p-TSA catalyzed Multicomponent synthesis of 12-(substituted phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione derivatives.

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ABSTRACT:

A simple procedure has been developed for the synthesis of 12-(substituted phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione derivatives using *β*-Napthol, Barbituric acid and different substituted aromatic Aldehydes were refluxed in ethanol for certain time period using *p*-Toluenesulphonic acid (*p*-TSA) as phase transfer catalyst to form 12-(phenyl)-8*H*-benzo [5,6]chromeno[2,3-*d*]pyrimidine-9, 11(10*H*,12*H*)-dione derivatives.

KEYWORDS: *β*-Napthol, Aromatic Aldehydes, Barbituric acid, *p*-TSA, MCRs.

INTRODUCTION:

Multicomponent reactions (MCRs) have been useful for the synthesis of highly well-designed complex organic molecules and biologically active heterocyclic compounds from simple and willingly available preliminary materials.[1-3] These reactions have attracted extraordinary attention owing to their cleanness, good organization, selectivity, convergence, shorter reaction time, atom-economic unique -eness and environmentally benign.[4-5] As a commanding and widely employed synth- etic etiquette, MCRs provide a highly efficient platform for the rapid synthesis of various fused-ring products, in which the formation of two or more new rings is allowed.[6-9] Fused heterocyclic architectures are extensive in natural products and pharmaceutical molecules, informative their great capacity as a source of novel proficient compounds.[10-13] Multicomponent reactions (MCRs) play an important role in modern synthetic organic chemistry because they generally occur in a single pot and exhibit a high atom-economy and selectivity.

Multicomponent reaction reduces time and saves energy and raw material.[14] Over the past decade, various advanced sequential MCRs have been developed where 1,3-dicarbonyl derivatives are important synthetic intermediates due to its multiple functionalities that can be involved either as nucleophilic or electrophilic species in a large variety of synthetic transformation.[15] Their versatility and effectiveness as potential multicomponent substrates has been used in various MCRs such as Hantzsch 1,4-dihydropyridine synthesis,[16] Biginelli reaction[17] and Michael addition reaction.[18] Multicomponent reactions (MCRs) have emerged as an attractive and powerful strategy for organic synthesis compaired to multistep reactions because of the creation of numerous new bonds in a one-pot reaction, low number of reaction and purification steps, high atom economy, simple procedures, facile implementation and generally excellent yields of products.[19] Therefore, academic and industrial research groups have increasingly focused on the use of MCRs to synthesize a broad range of products [20-21] and development of MCRs can lead to new efficient synthetic methodologies to afford many small organic compounds in the field of modern organic, bioorganic, and medicinal chemistry.[22-24]

LITERATURE REVIEW:

Naphthopyrano pyrimidine and benzo chromeno pyrimidine dione and its derivatives have attracted attention because structural motifs of these compounds are very useful in medicinal and biological chemistry.[25]Also these compounds exhibit promising physiological,[26] hypolipidemic,[27] molluscicidal,[28] antifungal,[29] antitumor,[30] analgesic,[31] antibacterial[32] and anticonvulsant activities.[33]The synthesis of Naphtho- pyrano pyrimidines via earlier methods has been reported so far using formic acid,[34] indium(III) chloride,[35] iodine,[36] ZnO nanoparticles,[37] $H_4[SiW_{12}O_{40}]$,[38]L-proline,[39]poly(AMPS-coAA),[40]alum$KAl(SO_4)_2.12H_2O$,[41]$Al(H_2PO_4)_3$,[42] Fe_3O_4 @SiO_2,Fe_3O_4@MCM-4,[43] basic ionic liquid[44] and trichloroisocyanuric acid (TCCA).[45] Conversely, some of these methods often involve long reaction times, harsh reaction conditions and expensive catalysts. Thus, there is a need to develop a simple and cost-effective etiquette

for the synthesis of novel benzo chromeno pyrimidine dione derivatives and Naphthopyrano pyrimidines with biological importance

