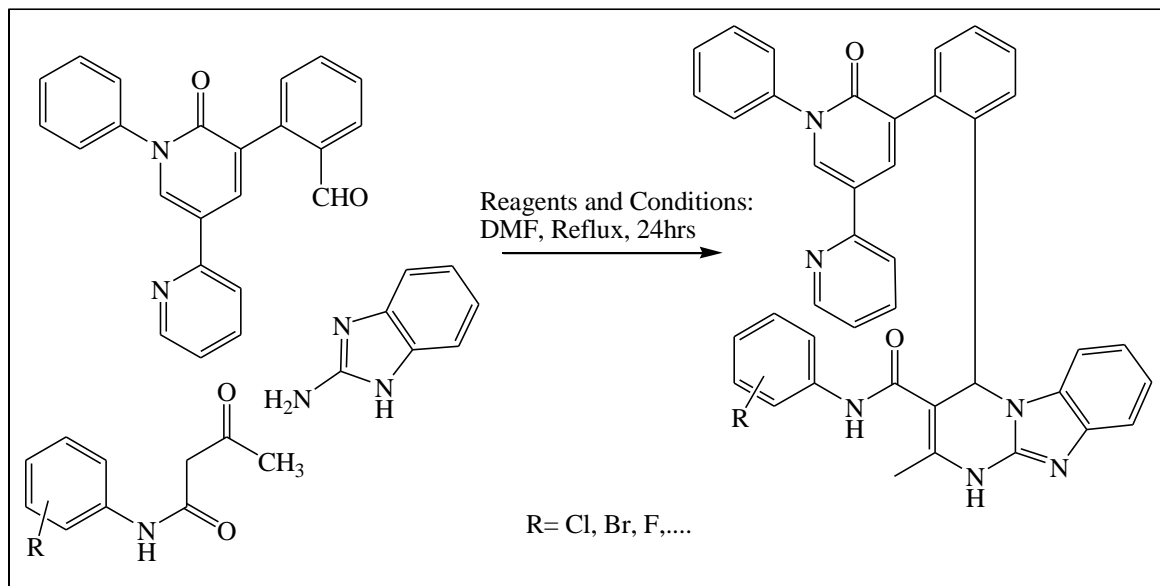


SYNTHESIS AND STRUCTURAL ELUCIDATION OF 1, 4 DIHYDROPYRIMIDO [1, 2-A] BENZIMIDAZOLE

ABSTRACT

Considering various biomedical significance & with a view of pharmacological actions of compound belong to this class, new series of N-(substituted phenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2-3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido [1, 2-a] benzimidazole-3-carboxamide were prepared. The formation of the compound was accomplished by cyclocondensation of 2-(1, 2-dihydro-2-oxo-1-phenyl-5-(pyridin-2-yl)pyridin-3-yl) benzaldehyde and N-(substituted phenyl)-3-oxobutanamide, 2-amino-benzimidazole under acid catalyzed conditions. The structures of pyrimidobenzimidazoles were determined by MASS, IR, ¹H NMR spectroscopic techniques & Elemental analysis technique.



Keywords: Pyrimidobenzimidazoles, Carboxamide, Biomedical, Biginelli, Dimethylformamide

1. INTRODUCTION

Heterocyclic compounds are important class of molecules in organic chemistry. They have been developed since many decades through chemists due to their significant role in medicinal chemistry and make available as a type pattern for the growth of a hodgepodge of therapeutic agents.¹⁻³ Nitrogen

containing heterocycles are widely used amongst all heterocyclic moieties whether it is five or six member ring. Most biologically-active chemical entities contain a heterocycle. It means that heterocycles have vital role to impart in the drug designing section. Heterocycles can be applied to modify various properties of biologically active compounds like polarity, solubility, toxicity etc in the drug development phase. The large variety of heterocyclic compounds is available due to advance synthetic methodologies developed by many scientists. Heterocycles can also be isolated from natural sources and it can be modify as per the requirements. Medicinal chemists play significant role here to identify the lead compound from natural sources and modify their structures to impart some characteristics or decreases the toxic effects during drug discovery process.

Multicomponent reaction a way out because they are good organized cost resourceful and less profligate than straight process. The products of temperature quick to respond reactions from kinetic path can be selectively inaccessible. As multicomponent coupling often complex compound in a single step, they are extensively studied in the era of modern organic chemistry.⁴ Poly substituted pyrimido [1, 2-a] benzimidazoles display a wide array of medicinal activities. They bear structural resemblance to purine bases.

