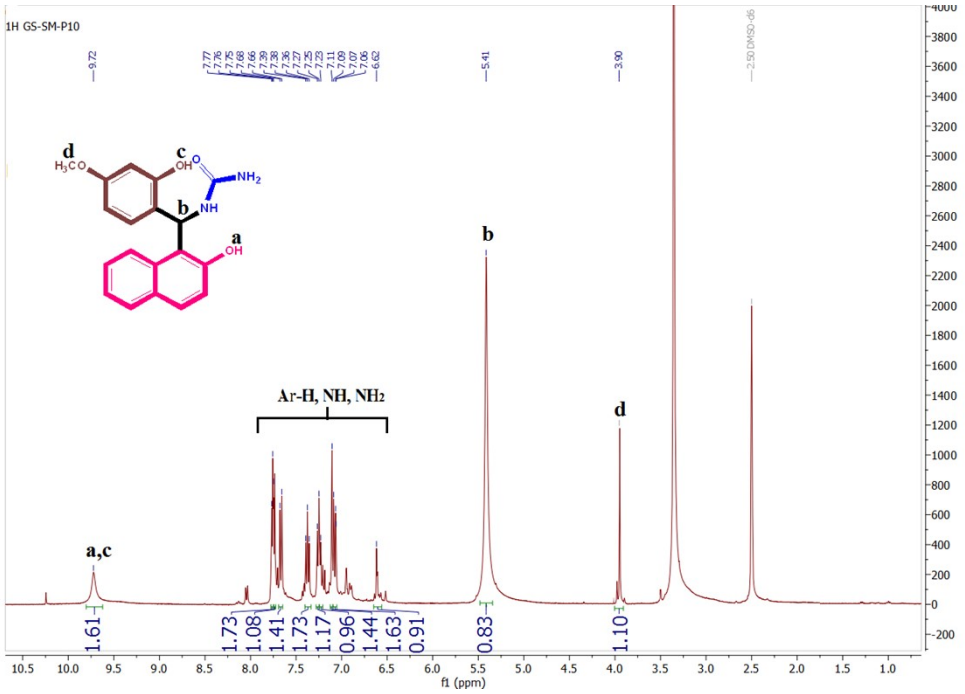

Fig.S2(i). ^{1}H NMR spectra of 4i

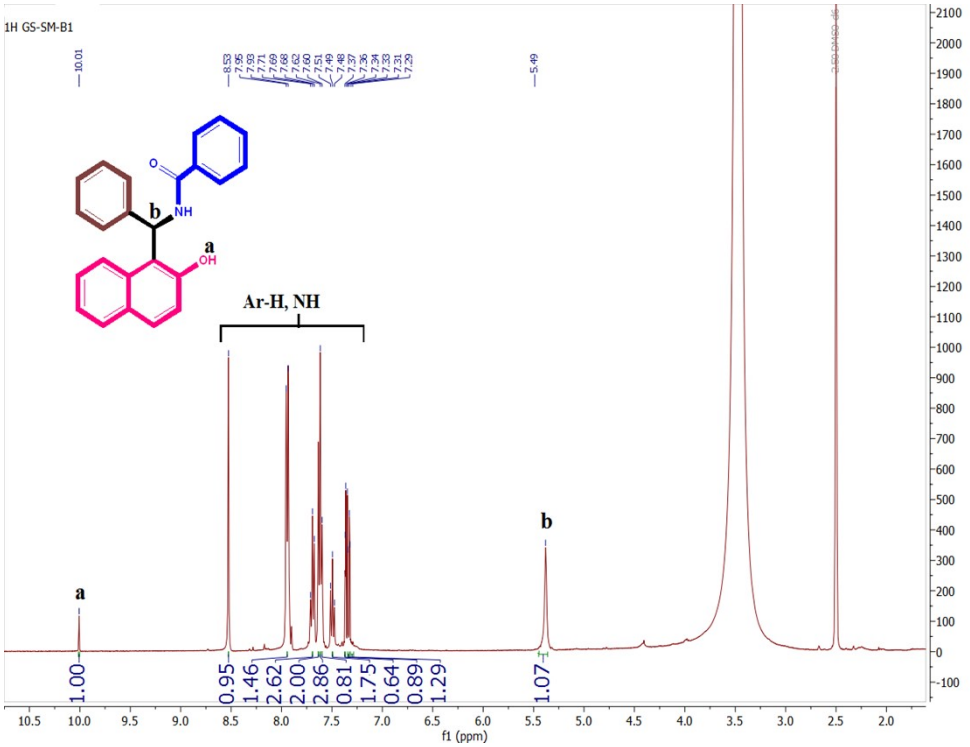

Fig.S2(j). ^{1}H NMR spectra of 4j

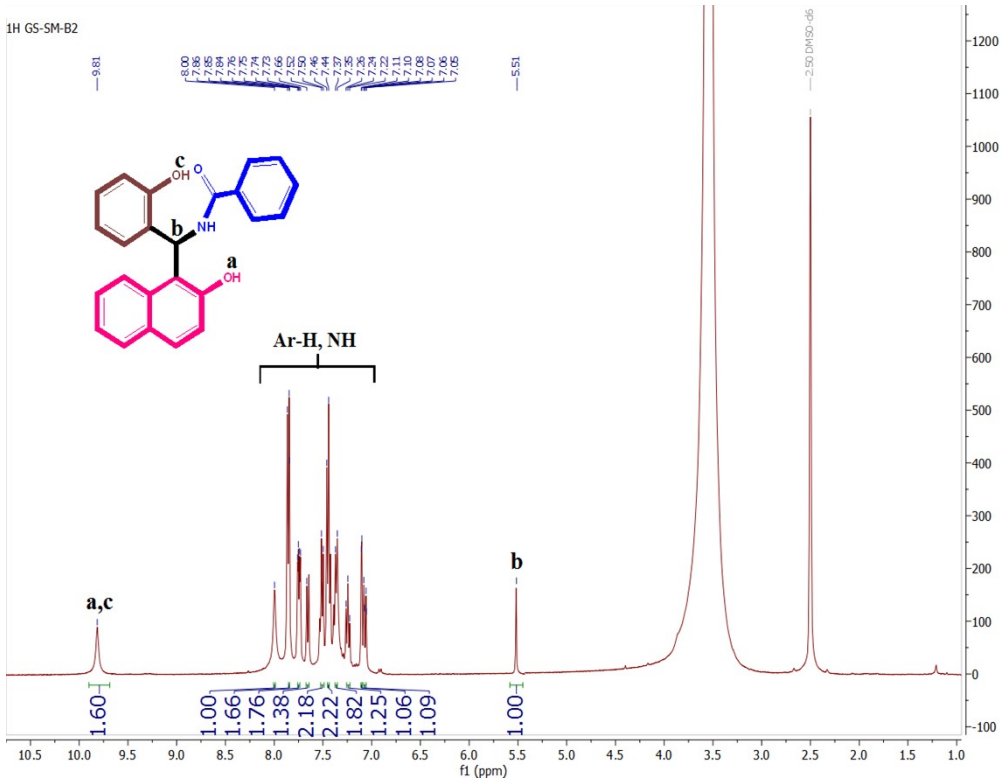

