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SYNTHESIS OF NOVEL PYRAZOLO PYRANOPYRIMIDINE DERIVATIVES USING (TEDA-SIO_2) IN AQUOUS MEDIA.

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ABSTRACT: Novel pyrazolo pyranopyrimidine derivatives containing both pyranopyrimidine and pyranopyrazole moieties having significant pharmacological activities are synthesized with the help of eco-friendly one pot four component reaction involving Ethyl acetoacetate, Phenyl hydrazine, Barbituric acid and substituted Aromatic aldehyde by using (TEDA-SiO_2) as an efficient and mild catalyst in aquous media It gives excellent yield over short reaction time and simple work-up procedure.

Keywords: Ethylacetoacetate, Aromatic aldehydes, Barbituric acid, TEDA-SiO_2, Water, MCRs.

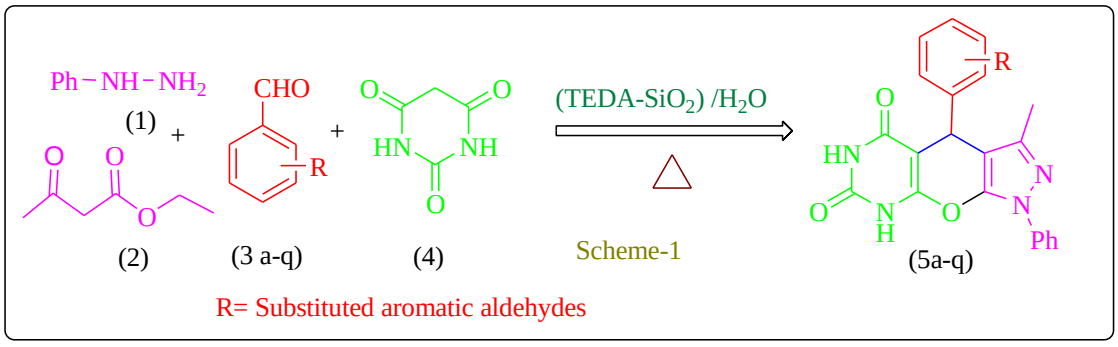
1. INTRODUCTION

The development of simple, efficient, environmentally benign and economically profitable process in organic synthesis is in great demand.Inregards the view of green approach multicomponent reactions (MCRs) have been refined as powerful and useful tool for the synthesis of novel organic molecules from readily available starting material. This reactions reduces the consumption of solvent, catalyst, time, energy, minimizing waste, cost compared to the corresponding series of individual reactions.[1–4]

Fused pyrazolo pyranopyrimidines containing both biologically active pyranopyrazole and pyranopyrimidine with wide spectrum of biological and pharmacological activities.[5-6] Among these pyranopyrazole is an important class of biologically active heterocycles. They are reported to possess a multiplicity of pharmacological properties including antimicrobial,[7-8] anticancer,[9-10] anti-inflammatory,[11-12] inhibitors of human Chk1 kinase[13] and also as

biodegradable agrochemicals.[14-15] Furthermore they play a significant role as crucial synthetic intermediates,[16] that find applications as pharmaceutical ingredients and biodegradable agrochemicals.[18] On the other hand, pyranopyrimidine scaffold, as a key component of the pyrimidine family, has received considerable attention because of wide range of antitumor,[19-20] antibronchitic,[21] hepatoprotective,[22] pronounced antitubercular and antimicrobial[23] activities. Pyrano pyrazole and pyrano pyrimidine heterocyclic systems hold a prominent position as they constitute an integral part of natural alkaloids and synthetic drugs.[24-25]

