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ORIENTAL JOURNAL OF CHEMISTRY
An International Open Access, Peer Reviewed Research Journal

www.orientjchem.org

ISSN: 0970-020 X
CODEN: OJCHEG
2022, Vol. 38, No.(4):
Pg. 1024-1030

Synthetic Strategy to Amido alkyl naphthols from Pyrazole Aldehydes using Silica Supported $NaHSO_4.SiO_2$ as an Efficient Heterogeneous Catalyst

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http://dx.doi.org/10.13005/ojc/380426

(Received: June 13, 2022; Accepted: July 21, 2022)

AbSTRACT

A facile and eminent method has been reported for the preparation of amido alkyl naphthols. Amido alkyl naphthol derivatives were synthesized by the condensation of pyrazole aldehydes, β-naphthol andacetamidein the presence of a heterogeneous SiO_2 supported sodium hydrogen sulphate catalyst using acetic acid as solvent. This protocol is advantageous for its shorter reaction hours, simplest workup technique, excellent yields with easy recovery and reusability of the catalyst. ^1H, and ^{13}C Nuclear magnetic resonance, Fourier transform infrared and Mass Spectroscopy were utilized for the characterization of synthesized products.

Keywords: Pyrazole aldehydes, β-naphthol, Amido alkyl naphthol.

INTRODUCTION

Basically, multicomponent reactions have been reported since 1850 by Strecker[1] in α-amino acids. To avoid sequential syntheses involving many steps and to simplify synthetic routes, MCRs have been a better solution. With the help of MCRs, target molecules are synthesized in fewer steps, as reported by Ugi *et al.,*[2]. Ugi-MCRs have been proven to be an important synthetic tool in medicinal chemistry. Ugi-MCRs have greatly expanded the scope of enabling the design of diverse molecular scaffolds in modern drug discovery[3]. To improve the already known conventional MCRs and to design new bioactive structures, multiple-component routes have become the starting point. Several re-engineered MCRs have been identified by applying retrosynthetic analysis to cognate MCR[4].

To discover novel chemical reactions, in combinatorial reaction findings, to understand the structure-reactivity relationships and to generate drug like chemical products,new multi-component reactions have become the heart of organic chemistry[5]. It was an extremely beneficial gadget for the convenient design of countless chemical

compounds and reactions.In library synthesis and in Diversity-Oriented Synthesis, MCR chemistry plays a vital role[6]. The most important class of heterocycles with various biological activities were amidoalkyl naphthols. Pharmaceutical compounds with amido alkyl naphthol cores were used to treat brady cardiac, hypotensive, and cardiovascular diseases. Amido alkyl naphthols also have potential pharmaceutical activities like antipsychotic, antitumor, antirheumatic, anti-HIV, anticonvulsant, antimalarial, and antihypertensive properties. The evolution and advancement of eco-friendly technologies have become the most demanding in contemporary chemistry and chemical industry.

Literature survey considers various synthetic methods to prepare amido alkyl naphthols. Several new methods have been developed to improve the synthesize the target compound involving various catalysts such as bipyridinium sulfonic acid chloride[7], Phosphoric acid supported on alumina[8], dodecylphosphonic acid[9], ionic liquid-benzimidazolium[10], Sulfonated polynaphthalene[11], bismuth(III) nitrate pentahydrate[12], trichloroacetic acid/cobalt (II) chloride[13], $SnCl_4 \bullet 5H_2O$[14], tannic acid[15], barium phosphate nano-powder[16], boric acid[17], Magnetic nanoparticle supported acidic ionic liquid[18], nano-grapheneoxide[19] and zinc oxide nanoparticles[20] were reported. We now describe our current research related to amido alkylnaphthols synthesis using reusable eco-friendly heterogeneous silica supported $NaHSO_4.SiO_2$ catalyst with a shorter reaction time (Scheme 1).

Herein, we have investigated a potential, $NaHSO_4.SiO_2$ catalysed preparation of amidoalkyl-2-naphthols via one-pot condensation reactions involving acetic acid solvent (Scheme 1).