Fig.S2(k). ^{1}H NMR spectra of 4k

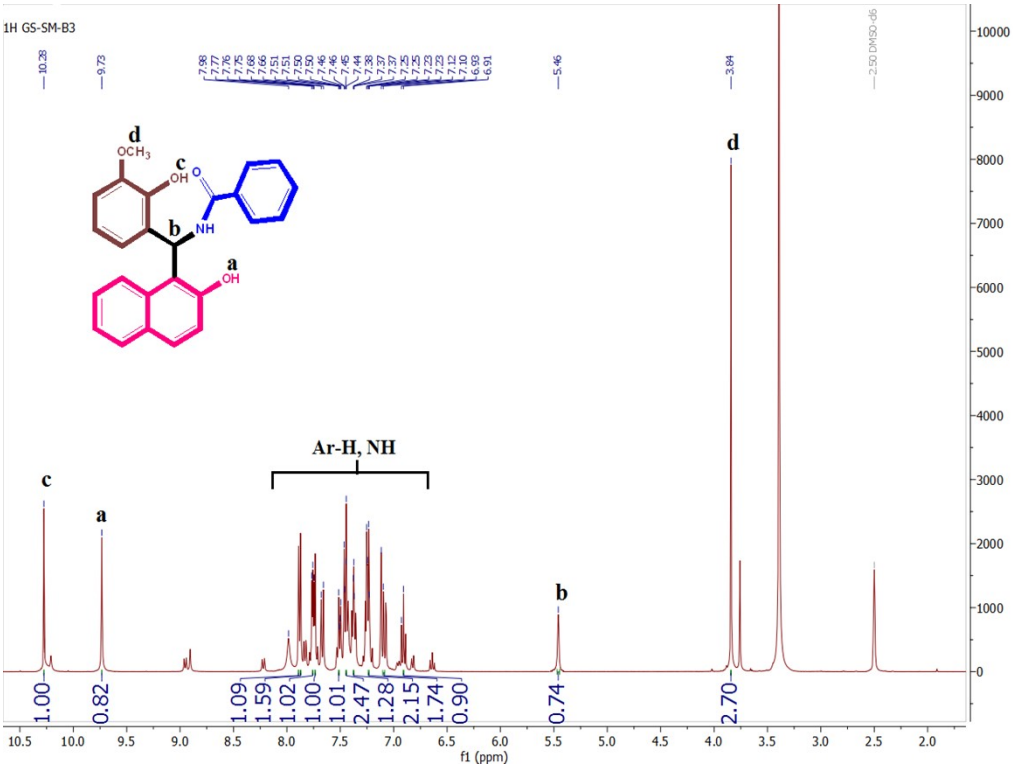

Fig.S2(l). ^{1}H NMR spectra of 4l

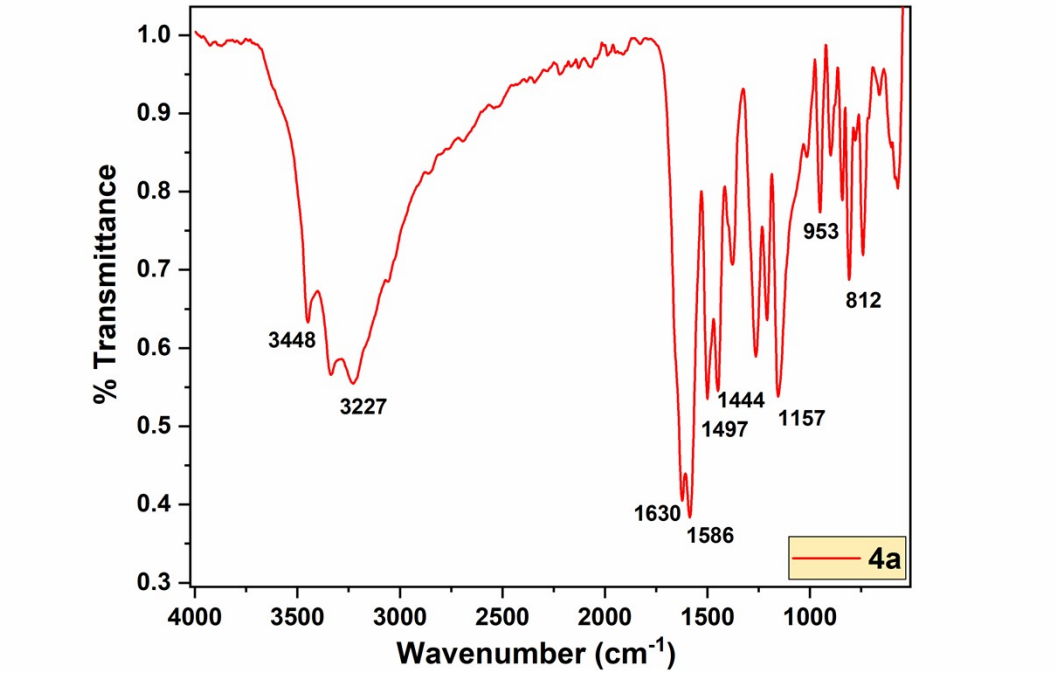

Fig.S3(a). IR spectra of 4a