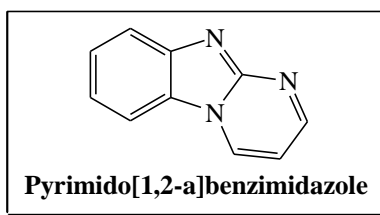


Figure 1. Structure of pyrimido [1, 2-a] benzimidazole

It serves as a trail to produce molecular environmental that combats the normally expensive and time-consuming drug detection process whereby a small number of novel therapeutics arrive at the market place. The added investigational benefits of generating multifaceted structures from easy raw materials without complex procedures, long-lasting purification procedures get better the synthetic approach for future youthful researchers wishing to donate products to a more systematically inventive civilization.

Pyrimidines have extensive and famed times gone by extending from the days of their judgment as significant part of nucleic acids to their present use in the AIDS chemotherapy.

Uracil, thymine and cytosine are the three important constituents of nucleic acids. Alloxan produces diabetes inducing reaction.⁵

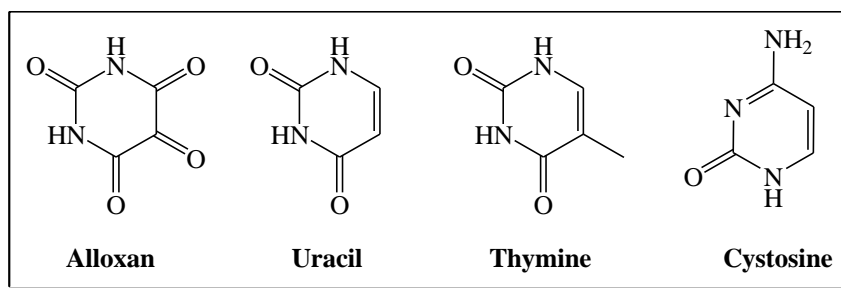


Figure 2. Structure of pyridine base compounds

Pyrimidine class of compound are widely researched & employed as antibacterial agents.⁶ Heterocycles bearing a benzimidazole moiety were reported to demonstrate a wide array of Medicinal properties like antimicrobial, anticonvulsant, anticancer, anti-inflammatory, analgesic, antidepressant, bioactivities, Antimicrobial, antimalarial, anticonvulsant, anticancer, anti-inflammatory, analgesic and antitubercular.⁷⁻¹⁵ Some other experiments work done in our laboratory^[14-18]

2. METHODOLOGY

An open capillary tubes method and uncorrected melting point were reported. Pattern of the compound was on the whole tartan by TLC on silica gel plates (0.51 mm) thickness and stain were situated by I₂ and Ultra Violet. Infrared spectra were documentation in Shimadzu instrument FTIR-8400 using KBr pellet method. Mass spectra were records on Shimadzu GC-MS-QP-2010 mold using express Injection probe method and Turbo spray model using chemical isolation technique. ¹H NMR was dogged in CDCl₃/dimethyl sulfoxide solution on a Bruker Ac 400 MHz spectrometer.

Common Methods for the preparation of N-(substitutedphenyl)-1,6-dihydro-[2,3'-bipyridin]-2-Methyl-4-(2-(6'-oxo-1'-phenyl-5'-yl)phenyl)- 1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK- 501 to 510)

A combination of the 2-amino benzimidazole (0.01 M) N-(substitutedaryl)-3-oxo-4-methyl pentanamides (0.02 M) and 2-(1,2-dihydro-2-oxo-1-phenyl-5-(pyridin-2-yl) pyridin-3-yl) benzaldehyde (0.01 M) was heated to reflux in dimethylformamide (20 ml) for 24 hrs. After attaining the room temperature methanol (~25 mL) was supplementary. The reaction mass was kept for 12-14 hours and then filtered to give the 1,4-dihydro pyrimido[1,2-a]benzimidazoles (VK- 501 to 510), which were crystallization from alcohol.

N-(Phenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1-6-dihydro-[2-3-bipyridin]-5-yl)phenyl)-1-4-dihydropyrimido[1,2-a] benzimidazole-3-carboxamide (VK-501).

Yield: 65.3%; MP 170°C; Mass: m/z 627.