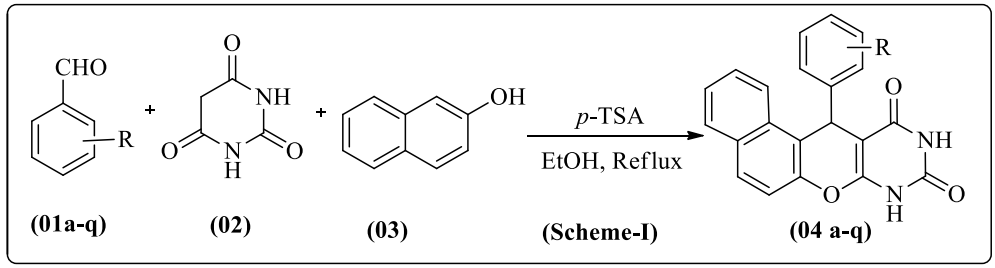
MATERIAL METHOD/ PRESENT WORK:

In present work, we have investigated the one pot three component synthesis of different substituted derivatives of 12-(phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*] pyrimidine-9,11(10*H*, 12*H*)-dione derivatives. The reaction followed by Knovengel condensation and then Michael addition reaction.

A mixture of *β*-Napthol (1mmol)(03), Barbituric acid (1mmol) (02) and different substituted aromatic Aldehyde(1mmol) (01a-q) was refluxed in ethanol for certain time period using *p*-Toluenesulphonic acid (*p*-TSA) (10 mol%) as a catalyst to form12-(phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9, 11(10*H*,12*H*)-dione derivatives. Progress of the reaction was monitored by TLC. Solid formed was filtered, washed with water and recrystallized from ethanol to give (04 a-q). These obtained products (04 a-q) were completely characterized by IR, ^{1}H-NMR, Mass and ^{13}C-NMR spectroscopic technique and also elemental analysis.

RESULT AND DISCUSSION:

A mixture of *β*-Napthol (1mmole) (03), Barbituric acid (1 mmol) (02) and Aromatic aldehyde (1mmol) (04 a-q) was refluxed independently in ethanol using *p*-TSA as an efficient catalyst for certain period of time (Scheme-I).



It was considered as a model reaction (Scheme-I) for investigating the effectiveness of different solvent using catalytic amount of *p*-TSA (10mol %). Solvent optimization clearly noted that ethanol is the best solvent for the desired transformation due to fast reaction rate and high yield (Table-01). We have carried out the model reaction using different stoichiometric amount of catalyst. The catalyst screening result are summarized in (Table-02). It was observed that the excellent yield was achieved by using 10 mol% of *p*-TSA (Table-02).These synthesized products (VA-47a-q) were characterized from IR, ^{1}H-NMR, Mass and ^{13}C-NMR spectroscopic technique and also elemental analysis.

The *p*-TSA acting as phase transfer catalyst (PTC) that's why reaction mechanism was accelerated, *p*-Toluenesulphonic acid (PTSA) is commercially available and is a very cheap chemical, white solid, non-volatile that is soluble in water, alcohols, and other polar organic solvents. Most often, TsOH refers to the monohydrate, TsOH.H_2O. TsOH is a strong organic acid, about a million times stronger than benzoic acid. This catalyst can act as ecofriendly for a variety of organic transformations.

We propose tentative plausible mechanism for the formation of 12-(phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione (04 a-q), in the presence of *p*-TSA. The overall, mechanism takes place according to Knoevenagel-Michael reaction (Scheme-I).The spectral and physical data of the compound is proved by agreement data.

Table.01. Optimization of the reaction conditions using different solvents.

Entry	Solvent	Reaction Time (h)	Yield (%)[b]
1	DMF	6.0	35
2	Ethylene glycol	5.5	50
3	THF	6.2	40
4	Acetonitrile	6.0	52
5	DCM	7.0	58
6	Ethanol	5.0	90
8	Water+Ethanol(1:1)	5.0	65

Reaction conditions: *β*-napthol **(1 mmol) (03)**, Barbituric acid **(1 mmol) (02)** and aromatic aldehyde **(1 mmol) (01)** was refluxed at 70 ^{0}C. **[d]** Isolated yields.

Table-02: Optimization Study for the amount of *p*-TSA.

Entry	Catalyst (mole %)	Temperature (^{0}C)	Reaction Time (h)	Yield %[b]

1	01	70	5.0	36
2	02	70	5.0	44
3	04	70	5.0	56
4	06	70	5.0	64
5	08	70	5.0	76
6	10	70	5.0	90
7	15	70	5.0	92

Reaction conditions: *β*-napthol **(1 mmol) (03)**, Barbituric acid **(1 mmol) (02)** and aromatic aldehyde **(1 mmol) (01)** was refluxed at 70 ^{0}C. **[d]** Isolated yields.

