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# Synthesis and application of melamine-based nano catalyst with phosphonic acid tags in the synthesis of (3´-indolyl)pyrazolo[3,4-*b*]pyridines *via* vinylogous anomeric based oxidation

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### ABSTRACT

A novel nanostructured melamine-based organo solid acid with phosphorus acid tags was designed, synthesized and fully characterized. The catalytic activity of the resulted melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA) as a heterogeneous catalyst was also investigated *via* an one-pot reaction between cyanoacetylindole, 3-methyl-1-phenyl-1*H*-pyrazol-5-amine, and an aromatic aldehyde. (3'-Indolyl)pyrazolo[3,4-*b*] pyridines were produced in good yields under refluxing ethanol. The solid catalyst can be recovered easily and reused without a significant loss of its catalytic activity.

### 1. Introduction

Molecules with indole nucleus are often characterized by biological activities, such as antibacterial [1], antifungal [2], antiviral [3], anticonvulsant, cardiovascular [4], optimal inhibitory [5], anti-hypertensive [6], anticholinergic [7] and antiproliferative [8] activities. Some natural products such as alkaloids have indole moiety in their structure. These compounds act as antitumor, anticancer, anti-inflammatory, hypoglycemic, antipyretic or analgesic reagents (Fig. 1) [4,9].

Pyrazole derivatives also have variety of biological and pharmacological properties including antibacterial [10], anti-inflammatory [11], antiviral [12], anticancer [13], antidiabetic [14], hypotensive [15] activities. Some of these compounds have been used in the therapy of Alzheimer's disease, drug addiction, infertility and gastrointestinal diseases [16].

Pyridine moiety is one of the most important scaffolds, which has found in the many pharmaceuticals and natural products because of their biological and medicinal properties, such as inhibiting HIV protease, antidepressant, anti-inflammatory, inhibiting acetylcholinesterase, treating hypotension or hypertension [17]. Therefore, efficient methods for their preparation are important task of organic chemists.

The hyperconjugative electrons sharing from a lone pair orbital to a vacant antibonding sigma orbital (n-o<sup>\*</sup>) had been named as anomeric effect. For example, triazaphenalene, a tricyclic ortho amide, easily produces the stable corresponding guadinium cation and molecular hydrogen when it is treated with HBF<sub>4</sub> at 110 °C (Scheme 1) [18]. It is well-known that the cooperative anomeric effect supports this unusual hydride transfer and molecular hydrogen (H<sub>2</sub>) releasing.

Recently, Alabugin and co-workers have been comprehensively reviewed the stereoelectronic effects [19]. They have explained various kinds of hyperconjugation, anomeric effect and  $\sigma$ -conjugation. Katritzky and coworkers had been shown that the stereoelectronic interactions can be extended through double bonds *via* a vinylogous anomeric effect [20]. On the other word, the vinylogous anomeric effect had been applied for the stereoelectronic supported interaction of unshared electron pairs to a vacant antibonding sigma orbital (n- $\sigma^*$ ) over four bonds (Scheme 2) [21–28].

In recent years, phosphorus acid tag has been used as a valuable synthon due to its application at the preparation of catalysts, inhibitors, metal adsorbent and extractants [29–33]. In this regards, we have reported novel catalysts with phosphorus acid tags for the

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**Scheme 1.** Hydrogen releasing supported by the three cooperative anomeric interactions in the triazaphenalene [18].

synthesis of a wide range of organic compound [34]. In the continuation of our previous studies on this research field, the melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA) as a highly efficient, heterogeneous and reusable organo solid acidic catalyst for various functional group transformations was prepared. Herein, we were interested to develop cooperative vinylogous anomeric based oxidation mechanism [35], at the preparation of (3'-indolyl)pyrazolo [3,4-*b*]pyridines from aryl aldehydes, 3-cyanoacetylindole and 3-methyl-1-phenyl-1*H*-pyrazol-5-amine in the presence a catalytic amount of described catalyst (MHMHPA) under mild and green conditions (Scheme 3).

### 2. Experimental

# 2.1. General procedure for the synthesis of melamine hexakis(methylene) hexakis(phosphonic acid) (MHMHPA)

In a 50 mL round-bottomed flask, a mixture of paraformaldehyde (6 mmol, 0.180 g, 0.2 mL), 1,3,5-triazine-2,4,6-triamine (1 mmol, 0.126 g), phosphorous acid (6 mmol, 0.492 g), HCl (0.5 mL) and 25 mL of ethanol was refluxed for 18 h. After this time, a white solid is appeared which was filtered off and dried under vacuum to afford MHMHPA (Scheme 4). The yield of obtained catalyst was 93% (0.641 g).