IR (cm⁻¹): 3424 (νN-H secondary amide), 3094 (νC-H of aromatic ring), 2944 (νC-H asym of CH₃ group), 2849 (νC-H asym of CH₃ group), 1682 (νC=O of amide), 1594 (νN-H of pyrimidine ring), 1525 (νC=C of aromatic ring), 1454 (δ C-H asym of CH₃ group), 1345 (νC-H sym of CH₃ group), 1265 (νC-N), 742 (νC-H in plane of aromatic ring)

¹HNMR (DMSO d₆) δ ppm: 1.48 (s 3H, Ha), 5.26 (s 1H, Hb), 6.86-6.88 (m 2H, Hcd), 6.98-7.00 (dd' 2H, Hee), 7.05-7.07 (dd' 2H, Hff'), 7.12-7.15 (m 4H, Hg-j), 7.28-7.29 (dd' 2H, Hkk), 7.47-7.49 (dd' 2H, Hll'), 7.68-7.69 (t 4H, Hm-p), 7.83-7.84 (dd' 2H, Hqq'), 8.45-8.48 (m 3H, Hr-t); 8.70 (s 1H, Hu), 8.88-9.2 (s 1H, Hv).

N-(4-Chlorophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro [2-3-bipyridin]-5-yl)-phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-502).

Yield: 57%; mp 187°C; MS: m/z 661.

IR (cm⁻¹): 3252 (νN-H secondary amide), 3075 (νC-H of aromatic ring), 2952 (νC-H asym of CH₃ group), 2824 (νC-H asym of CH₃ group), 1652 (νC=O of amide), 1602 (νN-H secondary amide), 1556 (νC=C of aromatic ring), 1496 (νC-H asym of CH₃ group), 1365 (νC-H asym of CH₃ group), 1266 (νC-N), 695 (νC-H in plane of aromatic ring), 695 (νC-Cl stretch).

¹H NMR (Dimethyl sulfoxide-d₆) δ ppm: 1.50 (s 3H, Ha), 5.36 (s 1H, Hb), 6.82-6.87 (m 2H, Hcd), 7.0-7.3 (dd' 2H, Hee), 7.07-7.1 (dd' 2H, Hff'), 7.11-7.18 (m 4H, Hg-j), 7.26-7.30 (dd' 2H, Hkk), 7.41-7.42 (dd' 2H, Hll'), 7.66-7.69 (t 4H, Hm-p), 7.81-7.84 (dd' 2H, Hqq'), 8.46-8.49 (m 3H, Hr-t); 8.72 (s 1H, Hu), 8.81 (s 1H, Hv).

N-(3-Chlorophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2-3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide(VK-503).

Yield: 63%; mp 188°C; MS: m/z 661.

IR (cm⁻¹): 3360 (νN-H secondary amide), 3082 (νC-H of aromatic ring), 2957 (νC-H asym of CH₃ group), 2850 (νC-H asym of CH₃ group), 1670 (νC=O of amide), 1608 (νN-H of pyrimidine ring), 1561 (νC=C of aromatic ring), 1450 (νC-H asym of CH₃ group), 1394 (νC-H asym of CH₃ group), 1263 (νC-N), 742 (νC-H in plane of aromatic ring), 690 (νC-Cl stretch).

¹H NMR (Dimethyl sulfoxide-d₆) δ ppm: 1.49 (s 3H, Ha), 5.32 (s 1H, Hb), 6.84-6.87 (m 2H, Hcd), 7.2-7.3 (dd' 2H, Hee), 7.09-7.1 (dd' 2H, Hff'), 7.13-7.19 (m 4H, Hg-j), 7.27-7.31 (dd' 2H, Hkk), 7.44-7.45 (dd' 2H, Hll'), 7.67-7.70 (t 4H, Hm-p), 7.82-7.85 (dd' 2H, Hqq'), 8.45-8.49 (m 3H, Hr-t); 8.75 (s 1H, Hu), 8.80 (s 1H, Hv).

N-(4-Bromophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide(VK-504).

Yield: 58%; mp 182°C; MS: *m/z* 706.

IR (cm⁻¹): 3217 (νN-H secondary amide), 3082 (νC-H of aromatic ring), 2950 (νC-H_{asym} of CH₃ group), 2848 (νC-H_{asym} of CH₃ group), 1645 (νC=O of amide), 1593 (νN-H of pyrimidine ring), 1548 (νC=C of aromatic ring), 1506 (νC-H_{asym} of CH₃ group), 1323 (νC-H_{asym} of CH₃ group), 1244 (ν C-N), 745 (νC-H in plane of aromatic ring), 690 (νC-Br stretch).