However a more pronounced effect is generally observed when two or more different heterocyclic moieties exist in a single molecule because it might possess the properties of all moieties and its pharmacological activities are thus enhanced. Therefore, synthesis of such compounds has attracted strong interest. A four-component Condensation of aldehydes, barbituric acid, ethyl acetoacetate, and hydrazine hydrate is one of the most prominent existing procedures used for the synthesis of pyrazolo pyranopyrimidines. Very few reports are available in the literature for the synthesis of pyrazolo pyrano pyrimidines, such as DABCO,[26] Nano-ZnO,[27] Halloysite claynanotubes,[28] silicabonded (SBDBU+Cl), (SBDABCO+Cl), (NSB-DBU+Cl),[29] Meglumine,[30] Magnitized water,[31] ChCl:Urea,[32] most of these protocols are having scope for further improvements. Owing to the harmful effects of volatile organic solvents on the environment, many green solvent systems have been recently introduced as alternative reaction media. Water is the most amazing gift of nature. It has gained a noteworthy eminence as a green medium in organic synthesis due to it being safe, cheap, eco-friendly, and non-toxic. However, catalyst used(DABCO-SiO_2)DABCO (1,4-diazabicyclo[2.2.2]octane),also known as triethylenediamine or TEDA, is a bicyclic organic compound . This colorless solid is a highly nucleophilic tertiary amine base, which is used as a catalyst and reagent in polymerization and organic synthesis acts as catalyst and it perform much organic transformation under mild condition. Hence, there is a vital need to develop a glowingly organized, simple and yield elevating protocol for the synthesis of pyrazolo pyranopyrimidines derivatives. As a part of our continual efforts toward the development of efficient, economical and new methods using green catalysts and solvents, we investigated the activity of the readily available, renewable, recyclable and environmentally benign DABCO as catalyst for the synthesis of pyrazolo pyrano pyrimidines derivatives.



2.0 MATERIAL AND METHODS

To a solution of Phenyl hydrazine (1.0 mmol) in H_2O (2 ml) Ethyl acetoacetate was added (1.0 mmol) and stirred for15 min. Then, an appropriate amount of Aromatic aldehyde (1.0 mmol) and Barbituric acid (1.0 mmol) was

refluxed using (DABCO-SiO_2)or (TEDA-SiO_2) (10 mol %) as catalyst for one hour. Progress of the reaction was monitored by TLC. Solid precipitate formed was filtered, washed with water and recrystallized from ethanol to give **(5a-q).** These synthesized products **(5a-q)** were completely characterized from IR, ^{1}H-NMR, Mass and ^{13} C-NMR spectroscopic technique and also elemental analysis.

2.1 EXPERIMENTAL

Melting points of synthesized compounds were determined by open capillary tubes and uncorrected. Purity of all the products was routinely checked by thin layer chromatography (TLC) on pre-coated sheets of silica gel-C (Merck 60 F_{254}) plates of 0.25 mm thickness using UV Chamber for detection. Perkin-Elmer FT-IR spectra were recorded in KBr pallets on infrared spectrophotometer. Bruckner advance spectrophotometer 300 or 400 MHz was used to record ^{1}H and ^{13}C-NMR spectra in DMSO-d_6 using TMS as internal standard. Mass spectra were recorded on FT-VC-7070H Mass spectrometer using the EI technique at 70 eV.

2.2SPECTRAL ANALYSIS

4-(4-hydroxyphenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyrimidine-5,7 (1*H*,4*H*)-dione(5a): IR (KBr / cm^{-1}) 3460 (-OH), 3420 (N-H stretching), 3195 (Ar-H stretching), 2949 (C–H stretching), 1665 (C═O stretching), 1590 (C═N stretching), 1252 (C–O–C stretching); ^{1}HNMR (300MHz, DMSOd_6/ppm) δ 2.32 (3H,s,CH_3), 5.07 (1H,s,pyran), 5.35 (1H,s,OH), 6.0 (1H,s,NH), 6.67 (2H,dd), 7.14-7.24 (3H,dd), 7.19 (1H,t), 7.41(2H,dd), 7.75 (2H,t), 10.0 (1H,s,NH); ^{13}CNMR (300MHz,DMSO d_6/ppm) δ: 37, 115, 128 , 129, 137 , 140 , 145 , 152 ,157, 161 ,163; EI-MS (m/z: RA %) : 388 (100.0%); Elemental Analysis (Calculated): C, 64.94; H, 4.15; N, 14.43. Elemental Analysis (Found): C, 64.91; H, 4.16; N, 14.42.

4-(3-bromophenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyrimidine-5,7 (1*H*,4*H*)-dione(5b): IR (KBr/cm^{-1}) 3420 (N-H stretching), 3195 (Ar-H stretching.) 2949 (C-H stretching), 1665 (C═O stretching), 1590 (C═N stretching), 1252 (C–O-C stretching), ^{1}HNMR (300MHz, DMSOd_6/ppm) δ 2.32 (3H,s,CH_3), 5.25 (1H,s,Pyran), 6.0 (1H,s,NH), 6.99 (1H,d), 7.19 (1H,d), 7.23 (1H,d),7.27 (1H,d), 7.29 (1H,d), 7.4 (2H,t), 7.75 (2H,d), 10.9 (1H,s,NH); ^{13}CNMR (300MHz, DMSOd_6/ppm) δ: 37, 104 , 106, 127,129, 130, 141, 152 ,161 ; EI-MS (m/z: RA %): 450 (100.0%) ; Elemental Analysis (Calculated): C, 55.89; H, 3.35; N, 12.42. Elemental Analysis (Found): C, 55.88; H, 3.37; N, 12.45.