# 2.2. General procedure for the synthesis of (3'-indolyl)pyrazolo[3,4-b] pyridine

3-Cyanoacetylindole was synthesized according to the previously reported procedure [39]. Then, in a 25 mL round-bottomed flask containing 5 mL of ethanol, a mixture of 3-cyanoacetylindole (1 mmol, 0.184 g), aromatic aldehyde (1 mmol), 3-methyl-1-phenyl-1*H*-pyrazol-5-amine (1 mmol, 0.173 g) and MHMHPA (5 mol%) as a catalyst, were stirred and refluxed (for an appropriate time). The Progress of the

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### Anomeric effect

### n<sub>x</sub>→σ<sup>\*</sup><sub>C-Y</sub>





**Scheme 2.** Usual anomeric interaction compares with vinylogous anomeric effect [19a].





Scheme 3. Synthesis of (3'-indolyl)pyrazolo[3,4-b]pyridines in the presence of MHMHPA.



### Scheme 4. Preparation of MHMHPA.



Scheme 5. Preparation of nanostructured melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA).



Fig. 2. UV-Vis spectrum of melamine and its corresponding catalyst (MHMHPA).



Fig. 3. FT-IR spectrum of melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA) and melamine and phosphorus acid as its starting materials.

reaction was monitored by TLC (*n*-hexane: ethyl acetate; 7:3). After completion of the reaction the precipitate was filtered off. Then, the catalyst was separated from reaction mixture by adding PEG-400 (5 mL) and centrifugation (1000 rpm) for 5 min. In the following, to remove PEG-400, water (10 mL) was added and the precipitate was filtered off and the final pure product was obtained (Scheme 3).

### 3. Results and discussion

### 3.1. Synthesis and characterization of the novel nano structure catalyst

In continuation of our investigation on the synthesis of organic molecules with indole moieties [36], herein we wish to report a new methodology for the synthesis of (3'-indolyl)pyrazolo[3,4-*b*]pyridines under mild and green conditions. Firstly, the desired nanostructured catalyst melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA) was prepared according our recently published procedure [34] *via* the reaction of melamine and paraformaldehyde in presence of HCl as catalyst (Scheme 5). The structure of MHMHPA was identified by various techniques such as UV–vis spectra, FT-IR, solid <sup>31</sup>P NMR, Mass spectra, EDX, scanning electron microscopy (SEM) with elemental mapping, transmission electron microscopy (TEM), thermogravimetry (TG) and derivative thermogravimetry (DTG) analysis.

The UV–vis spectrum of melamine and melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA) are shown in Fig. 2. The maximum absorptions of melamine and its corresponding catalyst were observed at 258 and 226 nm, respectively. The difference between  $\lambda_{max}$ of melamine and its corresponding catalyst confirmed the structure of target MHMHPA.

FT-IR spectrum of melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA), melamine and phosphorus acid are shown in Fig. 3. The absorption bands at 2400–3500 cm<sup>-1</sup> linked to the stretching vibrational modes of OH acid groups. The absorption bands at 1676 cm<sup>-1</sup> and 1517 cm<sup>-1</sup> can be assigned to C=N and C=C stretching, respectively. Also, the absorption bands at 909, 1075 and 1153 cm<sup>-1</sup> are related to P–O stretching. The FT-IR spectrum differences between starting materials and MHMHPA were confirmed the structure of catalyst.

As shown in Fig. 4, the solid <sup>31</sup>P NMR spectrum of melamine hexakis



Fig. 4. The solid <sup>31</sup>P NMR spectrum of melamine hexakis(methylene)hexakis(phosphonic acid) (MHMHPA).



Fig. 5. Mass spectrum of MHMHPA.



Fig. 6. Energy-dispersive X-ray spectroscopy (EDX) of MHMHPA.

(methylene)hexakis(phosphonic acid) (MHMHPA) showed the signal of the hydrogen-bonded in the P–OH as a single resonance at 8.11 ppm, confirming the presence of the phosphorous acid moiety in the MHMHPA. Also, mass spectrum of MHMHPA shown in Fig. 5. Observed peak in 690 m/z is related to the molecular weight of MHMHPA.

Energy-dispersive X-ray spectroscopy (EDX) and elemental mapping analysis of MHMHPA were achieved and are showing the expected elements *i.e.* carbon, nitrogen, oxygen and phosphor were confirmed in its corresponding structure (Figs. 6 and 7).

Next, the particles size, shape and morphology of MHMHPA were also studied by SEM and TEM techniques (Figs. 8 and 9). SEM micrographs of MHMHPA are showing that the particles have not completely agglomerated. It was also observed that the average size of the particles were between 29–49 nm. The TEM images of MHMHPA showed that its particles are in spherical shapes with proper dispersion.

In the direction study of thermal and behavioral stability of the melamine hexakis(methylene)hexakis(phosphonic acid), thermal gravimetric (TG), derivative thermal gravimetric (DTG) and differential thermal analysis (DTA) of the catalyst were also performed (Fig. 10). The first weight losing was happened between 25 and 100 °C,



Fig. 7. Elemental mapping images of (a) carbon (red), (b) nitrogen (green), (c) oxygen (blue) and (d) phosphor (yellow) in the structure of MHMHPA.

associated with the removal of solvent molecules (organic and water) that were absorbed in the course of the catalyst preparation. The main stage of weight losing (includes about 40% weight loss) was happened after 375 °C, which is related to disruption of the structure of MHMHPA. Therefore, this catalyst can be used up to 375 °C.