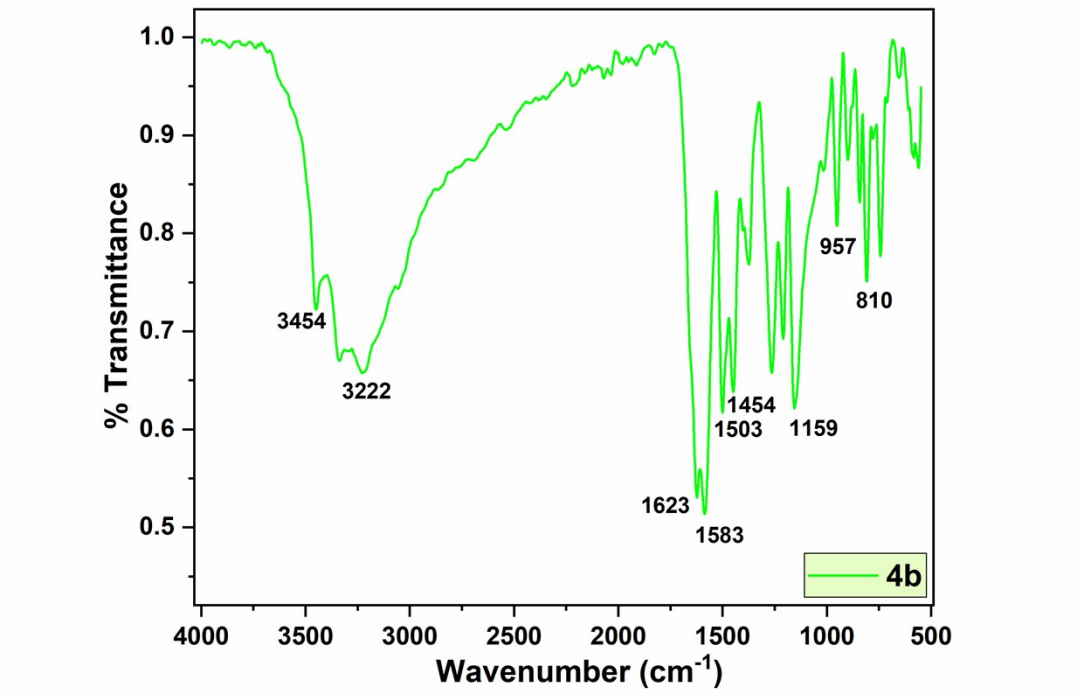

Fig.S3(b). IR spectra of 4b

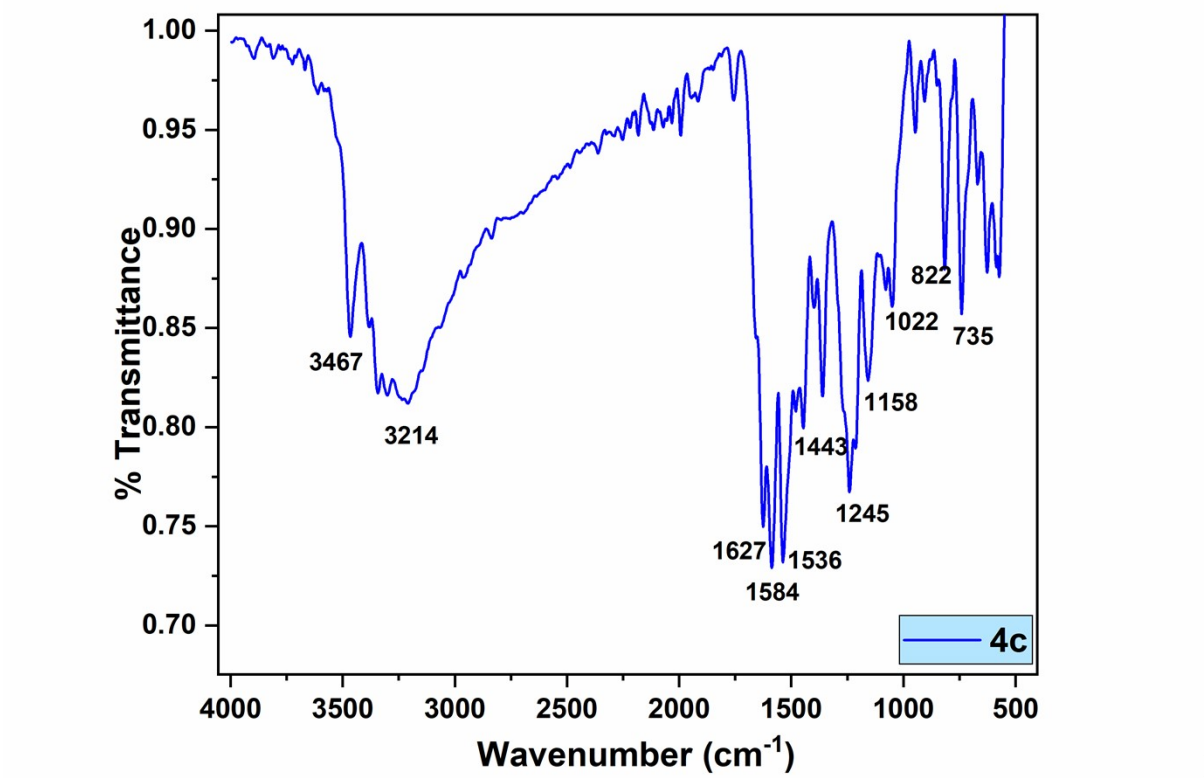

Fig.S3(c). IR spectra of 4c

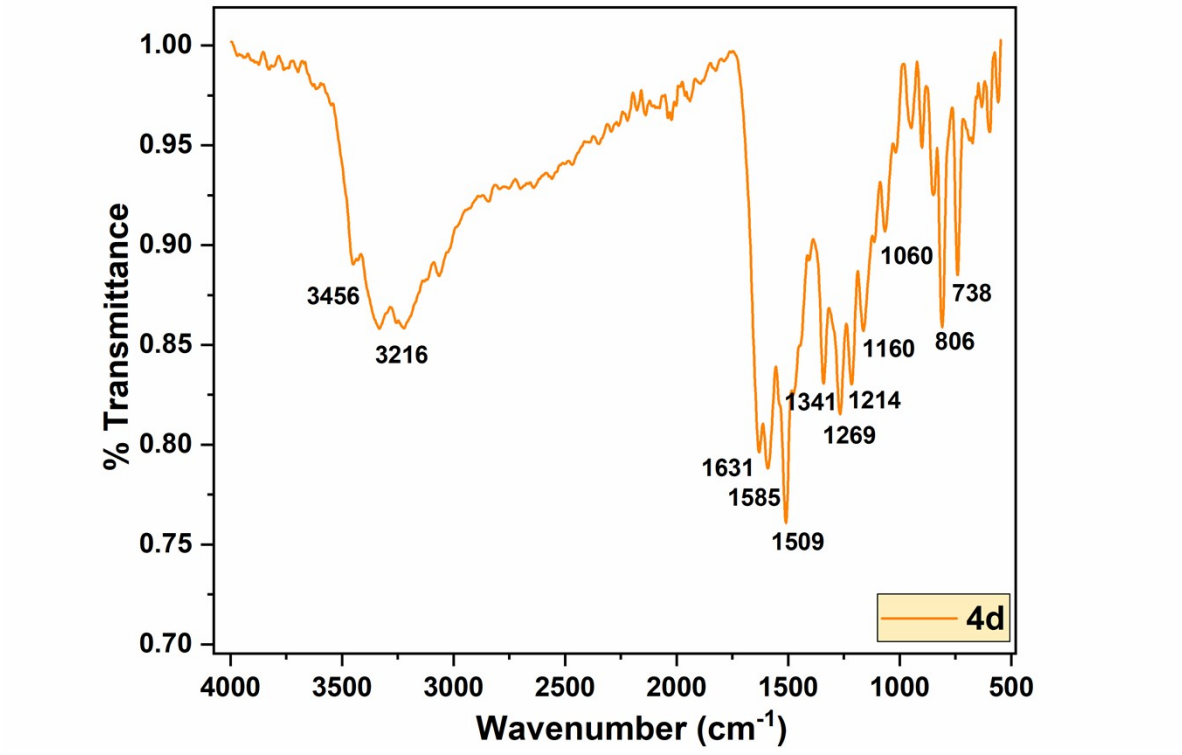

Fig.S3(d). IR spectra of 4d