4-(3,4-dimethoxyphenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyrimi dine-5,7(1*H*,4*H*)-dione.(5*d*): IR (KBr/cm^{-1}) 3420 (N-H stretching), 3195 (Ar-H stretching), 2940 (C-H stretching), 1590 (C═N stretching), 1660 (C═O stretching,1248 (C–O–C stretching); ^{1}HNMR (300MHz, DMSOd_6/ppm) δ 1.95 (3H,s,CH3), 3.83 (6H,s,2-OCH3), 5.15 (1H,s,pyran), 6.0 (1H,s,NH) , 6.69-6.76 (2H,dd), 6.95 (1H,dd), 7.19 (1H,d), 7.41 (2H,t), 7.75 (2H,d), 10.0 (1H,s,NH); ^{13}CNMR (300MHz, DMSOd_6/ppm): 13, 37, 106, 116, 129, 124, 137, 140, 146,152,163; EI-MS (m/z: RA %):432 (100.0%). Elemental Analysis (Calculated): C, 63.88; H, 4.66; N, 12.96.Elemental Analysis (Found): C, 63.90; H, 4.67; N, 12.98.

3-methyl-4-(4-nitrophenyl)-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyrimidine-5,7 (1*H*,4*H*)-dione.(5f): IR (KBr/cm^{-1}) 3420 (N-H stretching), 3195 (Ar-H stretching), 2949 (C-H stretching), 1665 (C═O stretching), 1595 (C═N stretching), 1260 (C–O–C stretching);^{1}HNMR (300MHz,DMSOd_6/ppm) δ 2.32 (3H,s), 5.40 (1H,s), 6.0 (1H,s,NH), 7.19 (1H,t), 7.36 (2H,t), 7.41 (2H,d), 7.45 (2H,d), 7.75 (2H,d), 8.06 (2H,d), 10.0 (1H,s,NH); ^{13}CNMR (300MHz, DMSOd_6 /ppm) δ: 13, 37,106, 117, 122, 129, 137, 140, 145, 152 , 163; EI-MS (m/z: RA %):417 (100.0%);Elemental Analysis (Calculated): C, 60.43; H, 3.62; N, 16.78. Elemental Analysis (Found): C, 60.44; H, 3.64; N, 16.77.

4-(furan-2-yl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3';5,6]pyrano[2,3*d*]pyrimidine-5,7(1*H*,4*H*)-dione.(5i): IR (KBr/cm-^{1}) 3420 (N-H stretching), 3150 (Ar-H stretching), 2922 (C-H stretching), 1650 (C═O stretching), 1590 (C═N stretching), 1252 (C-O-C stretching), ^{1}HNMR (300MHz, DMSOd6/ppm) δ 2.32 (3H,s), 5.06 (1H,s), 6.0 (1H,s), 6.18 (2H,dd), 6.20 (1H,dd), 7.19 (1H,td), 7.36 7.45 (3H,dd), 7.38 (1H,d), 7.75 (2H,d),10.0 (1H,s),^{13}C NMR (300MHz, DMSO d6 /ppm) δ:13, 34, 106, 109, 122, 124, 129, 137, 140, 152,155,163; EIMS (m/z: RA %): 362.10(100.0%); Elemental Analysis (Calculated): C, 62.98; H, 3.89; N, 15.46.Elemental Analysis (Found): C, 62.96; H, 3.90, N, 15.46,

4-(4-fluorophenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4'3':5,6]pyrano[2,3*d*]pyrimidine-5,7 (1*H*,4*H*)-dione.(5j): IR (KBr/cm-^{1}) 3420 (N-H stretching), 1650 (C═O stretching), 1590 (C═N stretching) 1252 (C–O–C stretching); ^{1}HNMR (300MHz, DMSO d6/ ppm) δ 2.32(3H,s), 5.13 (1H,s), 6.0 (1H,s,NH), 7.07 (2H,d), 7.19 (1H,t), 7.33-7.45 (4H,dd), 7.41 (2H,dd), 7.75 (2H,d), 10.0 (1H,s,NH); ^{13}CNMR (300MHz,DMSOd6/ppm) δ: 13, 37, 106, 115, 122, 129, 137 , 140, 145, 152,161,163**;** EI-MS (m/z: RA %):390 (100.0%); Elemental Analysis (Calculated): C, 64.61; H, 3.87; N, 14.35.Elemental Analysis (Found): C, 64.63; H, 3.86; N, 14.36.