Probable Mechanism:

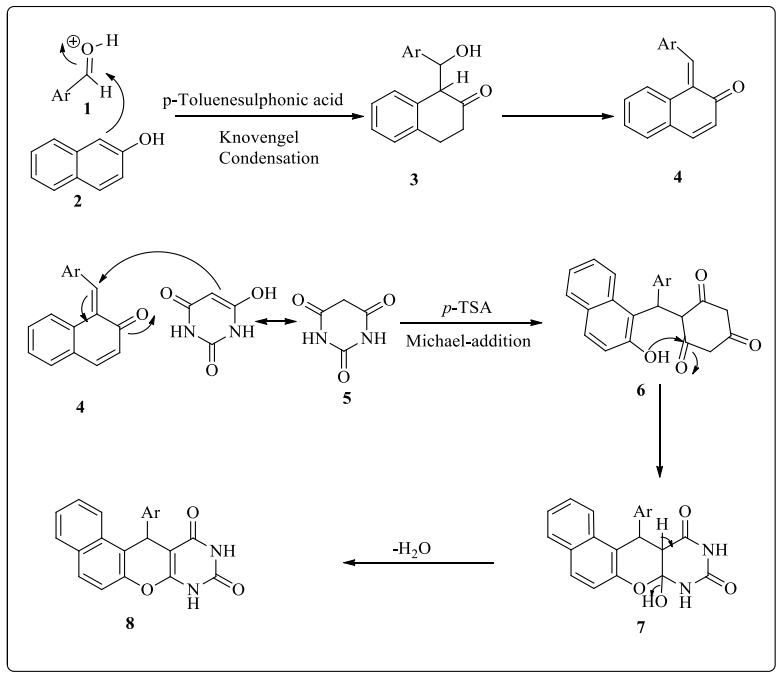
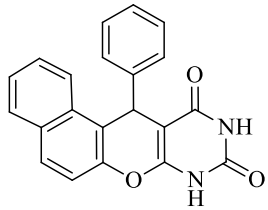
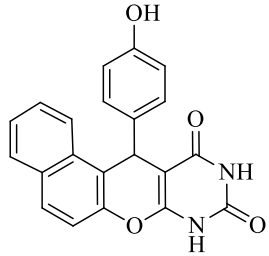
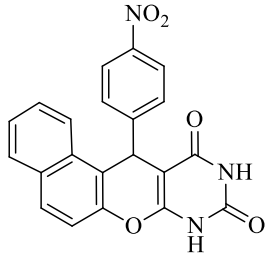
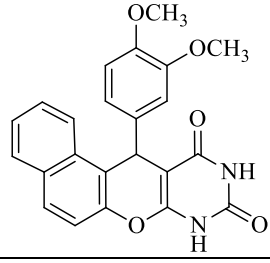
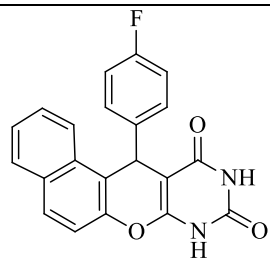
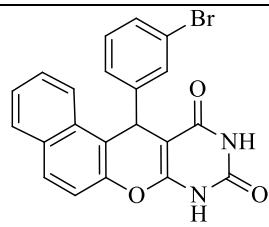
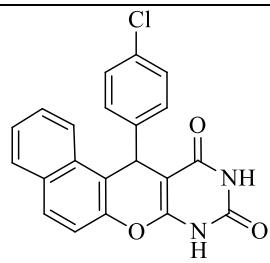
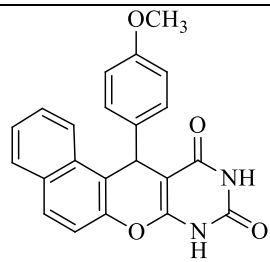
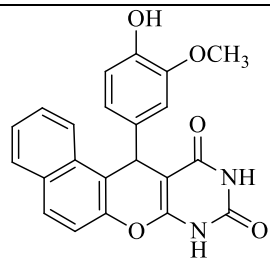
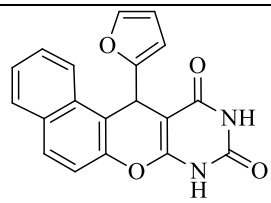
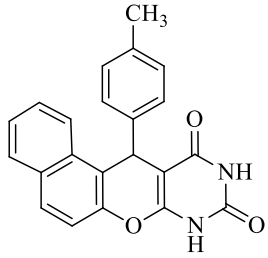
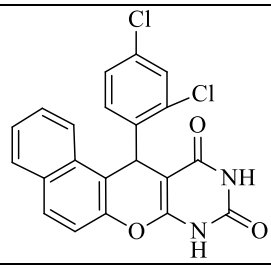
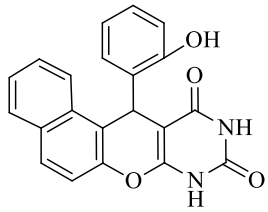
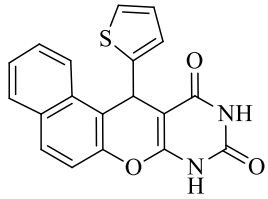
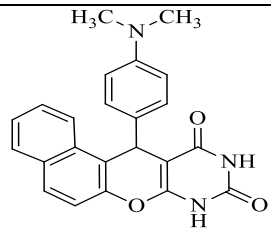
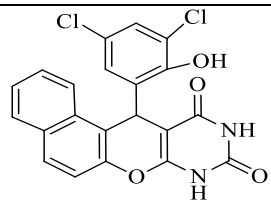
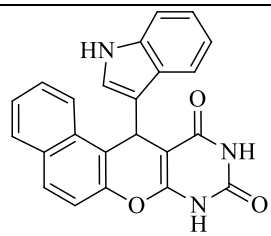