Ex PERIMENTAL

METHODS AND MATERIALS

Chemicals Used

The chemicals utilized for the reaction were purchased from Sigma-Aldrich U.S.A. Thin Layer Chromatography with aluminium sheets previously coated with silica gel [(Merck, F-254 from Germany) of 0.2 mm thickness] was used to monitor the reaction progress. Silica gel [(mesh size 230-400) Merck] was used to perform column chromatography.

Equipments and an alytical in struments

Bruker (300MHz and 75Hz) spectrometer using $CDCl_3$ solvent was utilized to record ^1H NMR and ^{13}C NMR. Spectrometer (4000-400 cm^{-1}) of Perkin Elmer variety,using KBr pellet was used for recording FT-IR spectrum. The HRMS spectrum was recorded using Q-T of-Mass Spectrometer.

Procedure for the synthesis of compounds 4a-4j

To a mixture of pyrazole aldehyde (1 equiv.), 2-naphthol (1 equiv.) and acetamide (1 equiv.), the hetereogeneous catalyst $NaHSO_4.SiO_2$ (0.002 g) was added. The whole content was dissolved in acetic acid solvent and stirred at 80^0C in an oil bath. The complete consumption of the starting materials was confirmed by TLC.Thenthe catalyst was recovered by filtration. The crude mixture was purified using a column chromatographic method [ethylacetate (10): petroleum ether (90)] and afforded pure amidoalkylnaphthol.

RESULT AND DISCUSSION

To synthesize N-[(1,4-diphenyl-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl)methyl] acetamide, condensation of β-naphthol, pyrazole aldehyde,acetamide was employed. The whole content of the reaction mixture was stirred in acetic acid and $NaHSO_4.SiO_2$ catalyst, in a preheated oil bath at 80^0C. The structures of the obtained products were characterized with various spectroscopic techniques like ^{1}HN-uclear magnetic resonance, ^{13}CN-uclear magnetic resonance, Fourier transform infrared and High Resolution Mass spectroscopic techniques.

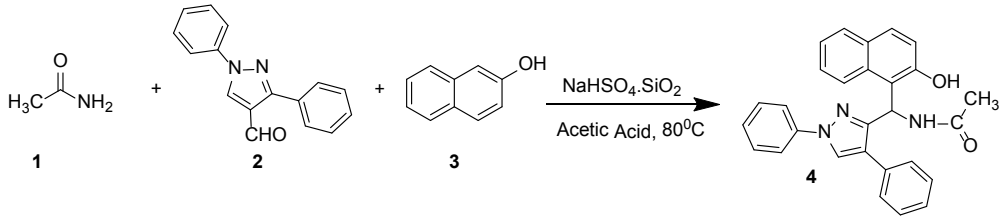
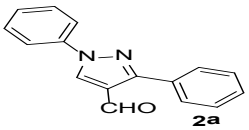
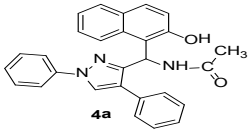
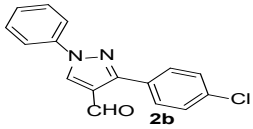
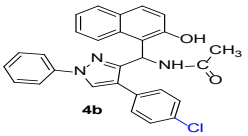
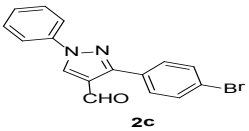
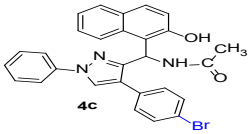
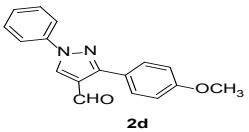
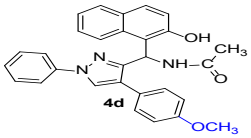
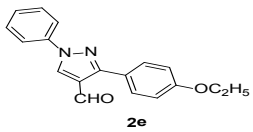
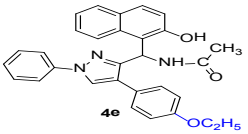
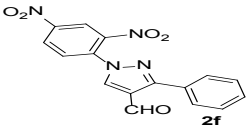
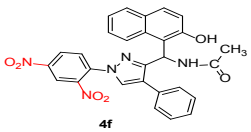
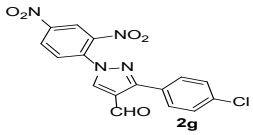
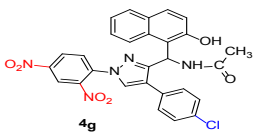
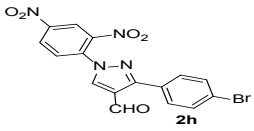
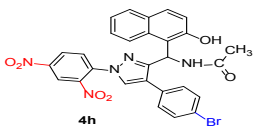
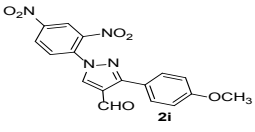
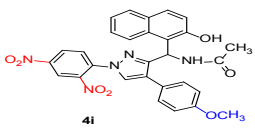
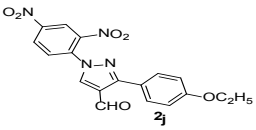
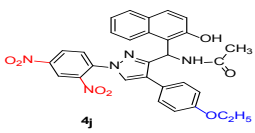