4-(4-chlorophenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyrimidine-5,7 (1*H*,4*H*)-dione.(5n): IR (KBr /cm-^{1}) 3420 (N-H stretching), 3195 (Ar-H stretching), 2949 (C–H stretching), 1665 (C═O stretching), 1590 (C═N stretching), 1252 (C–O–C stretching); ^{1}HNMR (300 MHz, DMSO d6/ ppm) δ 2.32 (3H,s) ,5.18 (1H,s), 6.0 (1H,s,NH), 7.19 (1H,td),7.34-7.50 (6H,dd), 7.37 (2H,d), 7.41 (2H,t),7.75 (2H,t),10.0 (1H,s,NH); ^{13}CNMR (300MHz,DMSOd6/ppm) δ: 37, 106, 122, 124, 129, 137, 140, 145,152, 161; EIMS(m/z:RA%): 406 (100.0%) ; Elemental Analysis (Calculated): C, 62.00; H, 3.72; N, 13.77. Elemental Analysis (Found): C, 62.02; H, 3.74; N, 13.76.

4-(4-(dimethylamino)phenyl)-3-methyl-1-phenyl-6,8-dihydropyrazolo[4',3':5,6]pyrano[2,3-*d*]pyri-midine-5,7(1*H*,4*H*)-dione.(5o): IR(KBr/cm-^{1}) 3433 (N-H stretching), 3195 (Ar-H stretching), 2949 (C-H stretching), 1665 (C═O stretching), 1570 (C═N stretching), 1252 (C–O–C stretching); ^{1}HNMR (300 MHz, DMSO d6/ ppm) δ 2.32 (3H,s,CH_3), 2.74 (6H,s,2-CH_3), 4.92 (1H,s,pyran), 6.0 (1H,s), 6.65 (2H,dd) 6.79 (2H,dd) 7.19 (1H,t) 7.41(2H,t) 7.75(2H,t) 10.0(1H,s); ^{13}CNMR (300MHz, DMSOd6/ppm) δ: 37, 40, 106, 112, 124, 128, 137,152,163; EI-MS (m/z: RA%):415 (100.0%); Elemental Analysis (Calculated): C, 66.49; H, 5.09; N, 16.86. Elemental Analysis (Found): C, 66.50; H, 5.10; N, 16.84.

3.0 RESULT AND DISCUSSION

In present investigation, we have reported the synthesis of pyrazolo pyranopyrimidines derivatives (**5a-q**) via Knovengel-Michael condensation pathways. Initially, a model reaction was examined using Phenyl hydrazine (**1**), Ethyl acetoacetate (**2**), was stirred for ten minutes and then Aromatic substituted aldehyde (**3d**) and Barbituric acid (**4**) were refluxed in water using (DABCO-SiO_2) After one hour derivative (**5d**) was obtained. Optimization of catalyst can be done by using variable quantity of(DABCO-SiO_2). It was observed that the excellent yield was obtained by using 10 mol % of D (DABCO-SiO_2) (Table no.**2).** Also, we checked the effect of different solvent on yield and reaction time it was found that H_2O is an appropriate solvent (**Table no.1**). After investigating the effect of different parameters on the model reaction, we synthesize efficient route for pyrazolo pyranopyrimidines derivatives (**5a-q**) result are summarized in (**Table no. 3).** The desired products (**5a-q**) were obtained to excellent yields. These synthesized products (**5a-q**) were completely characterized from IR, ^{1}H-NMR, Mass and ^{13}C NMR spectroscopic technique and also elemental analysis. We proposed tentative plausible mechanism for the formation of pyrazolo pyranopyrimidines derivatives (**5a-q**) in the presence of (DABCO-SiO_2). The overall, mechanism takes place according to Knovengel condensation followed by Michael addition reaction.

Table 1: Optimization of the reaction conditions using different solvents.