¹H NMR (Dimethyl sulfoxide-d₆) δ ppm: 1.52 (s 3H, Ha), 5.39 (s 1H, Hb), 6.87-6.89 (m 2H, Hcd), 7.3-7.5 (dd' 2H, Hee), 7.1-7.2 (dd' 2H, Hff'), 7.12-7.19 (m 4H, Hg-j), 7.26-7.30 (dd' 2H, Hkk), 7.43-7.45 (dd' 2H, Hll'), 7.65-7.70 (t 4H, Hm-p), 7.84-7.88 (dd' 2H, Hqq'), 8.47-8.51 (m 3H, Hr-t); 8.75 (s 1H, Hu), 8.86 (s 1H, Hv).

N-(3-Bromophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-505).

Yield: 55%; MP 178°C; MS: *m/z* 706.

IR (cm⁻¹) : 3446 (νN-H secondary amide), 3096 (νC-H of aromatic ring), 2948 (νC-H_{asym} of CH₃ group), 2805 (νC-H_{asym} of CH₃ group), 1705 (νC=O of amide), 1633 (νN-H of pyrimidine ring), 1528 & 1440 (νC=C of aromatic ring), 1386 (δC-H_{asym} of CH₃ group), 1300 (νC-H_{asym} of CH₃ group), 1256 (νC-N), 775 (νC-H in plane of aromatic ring), 695 (νC-Br stretch)

¹H NMR (Dimethyl sulfoxide-d₆) δ ppm: 1.51 (s 3H, Ha), 5.38 (s 1H, Hb), 6.87-6.88 (m 2H, Hcd), 7.1-7.3 (dd' 2H, Hee), 7.09-7.1 (dd' 2H, Hff'), 7.13-7.19 (m 4H, Hg-j), 7.29-7.31 (dd' 2H, Hkk), 7.46-7.47 (dd' 2H, Hll'), 7.67-7.69 (t 4H, Hm-p), 7.83-7.87 (dd' 2H, Hqq'), 8.46-8.49 (m 3H, Hr-t); 8.75 (s 1H, Hu), 8.84 (s 1H, Hv).

N-(4-Fluorophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-506).

Yield: 62.82%; MP 189°C; MS: *m/z* 645.

IR (cm⁻¹): 3392 (νN-H secondary amide), 3059 (νC-H of aromatic ring), 2926 (νC-H_{asym} of CH₃ group), 2847 (νC-H_{asym} of CH₃ group), 1647 (νC=O of amide), 1591 (νN-H of pyrimidine ring), 1548 & 1508 (νC=C of aromatic ring), 1406 (δ C-H_{asym} of CH₃ group), 1340 (νC-H_{asym} of CH₃group), 1271 (νC-N), 1026 (νC-Fstretch), 730 (νC-H in plane of aromatic ring).

N-(3-Fluorophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-507).

Yield: 66%; MP 187°C; MS: *m/z* 645.

IR (cm⁻¹): 3300 (νN-H secondary amide), 3001 (νC-H of aromatic ring), 2933 (νC-H_{asym} of CH₃ group), 2850 (νC-H_{asym} of CH₃ group), 1687 (νC=O of amide), 1526 (νN-H of pyrimidine ring), 1533 & 1465 (νC=C of aromatic ring), 1344 (δ C-H_{asym} of CH₃ group), 1292 (νC-H_{asym} of CH₃ group), 1197 (νC-N), 1010 (νC-F stretch), 748 (νC-H in plane of aromatic ring).

N-(4-Methylphenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-508).

Yield: 63%; mp 183°C; MS: *m/z* 641.

IR (cm⁻¹): 3116 (νN-H secondary amide), 3000 (νC-H of aromatic ring), 2942 (ν C-H_{asym} of CH₃ group), 2856 (νC-H_{asym} of CH₃ group), 1627 (νC=O of amide), 1528 (νN-H of pyrimidine ring), 1454 & 1442 (νC=C of aromatic ring), 1365 (νC-H_{asym} of CH₃ group), 1299 (νC-H_{asym} of CH₃ group), 1238 (νC-N), 774 (νC-H in plane of aromatic ring)

N-(4-Methoxyphenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-509).

Yield: 61%; mp 184°C; MS: *m/z* 657.