Table 1: Preparation of N-[(1,4-diphenyl-1H-pyrazol-3-yl)(2-hydroxynaphthalen-yl)methyl]acetamides

S. No	Pyrazole Aldehyde(2a-2j)	Product[a] (4a-4j)	Time (h)	Temperature(°C)	Yield[b] (%)
1	 **2a**	 **4a**	4.5	80	92
2	 **2b**	 **4b**	4.5	75	90
3	 **2c**	 **4c**	4.0	78	87
4	 **2d**	 **4d**	5.0	80	85
5	 **2e**	 **4e**	5.5	80	80
6	 **2f**	 **4f**	5.5	85	75
7	 **2g**	 **4g**	6.0	90	70
8	 **2h**	 **4h**	5.5	95	80
9	 **2i**	 **4i**	5.0	90	75
10	 **2j**	 **4j**	6.0	95	85

[a]All the purified products were characterized by ^{1}HNMR, ^{13}CNMR and HRMS spectroscopy.
[b]Confined product

Table 2: Screening of catalysts and solvents

S. No	Catalysts used	Solvents used	Time(h)	Yield(%)
1	None	EtOH	23.5	4.5
2	ZnO	Alcohol	18	30
3	Na_2SO_4	Ethanol	15	50
4	$MgSO_4$	Ethanol	15	60
5	$NaHSO_4.SiO_2$	Water	20	40
6	$NaHSO_4.SiO_2$	Ethanol	15	55
7	$NaHSO_4.SiO_2$	Acetone	10	65
8	$NaHSO_4.SiO_2$	Acetic Acid	4-6	85-90

An optimization study for the synthesis of desired compounds was undertaken in order to improve the yield of the products. Initial screening trials were performed for the optimization of certain reaction parameters like temperature and time (Table 1). The reaction was carried out using various solvents inorder to monitor the solvent effect on the product formation(Table 2). While EtOH, water and acetone were used as solvents, lower yields were observed. On using acetic acid as a solvent, the rate of reaction increased. Among the various catalysts used, $NaHSO_4.SiO_2$ was found to be the most efficient one. On conducting the reaction at 80°C using $NaHSO_4.SiO_2$ (0.002 g) catalyst, the best result was obtained. On further increase in temperature and quantity of catalyst, the yield of the product did not increase. On seeing the astonishing results of the above reaction conditions, in order to improvise the scope of the present protocol, a library of amidoalkylnaphthols was synthesized under optimized conditions (Table 2). The reaction modes of the different pyrazole aldehydes were quite similar and we got good yield of desired products while using pyrazole aldehydes with different substituents.