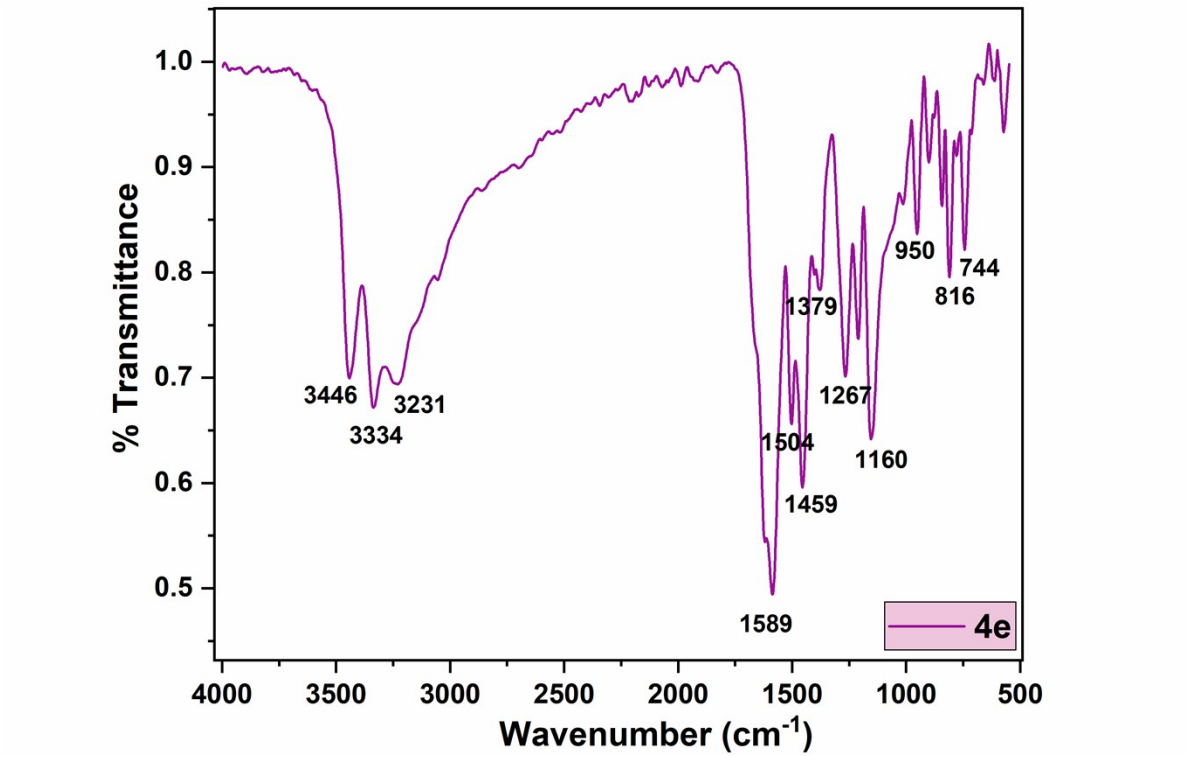
% Transmittance
Wavenumber (cm-1)
1.0
0.9
0.8
0.7
0.6
0.5
4000
3500
3000
2500
2000
1500
1000
500
3446
3334
3231
1589
1504
1459
1379
1267
1160
950
816
744
4e