IR (cm⁻¹): 3114 (νN-H secondary amide), 3009 (νC-H of aromatic ring), 2956 (νC-H_{asym} of CH₃ group), 2894 (νC-H_{asym} of CH₃ group), 1657 (νC=O of amide), 1576 (νN-H of pyrimidine ring), 1518, 1502 & 1460 (νC=C of aromatic ring), 1345 (νC-H_{asym} of CH₃ group), 1324 (νC-H_{asym} of CH₃ group), 1257 (νC-N), 731 (νC-H in plane of aromatic ring).

N-(4-Nitrophenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1,6-dihydro-[2,3-bipyridin]-5-yl)phenyl)-1,4-dihydro pyrimido[1,2-a] benzimidazole-3-carboxamide (VK-510).

Yield: 71%; mp 181°C; MS: *m/z* 672.

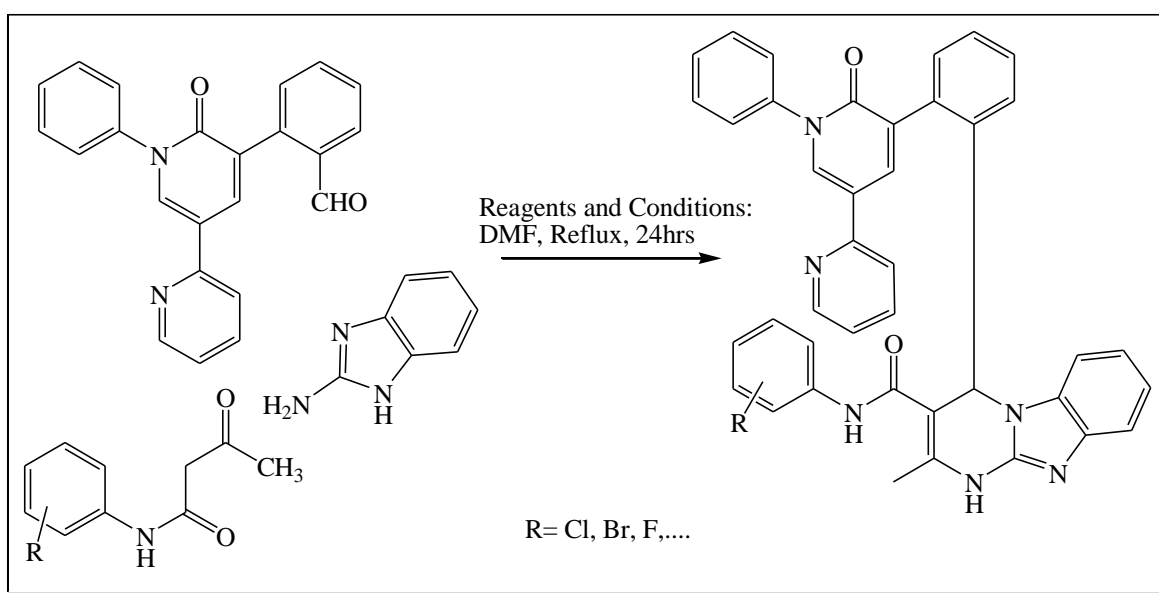
IR (cm⁻¹): 3390 (νN-H secondary amide), 3028 (νC-H of aromatic ring), 2936 (νC-H_{asym} of CH₃ group), 2853 (νC-H_{asym} of CH₃ group), 1687 (νC=O of amide), 1611 (νN-H of pyrimidine ring), 1532, & 1446 (νC=C of aromatic ring), 1364 (νC=C of Aromatic ring), 1333 (νC-H_{sym} of CH₃ group), 1275 (νC-N), 1275 (νC-N), 750 (νC-H in plane of Aromatic ring)

3. RESULTS AND DISCUSSION

The bioactivity potential of 1, 4-dihydropyrimido [1, 2-a] benzimidazoles is widely reported. Further, diversely substituted derivatives of this class of compound have depicted impressive medicinal profile.

Man-made ways to 1, 4-dihydropyrimido [1, 2-a] benzimidazoles is pedestal on the **Biginelli** like cyclo combination of new -CHO and N-(substituted phenyl)-3-oxobutanamide with 2-amino benzimidazole.