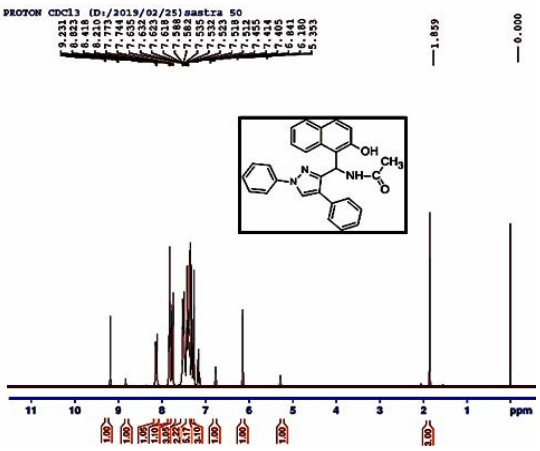


Fig. 1. ^{1}H NMR spectrum of the product 4a

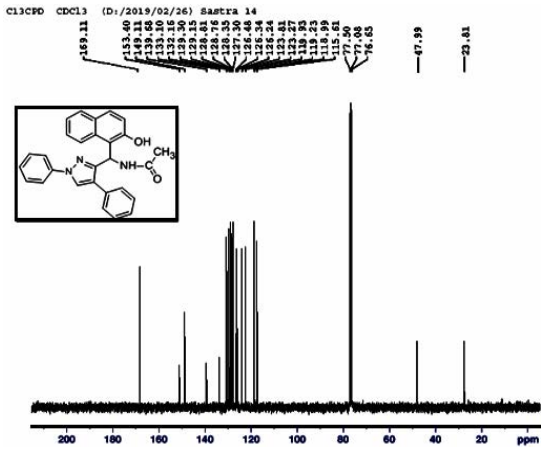


Fig. 2. ^{13}C NMR spectrum of the product 4a

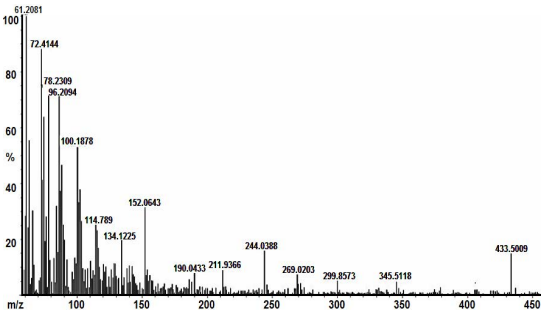


Fig. 3. HR-MS data of the product 4a

The ^{1}H NMR data of 4a showed, a three proton singlet-1.86 ppm which was attributed to methyl group protons. A singlet at 6.18 ppm was due to methine proton and the one proton singlet at 9.23 ppm was designated for aromatic alcohol. The singlet at 8.21 ppm was attributed to an N-H proton. The singlet at 8.41 ppm was attributed to the proton of the pyrazole group. Peaks from 6.84-8.82 ppm were due to protons of the naphthalene group, and peaks from 7.40-7.58 ppm were assigned to protons of the aromatic rings (Fig. 1). In the ^{13}C NMR spectrum, peaks at 23.81 ppm and

47.99 ppm were due to aliphatic carbons. The peaks ranging between 115.6-153.4 ppm were assigned to aromatic and naphthalene carbons (Fig. 2). The peak at 169.1 ppm was attributed to carbonyl carbon. The HR-MS spectrum declared the peak of molecular ion (M+) at m/z 433.5 (Fig. 3). Using elemental analysis, the formation of the product was firmly authenticated.