Fig.S3(e). IR spectra of 4e

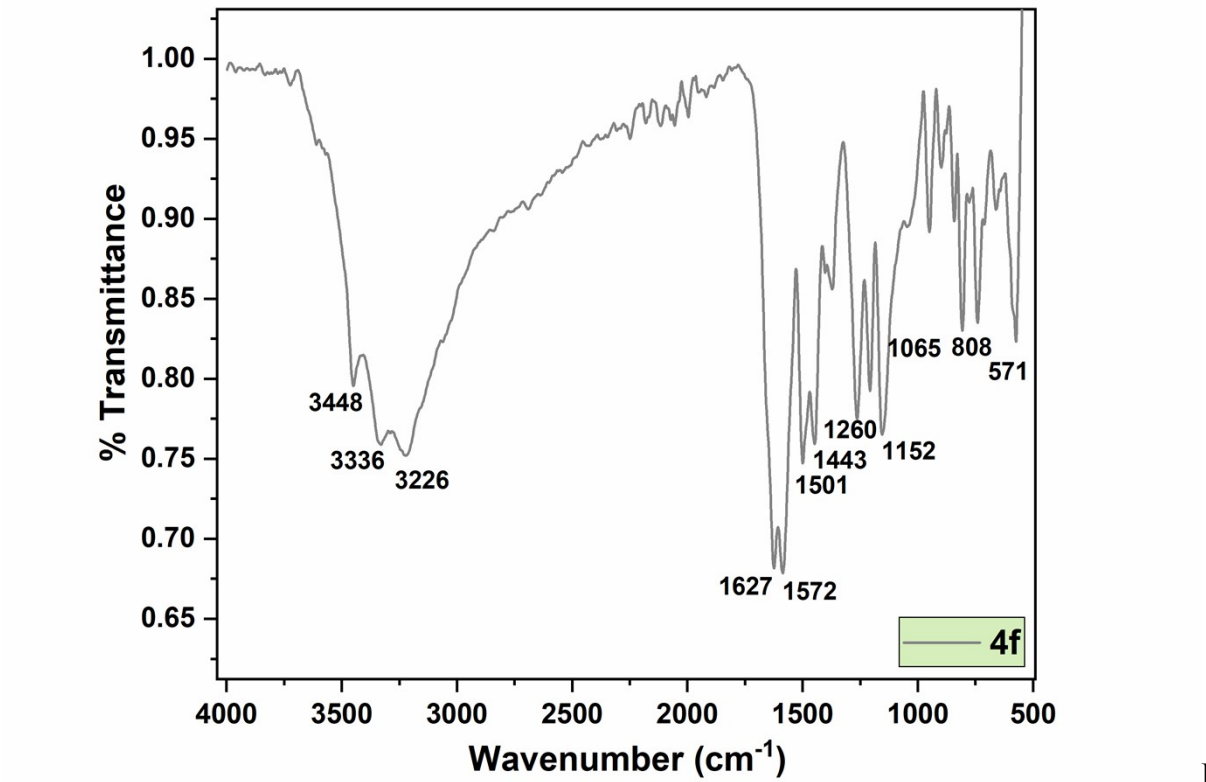

Fig.S3(f). IR spectra of 4f

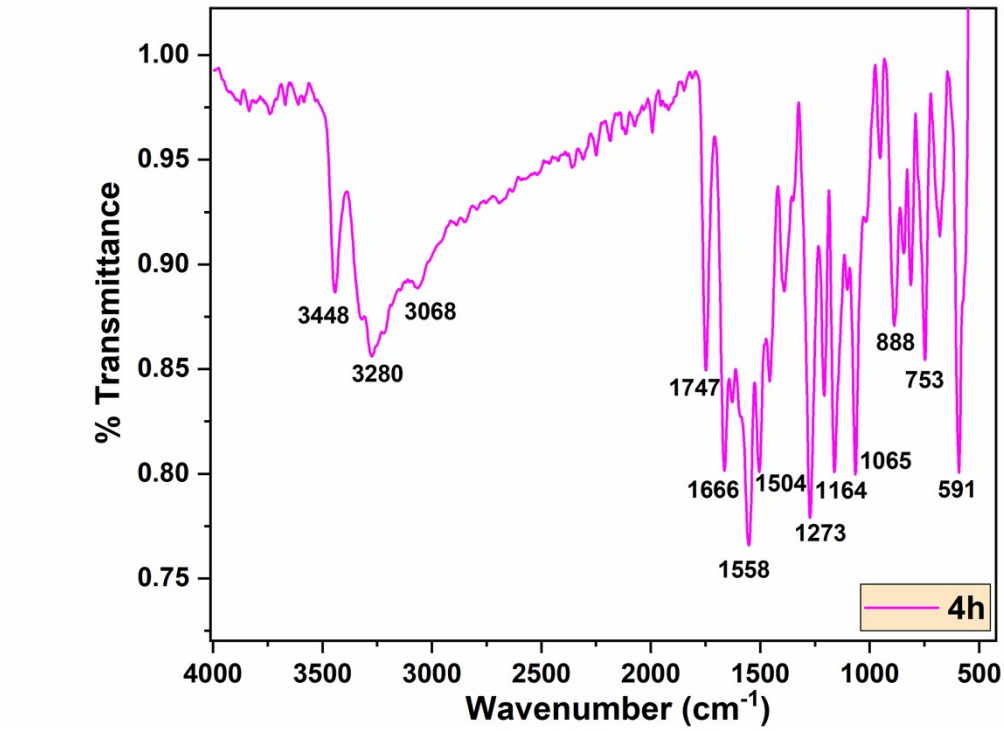

Fig.S3(g). IR spectra of 4h

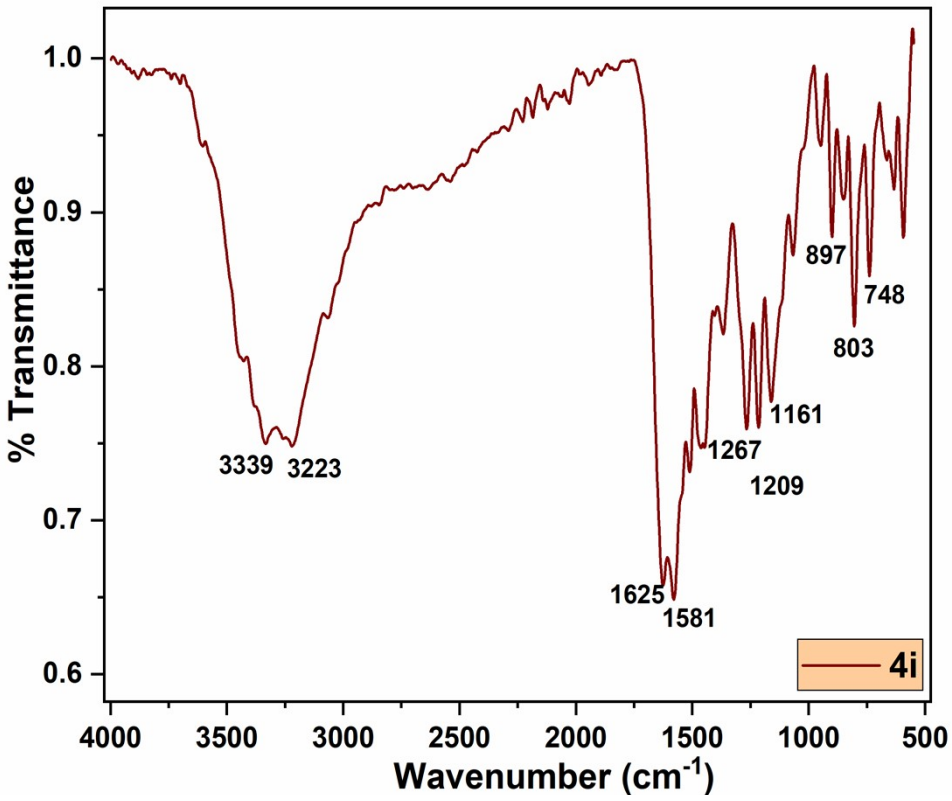

Fig.S3(h). IR spectra of 4i

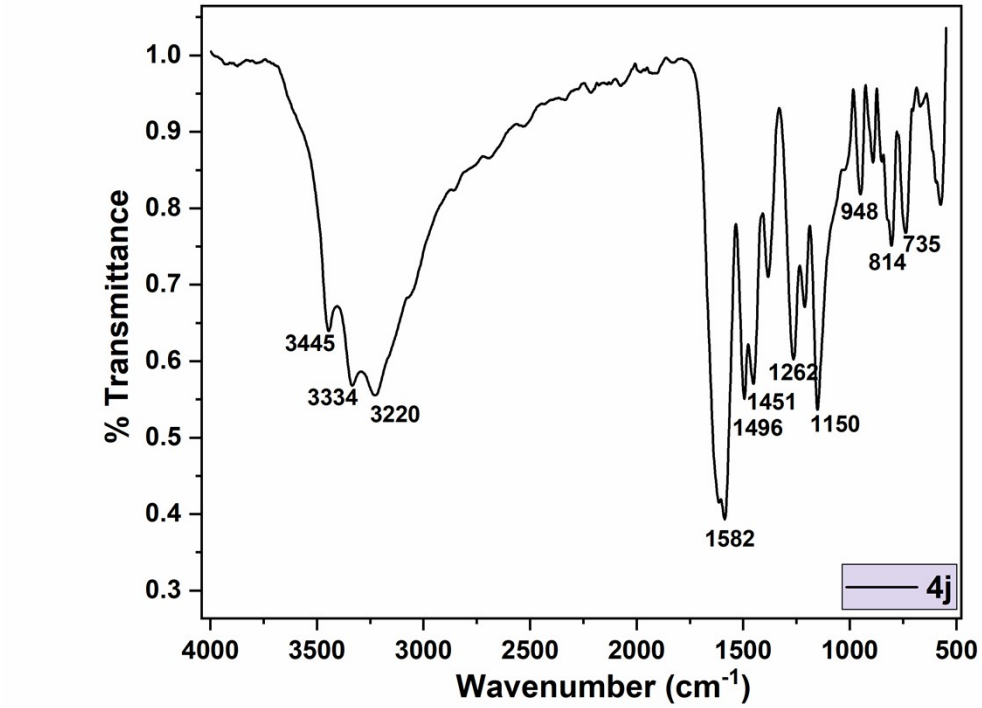

Fig.S3(i). IR spectra of 4j

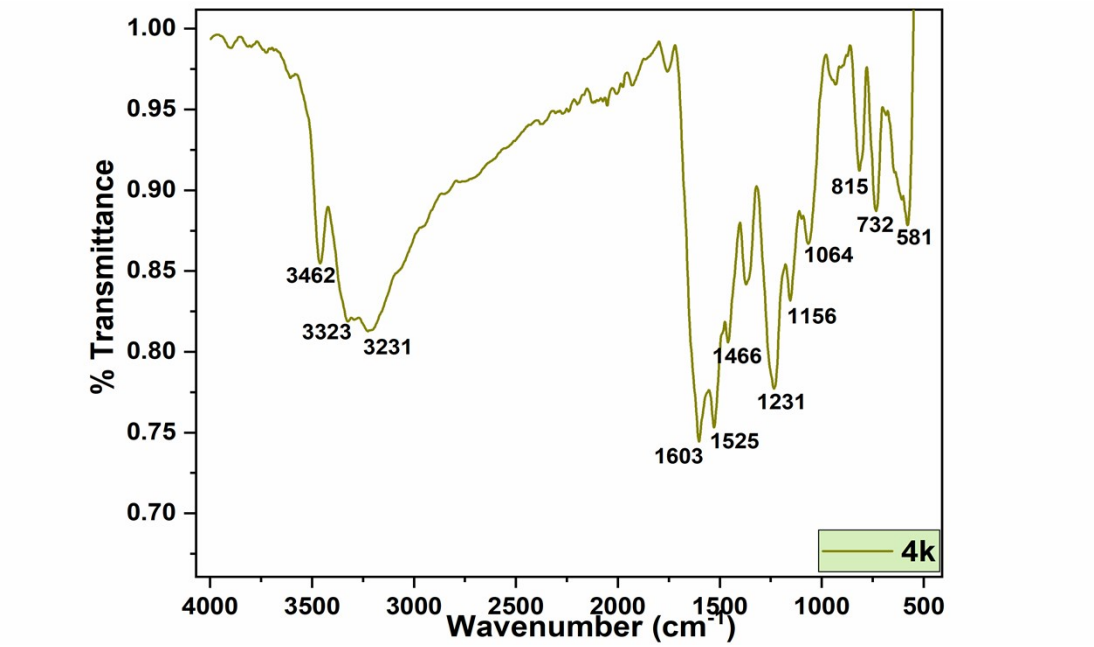

Fig.S3(j). IR spectra of 4k

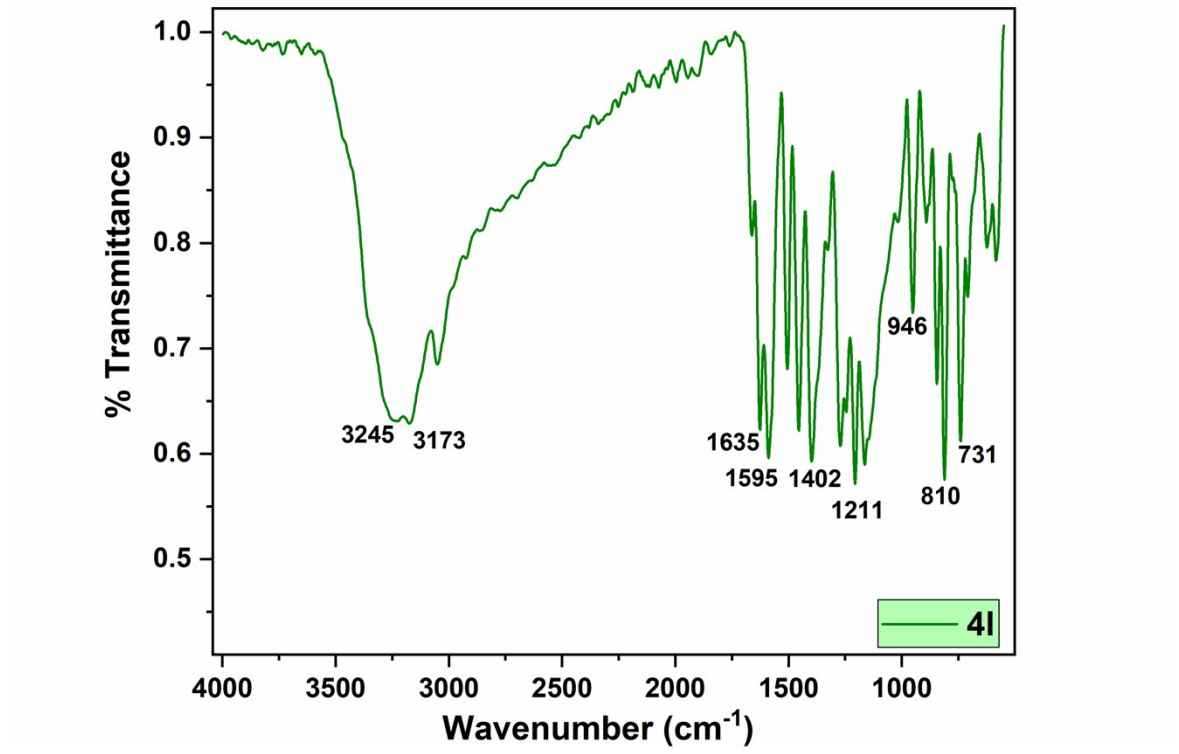

Fig.S3(k). IR spectra of 4l

Spectral analysis of the synthesized 1-amidoalkyl-2-napthol derivatives

4a. 1-(naphthalen-1-yl(phenyl)methyl)urea

Appearance- white solid, M.P: 218-220 °C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.71(s, 1H, OH), 7.06-7.77 (m, 14H, Ar-H, NH, NH_2), 5.41 (s, 1H, CH); IR (υ, cm^{-1}): 3448, 3227, 1630, 1586, 1497, 1444, 1157, 953, 812

4b. 1-((2-hydroxyphenyl)(naphthalen-1-yl)methyl)urea

Appearance- white solid, M.P: 215-217°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.71(s, 2H, 2OH), 7.07-7.76 (m, 13H, Ar-H, NH, NH_2), 5.40 (s, 1H, CH); IR (υ, cm^{-1}): 3454, 3222, 1623, 1583, 1503, 1454, 1159, 957, 810