TABLE.03 Reaction Time, Yields and M.P.of 12-(phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione derivatives.

Entry	Comp. code	Structure of compounds	Time (Hrs)	Yield (%)	M.P. (Obs. ^{0}C)	M.P. (Lit. ^{0}C)
1	04 a		5.2	80	275-277	276-278
2	04 b		5.0	90	248-250	250-252

3	04 c		5.5	78	304-306	305-307
4	04 d		5.0	90	288-289	288-290
5	04 e		5.2	82	278-279	280-282
6	04 f		5.5	77	262-264	265-266
7	04 g		5.2	82	298-300	301-302
8	04 h		5.0	86	257-259	258-260
9	04 i		5.0	84	284-286	286-288

10	04 j		6.5	72	272-274	274-276
11	04 k		5	86	279-280	280-281
12	04 l		5.5	74	290-291	291-292
13	04 m		5.5	80	250-252	252-254
14	04 n		6.5	70	266-268	268-270
15	04 o		5	87	274-276	276-278
16	04 p		5.0	80	305-306	306-308
17	04 q		6.0	70	277-278	278-279

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 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-7070 H Mass spectrometer using the EI technique at 70 eV.

SPECTRAL ANALYSIS:(FINDINGS)

1) **12-(4-nitrophenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)dione.(04c) (** IR (KBr/cm^{-1}) 3234 (-NH), 3080 (Ar C-H), 1716 (-C=O), 1694 (Ar C=C), 1531 (-NO_2) cm^{1}; ^{1}HNMR (300MHz, DMSO d_6/ ppm); δ 4.75(1H,s,CH),7.32 (1H,d,Ar-H), 7.50 (2H,d,Ar-H), 7.60-7.72 (2H,t,Ar-H), 7.80 (H,d,Ar-H), 7.94 (1H,d,Ar-H), 8.15 (1H,d, Ar-H), 8.27 (1H,d,Ar-H) 11.30 (1H,s,NH), 11.86(1H,s,NH)EI-MS (m/z: RA %): 387 ($M^{+\cdot}$, 100%). ^{13}C NMR (300 MHz, DMSO–d_6/ppm) δ:37.1, 81.30, 118.91,122.12, 123.23, 123.24, 125.55, 125.55,128.56,128.86,133.57,145.48,151.29,151.81,154.12,157.93,164.10;Elemental analysis **:** Calculated data for $C_{21}H_{13}N_3O_5$; C, 65.12; H 3.38, N, 10.85. Found: C 65.06; H, 3.32; N, 10.20.
2) **12-(4-fluorophenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*) dione.(04e)** IR (KBr/cm^{-1}) 3262 (-NH), 3112 (Ar C-H), 1744 (-C=O),1354 (C-F) cm^{-1}; ^{1}HNMR(300MHz, DMSO d_6/ ppm); δ 4.75(1H,s,CH),6.90 (2H,d,Ar-H), 7.02 (2H,d,Ar-H), 7.25 (1H,d,Ar-H), 7.45 (2H,t,Ar-H), 7.65 (1H,d,Ar-H), 8.00 (1H,d,Ar-H), 8.20 (1H,d,Ar-H) 11.05 (1H,s,NH), 12.07(1H,s,NH). EI-MS (m/z: RA %): 360 ($M^{+\cdot}$, 100%); ^{13}CNMR (300MHz, DMSOd_6/ppm) δ: 37.10, 82.10, 116.56, 119.02, 122.10, 123.25, 126.34, 129.14, 130.02, 135.28, 142.37, 148.20, 150.70, 151.82, 156.13, 160 .40, 164.92.; Elemental analysis**:** Calculated data for $C_{21}H_{13}FN_2O_3$; C 70.00, H 3.64, N 7.77 Found: C 70.02, H 3.60, N 7.72)