Entry No.	Solvent	Reaction Time (H)	Yield (%)[d]
1	Toluene	2.0	40
2	Ethane1,2-diol	1.5	60
3	DMF	1.5	42
4	Methanol	1.0	68
5	Ethanol	1.0	70
6	Water+Ethanol(1:1)	1.0	83
7	Water	1.0	95

Reaction conditions: phenyl hydrazine **(1) (**1.0 mmol), ethylactoacetate **(2) (**1.0 mmol), substituted aromatic aldehydes **(3)** (1.0 mmol), barbituric acid **(4)** (1.0 mmol) reflux using (DABCO-SiO_2) catalyst in water **[d]** Isolated yields.

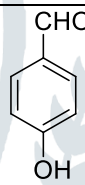
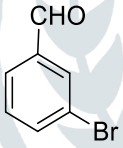
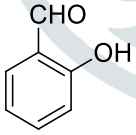
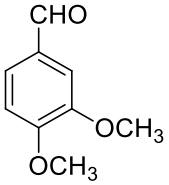
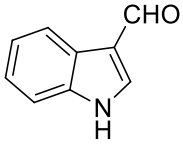
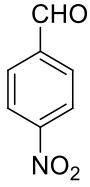
Table 2: Optimization Study for the amount of (DABCO-SiO_2)

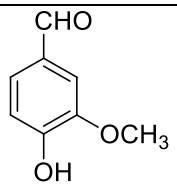
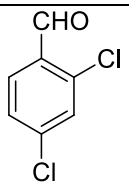
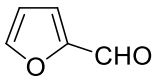
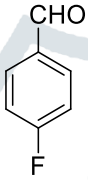
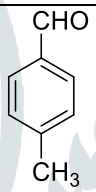
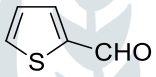
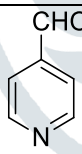
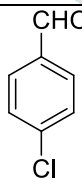
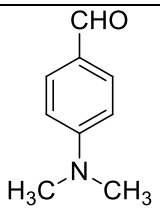
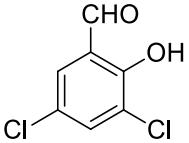
Entry No.	**Catalyst (mol %)**	**Reaction Time (hrs)**	**Yield (%)[d]**
1	01	01	40
2	02	01	60
3	05	01	70

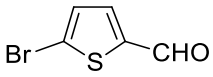
4	10	01	95
5	15	01	92
6	20	01	93
7	25	01	95

Reaction conditions: phenyl hydrazine **(1) (**1.0 mmol), ethylactoacetate **(2) (**1.0 mmol), substituted aromatic aldehydes **(3)** (1.0 mmol), barbituric acid **(4)** (1.0 mmol) reflux using (DABCO-SiO_2) catalyst in water. [**d**] Isolated yields.

Table 3: Four component reaction of phenyl hydrazine (**1**) (1.0 mmol), ethylactoacetate (**2**) (1.0 mmol) substituted aromatic aldehydes (**3**) (1.0 mmol), and barbituric acid (**4**) (1.0 mmol) for the synthesis of (**5a-5q**).

Entry	**Aldehydes**	**Time (Hrs)**	**Yield (%)**	**M.P. (^{0}C)**
3a		1	92	254-256
3b		1	90	235-237
3c		1	93	267-268
3d		1	95	270-271
3e		1	85	282-283
3f		1	85	230-232

3g		1	94	262-263
3h		1	90	233-234
3i		1	86	245-247
3j		1	88	220-221
3k		1	94	188-189
3l		1	82	252-253
3m		1	80	277-278
3n		1	87	224-225
3o		1	95	276-277
3p		1	86	268-269

3q		1	85	258-259

Reaction conditions: phenyl hydrazine **(1)** (1.0 mmol), ethylactoacetate **(2)** (1.0 mmol), substituted aromatic aldehydes **(3)** (1.0 mmol), barbituric acid **(4)** (1.0 mmol) reflux using (DABCO-SiO_2) catalyst in water. [d] Isolated yields.

4.0 CONCLUSION

We have developed a novel efficient and ecofriendly synthesis for the preparation of pyrazolo pyranopyrimidines derivatives by one-pot four component cyclocondensation reaction of phenyl hydrazine, ethylactoacetate ,substituted aromatic aldehydes and barbituric acid were refluxed for short time using(DABCO-SiO_2)also called (TEDA-SiO_2) as catalyst in water Product formed is recrystallized by ethanol, eco-friendly solvent, short reaction time, excellent isolated yields and easy work up make this methodology more facile for synthesis of pyrazolo pyranopyrimidines derivatives.

4.1ACKNLODGEMENT

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5.0 REFRENCES

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