4c. 1-((2-hydroxy-3-methoxyphenyl)(naphthalen-1-yl)methyl)urea

Appearance- yellow solid, M.P: 221-225°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.26 (s, 1H, OH), 9.81(s, 1H, OH), 6.61-8.00 (m, 12H, Ar-H, NH, NH_2), 5.97 (s, 1H, CH); 3.82 (s, 3H, OCH_3); IR (υ, cm^{-1}):3467, 3214, 1627, 1584, 1536, 1443, 1245, 1158, 1022, 822, 735

4d.1-(naphthalen-1-yl(4-nitrophenyl)methyl)urea

Appearance- brown, M.P: 200-202°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.15 (s, 1H, OH), 7.06-8.42 (m, 13H, Ar-H, NH, NH_2), 5.45 (s, 1H, CH); IR (υ, cm^{-1}):3456, 3216, 1631, 1586, 1509, 1341, 1269, 1214,1160, 1060, 806, 738

4e. 1-((4-chlorophenyl)(naphthalen-1-yl)methyl)urea

Appearance- yellow solid, M.P: 160-163°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.75 (s, 1H, OH), 7.06-8.19 (m, 13H, Ar-H, NH, NH_2), 5.42 (s, 1H, CH); IR (υ, cm^{-1}): 3446, 3334, 3231, 1589, 1504, 1459, 1379, 1267, 1160, 950, 816, 744