3) **12-(4-chlorophenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione.(04.g)** IR (KBr/cm^{-1}) 3219 (-NH), 3091 (Ar C-H), 1755 (-C=O), 1288 (C-O-C), 792(C-Cl) cm^{-1}; ^{1}HNMR (300MHz, DMSO d_6/ ppm) δ: 4.85(1H,s,CH),7.12 (2H,d,Ar-H), 7.37-7.40 (2H,d,Ar-H), 7.52 (1H,d,Ar-H), 7.75 (2H,t,Ar-H), 7.85 (1H,d,Ar-H), 8.07 (1H,d, Ar-H), 8.20 (1H,d,Ar-H) 11.26 (1H,s,NH), 11.45 (1H,s,NH); EI-MS (m/z: RA %): 376 ($M^{+\cdot}$, 100%)^{13}CNMR (300MHz, DMSOd_6/ppm) δ: 37.46, 81.30, 117.90, 119.03, 123.20, 125.70, 126.23, 128.82, 129.42, 131.80, 135.50, 146.70, 150.70, 151.90, 156.19, 163.00. Elemental analysis**:** Calculated data for $C_{21}H_{13}ClN_2O_3$; C 66.94, H 3.48, N 7.43 Found: C 66.90, H 3.39, N 7.3
4) **12-(4-methoxyphenyl)-8-*H*benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione.(04 h)** IR (KBr/cm^{-1}) 3211 (-NH), 3064(Ar C-H), 1732 (-C=O), 1269 (C-O-C) cm^{-1}; ^{1}HNMR (300MHz, DMSO d_6/ ppm) δ: 3.85 (3H,s,-OCH_3), 4.90 (1H,s,-CH), 7.05 (2H,d,Ar-H), 7.18 (2H,d,Ar-H), 7.35 (1H,d,Ar-H), 7.50 (1H,t,Ar-H), 7.60 (1H,t,Ar-H), 7.70 (1H,d,Ar-H), 8.05 (1H,d,Ar-H), 8.22 (1H,d,Ar-H) 11.16 (1H,s,NH), 11.29 (1H ,s,NH). ; EI-MS (m/z: RA %): 372 ($M^{+\cdot}$, 100%); ^{13}CNMR (300MHz, DMSOd_6/ppm) δ:38.12,82.00,115.20, 115.20,118.90,122.36,123.01,123.14,126.19,128.40,129.12,129.12,130.06,139.93,150.70,151.10,156.86,159.00,163.00 Elemental analysis**:** Calculated data for $C_{21}H_{13}N_3O_3$; C 70.96, H 4.33, N 7.52 Found: C 70.90, H 4.29, N 7.48)..

CONCLUSION:

We have proposed a novel efficient and eco-friendly synthesis of 12-(substituted phenyl)-8*H*-benzo[5,6]chromeno[2,3-*d*]pyrimidine-9,11(10*H*,12*H*)-dione derivatives by one-pot three component condensation reactions. The product can be easily isolated by simple work up technique, ecofriendly catalyst. Furthermore, the procedure offers a number of advantages including improved yields, simple experimental procedure, cleaner reactions and low cost which makes it a useful and attractive strategy with respect to economic and environmental advantages.

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