Noting a range of claim biomedical and with a view further to deem the Medicinal profile of this set of compound, narrative series of N-(substituted phenyl)-2-methyl-4-(2-(6-oxo-1-phenyl-1-6-dihydro-[2-3-bipyridin]-5-yl)phenyl)-1-4-dihydropyrimido[1,2-a] benzimidazole-3-carboxamide (VK-501 to VK-510) were manufactured. The manufacture of (VK-501 to VK-510) were achieved by reaction of N-(substituted phenyl)-3-oxobutanamide, 2-aminobenzimidazole and 2-(1,2-dihydro-2-oxo-1-phenyl-5-(pyridin-2-yl)pyridin-3-yl)benzaldehyde in the presence of HCl.



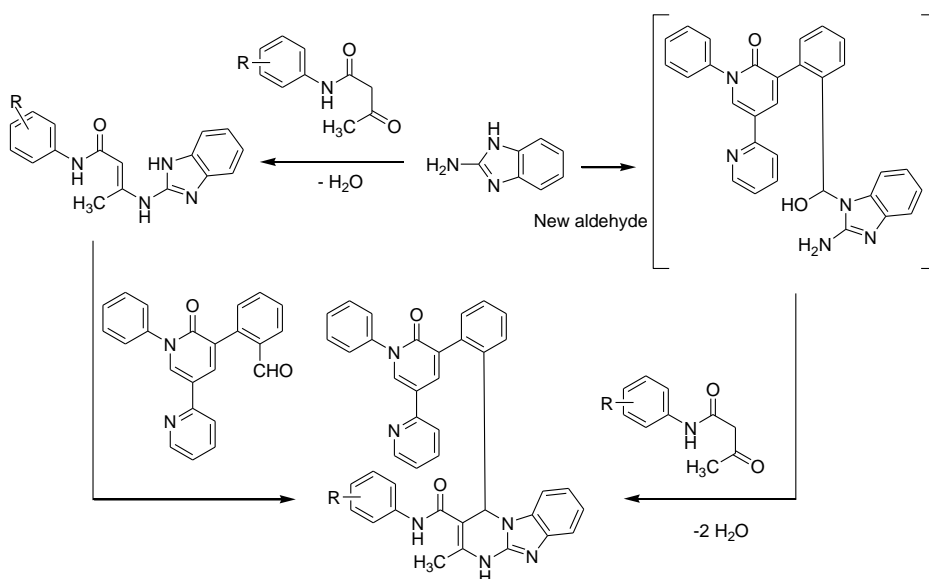
Scheme 1. Reaction scheme of pyrimidine derivatives

Table 1. Physical data

Code	R	Molecular formula	Molecular Weight	Melting Point °C	R _f	Yield
VK-501	H	C ₄₀ H ₃₀ N ₆ O ₂	627	170	0.43	64
VK-502	4-Cl	C ₄₀ H ₂₉ ClN ₆ O ₂	661	187	0.44	57
VK-503	3-Cl	C ₄₀ H ₂₉ ClN ₆ O ₂	661	188	0.50	63
VK-504	4-Br	C ₄₀ H ₂₉ BrN ₆ O ₂	706	182	0.49	58
VK-	3-Br	C ₄₀ H ₂₉ BrN ₆ O ₂	706	178	0.43	55

505	VK-	4-F	C ₄₀ H ₂₉ FN ₆ O ₂	645	189	0.44	62
506	VK-	3-F	C ₄₀ H ₂₉ FN ₆ O ₂	645	187	0.46	66
507	VK-	4-	C ₄₁ H ₃₂ N ₆ O ₂	641	183	0.40	63
508		Me					
509	VK-	4-	C ₄₁ H ₃₂ N ₆ O ₃	657	184	0.50	61
510		OMe					
	VK-	4-	C ₄₀ H ₂₉ N ₇ O ₄	672	181	0.47	71
		NO ₂					

3.1 Plausible reaction mechanism



Scheme 2. Plausible reaction mechanism

4. CONCLUSION

In conclusion, Our study commenced with different types of N-(substituted phenyl)-3-oxobutanamide, 2-aminobenzimidazole, two new special kind of aldehyde as 4-(4-(trifluoromethyl)-2-nitrophenoxy)-2-hydroxybenzaldehyde and 2-(1,2-dihydro-2-oxo-1-phenyl-5-(pyridin-2-yl)pyridin-3-yl)benzaldehyde in presence of DMF (N,N'-Dimethyl formamide) as solvent. The reaction proceeds with fast rate and yields good to high percentage of product. Structure were elucidated by IR, mass and NMR.

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