4f. 1-((4-methoxyphenyl)(naphthalen-1-yl)methyl)urea

Appearance-white, M.P: 205-208

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.86 (s, 1H, OH), 6.70-7.88 (m, 13H, Ar-H, NH, NH_2), 5.42 (s, 1H, CH), 3.76 (s, 3H, OCH_3); IR (υ, cm^{-1}):3448, 3336, 3226, 1627, 1572, 1501, 1443, 1260, 1152, 1065, 808, 571

4g. 2-(naphthalen-1-yl(ureido)methyl)benzoic acid

Appearance- white, M.P: 176-179°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.45(s, 1H, COOH), 9.72 (s, 1H, OH), 6.97-7.83 (m, 13H, Ar-H, NH, NH_2), 5.41 (s, 1H, CH);

4h. 1-((4-bromo-2-hydroxyphenyl)(naphthalen-1-yl)methyl)urea

Appearance- grey sticky solid, M.P: 207-209 °C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.23(s, 1H, OH), 9.74 (s, 1H, OH), 6.83-7.83 (m, 13H, Ar-H, NH, NH_2), 5.43 (s, 1H, CH).

4i. 1-((2-hydroxy-4-methoxyphenyl)(naphthalen-1-yl)methyl)urea

Appearance- yellow solid, M.P: 198-200°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.72(s, 1H, OH), 6.62-7.77 (m, 12H, Ar-H, NH, NH_2), 5.41 (s, 1H, CH), 3.90 (s, 3H, OCH_3); IR (υ, cm^{-1}):3339, 3223, 1625, 1581, 1267, 1209, 1161, 897, 803, 748