Characterization of synthesized compounds (4a-4j)

Compound4a:N-[(1,4-diphenyl-1H-pyrazol-3-yl) (2-hydroxynaphthalen-1-yl)methyl]acetamide;

White solid, R_f; 0.40.(20%EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ = 1.86(s, 3H), 5.35(s,1H), 6.18(s,1H), 6.84-9.23(m,6H), 7.40-7.58 (m,10H), 8.4(s,1H), 8.82(s,1H); ^{13}C NMR: (75 MHz, $CDCl_3$) δ = 23.8, 48.0, 115.6, 119.0, 119.2, 119.9, 123.3, 123.8, 126.2, 126.3, 126.5, 127.3, 128.4, 128.8, 128.8, 129.2, 129.3, 132.2, 133.1, 139.7, 149.1, 153.4, 169. 1; HR-MS (ESI) $[M]^+$ m/z: 433.50; Anal. Calcd. for $C_{28}H_{23}N_3O_2$: C, 77.55; H, 5.33; N, 9.66; O, 7.38; Found: C, 77.53; H, 5.46; N, 9.73; O, 7.32.

Compound4b:N-[(4,4-chlorophenyl)-1-phenyl-1H-pyrazol-3-yl)(2-hydroxy naphthalen-1-yl) methyl]acetamide;

Whitish solid, R_f; 0.43(20% EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ = 1.84(s,3H), 5.35(s, 1H), 6.16(s,1H), 6.85-9.21 (m,6H), 7.45-7.73 (m, 9H), 8.03(s,1H), 8.65(s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ = 23.6, 47.9, 115.4, 118.9, 119.2, 123.2, 123.8, 126.2, 126.4, 128.3, 128.8, 129.3, 130.2, 133.5, 134.3, 139.7, 149.2, 153.4, 169; HR-MS (ESI) $[M]^+$ (m/z): 467.14; Analysis, Calculated for $C_{28}H_{22}ClN_3O_2$: C, 71.87; H, 4.74; Cl, 7.58; N, 8.98; O, 6.84; Found: C, 71.77; H, 4.64; Cl, 7.68; N, 8.88; O, 6.85.

Compound4c: N-[(4,4-bromophenyl)-1-phenyl-1H-pyrazol-3-yl)(2-hydroxy naphthalen-1-yl) methyl]acetamide;

Brownishsolid, R_f; 0.43 (20% EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ ppm = 1.84 (s, 3H), 5.35(s,1H), 6.16(s,1H), 6.85-9.21(m,6H), 7.45-7.66 (m,9H), 8.03(s,1H), 8.65(s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ ppm = 23.6, 47.9, 115.4, 118.9, 119.2, 123.2, 123.8, 126.2, 126.4, 128.3, 128.8, 129.3, 130.2, 133.5,134.3, 139.7, 149.2, 153.4, 169; HR-MS (ESI) $[M]^+$ (m/z): 511.09; Anal. Calcd. for $C_{28}H_{22}BrN_3O_2$: C, 65.63; H, 4.33; Br, 15.59; N, 8.20; O, 6.24; Found C, 66.01; H, 4.27; Br, 15.68 N, 8.28; O, 6.25.

Compound4d: N-[(2-hydroxynaphthalen-1-yl) (4-(4-methoxyphenyl)-1-phenyl-1H-pyrazol-3-yl) methyl]acetamide;

Brown solid, R_f; 0.38(20%EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δppm = 1.84 (s,3H), 3.83(s,3H), 5.35(s,1H), 6.16(s,1H), 6.85-9.21 (m,6H), 7.05-7.68 (m,9H), 8.03 (s,1H), 8.65 (s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ ppm = 23.6, 47.9, 55.8, 114.8, 115.4, 118.9, 119.2, 119.9, 123.2, 123.8, 124.4, 126.2, 126.3, 126.4, 128.3, 128.8, 129.3, 133.5, 139.7, 153.4, 160.6, 169.0; HR-MS(ESI) $[M]^+$ (m/z): 463.19; Elemental Analysis, Calculated for $C_{29}H_{25}N_3O_3$:C, 75.14; H, 5.44; N, 9.07; O, 10.35; Found: C, 75.13; H, 5.34; N, 9.08; O, 10.38.