4j. N-(naphthalen-1-yl(phenyl)methyl)benzamide

Appearance- white, M.P: 223-226°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.01(s, 1H, OH), 7.29-8.53 (m, 17H, Ar-H, NH, NH_2), 5.49 (s, 1H, CH); IR (υ, cm^{-1}): 3445, 3334, 3220, 1582, 1496, 1451, 1262, 1150, 948, 814, 735

4k. N-((2-hydroxyphenyl)(naphthalen-1-yl)methyl)benzamide

Appearance- grey, M.P: 230-234°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 9.81 (s, 2H, 2OH), 7.05-8.00 (m, 16H, Ar-H, NH, NH_2), 5.51 (s, 1H, CH); IR (υ, cm^{-1}): 3462, 3323, 3231, 1603, 1525, 1466, 1231, 1156, 1064, 815, 732, 581

4l. N-((2-hydroxy-3-methoxyphenyl)(naphthalen-1-yl)methyl)benzamide

Appearance- grey, M.P: 227-230°C

^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 10.28(s, 1H, OH), 9.73(s, 1H, OH), 6.91-7.98 (m, 15H, Ar-H, NH, NH_2), 5.46 (s, 1H, CH), 3.84 (s, 3H, OCH_3); IR (υ, cm^{-1}):3245, 3173, 1635, 1595, 1402, 1211, 946, 810, 731