Compound4e: N-[(4,4-ethoxyphenyl)-1-phenyl-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl) methyl]acetamide;

Yellowish white solid, R_f ; 0.36 (20% EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ ppm = 1.31 (s,3H), 1.84 (s,3H), 4.09 (s, 2H), 5.35(s, 1H), 6.16(s,1H), 6.85-9.21 (m,6H), 7.05-7.68 (m,9H), 8.03 (s,1H), 8.65 (s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ = 14.8, 23.6, 115.4, 47.9, 64.6, 114.9, 115.4, 118.9, 119.2, 119.9, 123.2, 123.7, 123.8, 126.2, 126.3, 126.4, 128.3, 128.8, 129.3, 133.5, 139.7, 149.2, 153.4, 159.4, 169.0; HRMS (ESI) $[M]^+$ m/z : 477.21; Elemental Analysis, Calculated for $C_{30}H_{27}N_3O_3$: C, 75.45; H, 5.70; N, 8.80; O, 10.05; Found: C, 75.77; H, 5.67; N, 8.82; O, 10.05.

Compound4f: N-[(1-(2,4-dinitrophenyl)-4-phenyl-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl) methyl]acetamide;

Yellow solid, R_f; 0.48(20% EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ ppm = 1.85 (s,3H), 5.35 (s,1H), 6.16 (s,1H), 6.85-9.21 (m,6H), 7.41-8.92 (m,8H), 8.03 (s,1H), 8.65 (s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ = 23.6, 47.9, 115.4, 118.9, 119.2, 120.6, 123.2, 123.8, 124.7, 126.3, 126.4, 127.5, 127.6, 128.3, 128.7, 128.8, 129.2, 132.1, 133.5, 136.1, 142.1, 146.3, 149.2, 153.4, 169; HR-MS (ESI) $[M]^+$ (m/z): 523.16; Elemental Analysis, Calculated for $C_{28}H_{21}N_5O_6$: C,64.24; H, 4.04; N, 13.38; O, 18.34; Found C, 63.89; H, 4.02; N, 13.41; O, 18.29.

Compound4g: N-[(4-(4-chlorophenyl)-1-(2,4-dinitrophenyl)-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl)methyl]acetamide;

Yellow solid, R_f; 0.52 (20% EAPE): ^{1}H NMR (300MHZ, $CDCl_3$) δ ppm = 1.84(s,3H), 5.35(s,1H),

6.16(s,1H), 6.85-9.21(m,5H), 7.41-8.92(m,8H), 8.03(s,1H), 8.65(s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ ppm = 23.6, 47.9, 115.4, 118.9, 119.2, 123.2, 123.8, 124.7, 126.3, 126.4, 127.6, 128.3, 128.8, 128.9, 129.3, 130.2, 133.5, 134.3, 136.1, 142.1, 146.3, 149.2, 153.4, 169; HR-MS (ESI) $[M]^+$ (m/z): 557.11; Elemental Analysis, Calculated for $C_{28}H_{20}ClN_5O_8$: C, 60.28; H, 3.61; Cl, 6.35; N, 12.55; O, 17.21; Found: C,60.31; H, 3.59; Cl, 6.36 N, 12.58; O, 17.19.

Compound4h: N-[(4-(4-bromophenyl)-1-(2,4-dinitrophenyl)-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl)methyl]acetamide;

Brown solid, R_f ;0.58 (20% EAPE): ^{1}H NMR (300MHZ, $CDCl_3$) δppm = 1.85(s,3H), 5.33(s,1H), 6.16(s,1H), 6.85-9.21(m,6H), 7.53-8.92(m,7H), 8.03(s,1H), 8.65(s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$) δ = 23.6, 47.9, 115.4, 118.9, 119.2, 120.6, 121.7, 123.1, 123.2, 123.8, 124.7, 126.3, 126.4, 127.6, 128.3, 128.8, 129.7, 131.1, 133.5, 136.1, 142.1, 146.3, 149.2, 153.4, 169; HR-MS(ESI) $[M]^+$ (m/z): 601.06; Elemental Analysis, Calculated for $C_{28}H_{20}BrN_5O_6$: C, 55.83; H, 3.35; Br, 13.26; N, 11.63; O, 15.94; Found C, 55.84; H, 3.38; Br, 13.31; N, 11.71; O, 15.89.

Compound4i: N-[(1-(2,4-dinitrophenyl)-4-(4-methoxyphenyl-1H pyrazol-3-yl)(2-hydroxynaphthalen-1-yl)methyl]acetamide;

White solid, R_f; 0.50 (20% EAPE): ^{1}H NMR (300MHZ, $CDCl_3$) δppm = 1.84 (s,3H), 3.83 (s,3H), 5.35(s,1H), 6.16(s,1H), 6.85-9.21(m,6H), 7.05-8.92(m,7H), 8.03(s,1H), 8.65(s,1H);^{13}C NMR: (75MHZ, $CDCl_3$) δppm = 23.6, 47.9, 55.8, 114.8, 115.4, 118.9, 119.2, 120.6, 123.2, 123.8, 124.4, 124.7, 126.2, 126.3, 126.4, 127.6, 128.3, 128.8, 133.5, 136.1, 142.1, 146.3, 149.2, 153.4, 160.6, 169; HR-MS (ESI) $[M]^+$ (m/z): 553.16; Elemental Analysis, Calculated for $C_{29}H_{23}N_5O_7$: C, 62.93; H, 4.19; N, 12.65; O, 20.23; Found C, 62.89; H, 4.21; N, 12.71; O, 20.31.

Compound4j: N-[(1-(2,4-dinitrophenyl)-4-(4-ethoxyphenyl-1H-pyrazol-3-yl)(2-hydroxynaphthalen-1-yl)methyl]acetamide;

White solid, R_f; 0.46 (20% EAPE); ^{1}H NMR (300MHZ, $CDCl_3$) δ ppm = 1.84(s,3H), 5.35(s,1H), 6.16(s,1H), 6.85-9.21(m,6H), 7.05-8.92(m,7H), 8.03(s,1H), 8.65(s,1H); ^{13}C NMR: (75MHZ, $CDCl_3$)δ ppm = 14.8, 23.6, 47.9, 64.6, 114.9, 115.4, 118.9, 119.2, 120.6, 123.2, 123.7, 123.8, 124.7, 125.8, 126.3, 126.4, 127.6, 128.3, 128.8, 133.5, 136.1, 142.1, 146.3, 149.2, 153.4, 159.4, 169; HR-MS(ESI) $[M]^+$ (m/z): 567.18; Elemental Analysis, Calculated for $C_{30}H_{25}N_5O_7$: C, 63.49; H, 4.44; N, 12.34; O, 19.73; Found: C, 63.51; H, 4.43; N, 12.44; O,19.68.

CONCLUSION

We have successfully determined that silica supported sodium hydrogen sulphate is a dynamic and ecofriendly green catalyst for the synthesis of N-[(1,4-diphenyl-1H-pyrazol-3-yl) (2-hydroxynaphthalen-1-yl)methyl]acetamide. The derivatives of synthesized compounds (4a-4j) were obtained via domino reaction of pyrazole aldehydes, 2-naphthol, acetamide using acetic acid as solvent with heterogeneous $NaHSO_4.SiO_2$ as a catalyst at 80^0C in excellent yields. The results were upgraded by catalyst screening and solvent screening (Table 2).The primary aim of the present study is to develop a highly effic ent, cost-effective, and environmentally benign catalyst. This methodology involves conveniently obtainable solvents, a clean process, and precise reaction time. We have done the structural confirma ion with various spectral characterizations of the synthesized compounds. The assessments of the biological activities of the synthesized products are under study.

ACKNOWLEDGEMENT

The Authors Thanks (CDST-FIST) Department of chemistry, A.V.V.M. SPC for recording FTIR, C.L.R.I, Adayar, Chennai, thanks to Sastra University, Thanjavur for recording NMR spectroscopy, IIT Madras for recording HRMS.

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