3.2. Catalytic activity of MHMHPA in the synthesis of (3'-indolyl)pyrazolo [3,4-b]pyridine derivatives

After a fully characterization of the catalyst, it was used in the synthesis of (3'-indolyl)pyrazolo[3,4-b]pyridine derivatives. Initially,



Fig. 8. SEM micrographs of the MHMHPA.



Fig. 9. TEM micrographs of the MHMHPA.

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Fig. 10. Thermal gravimetric (TG), derivative thermal gravimetric diagram (DTG) and differential thermal analysis (DTA) of MHMHPA.

the reaction of benzaldehyde (1 mmol, 0.106 g), 3-cyanoacetylindole (1 mmol, 0.184) and 3-methyl-1-phenyl-1*H*-pyrazol-5-amine (1 mmol, 0.173) was selected as model reaction for the optimization of the

reaction conditions. As shown in the Table 1, the worthy results were obtained when the reaction was achieved in the presence of 5 mol% of the catalyst (Table 1, entry 5) under refluxing ethanol. No improvement

# Table 1Condition optimization.<sup>a</sup>



Entry	Amount of Catalyst (mol %)	Solvent	Temp. (°C)	Time (h)	Yield (%) <sup>b</sup>
1	-	-	-	12	0
2	-	EtOH	Reflux	6	30
3	1	EtOH	Reflux	4.5	38
4	3	EtOH	Reflux	3.5	45
5	5	EtOH	Reflux	2	75
6	7	EtOH	Reflux	2	75
7	10	EtOH	Reflux	2	75
8	5	EtOH	rt	6	25
9	5	EtOH	50	5	43
10	5	PEG-400	110	2.5	71
11	5	H <sub>2</sub> O	Reflux	4	45
12	5	CHCl <sub>3</sub>	Reflux	5	25
13	5	EtOAc	Reflux	4.5	38
14	5	Toluene	Reflux	4	52
15	5	CH <sub>3</sub> CN	Reflux	3	45
16	5	Solvent-free	100	4	62

<sup>a</sup> The reaction was carried out in 5 mL of solvent, benzaldehyde: 1 mmol, 0.106 g, 3-methyl-1-phenyl-1*H*-pyrazol-5-amine: 1 mmol, 0.173 g and 3-(1*H*-indol-3-yl)-3-oxopropanenitrile: 1 mmol, 0.184 g.

<sup>b</sup> Isolated yield.

was detected in the yield of reaction using increasing the amount of the catalyst (Table 1, entries 6–7). Table 1 clearly displayed that in the absence of catalyst, the product was produced in a low yield (Table 1, entry 2). To investigate the solvent effect on the reaction, several solvents, such as  $H_2O$ , CHCl<sub>3</sub>, EtOAc, toluene, CH<sub>3</sub>CN, PEG-400 and EtOH, were tested and compared with solvent-free conditions in the presence of 5 mol% MHMHPA (Table 1, entries 10–16). The results showed that ethanol was the best solvent for the target reaction.

The efficiency and applicability of melamine hexakis(methylene) hexakis(phosphonic acid) were studied in a reaction of 3-cyanoacetylindole, 3-methyl-1-phenyl-1*H*-pyrazol-5-amine with various aldehydes. The results are summarized in Scheme 6. All aldehydes including benzaldehyde as well as other aromatic aldehydes possessing electronreleasing and electron withdrawing substituents, basic groups and/or halogens afforded the (3´-indolyl)pyrazolo[3,4-*b*]pyridine derivatives in high to excellent yields (71–86%) and short reaction times (90–150 min) (Scheme 6).

In a propose mechanism, aldehyde is activated with MHMHPA and intermediate (I) is prepared by the loss of one molecule of water. In the second step, 3-cyanoacetylindole reacts with intermediate (I) to gives intermediate (II) after tautomerization. Then, intermediate (II) gives intermediate (III) after intramolecular cyclization and loss another molecule of H<sub>2</sub>O. Finally, intermediate (III) converted to the desired product *via* a "cooperative vinylogous anomeric based oxidation" and releasing of molecular hydrogen (H<sub>2</sub>) [35]. It has been suggested that the latter stage supported by driving force of aromatization *via* a cooperative vinylogous anomeric (Scheme 7) [20–28]. In order to approve the abovementioned suggestion, the model reaction was

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Scheme 6. Synthesis of (3'-indolyl)pyrazolo[3,4-b]pyridine derivatives using MHMHPA as a novel nano-heterogeneous catalyst under refluxing ethanol.

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Scheme 7. Proposed mechanism for the synthesis of (3'-indolyl)pyrazolo[3,4-b]pyridines.



**Fig. 11.** Recyclability of MHMHPA as catalyst in the synthesis of 6-(1*H*-indol-3-yl)-3-methyl-1,4-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile in ethanol under reflux condition.

performed under argon atmosphere (in the absence of oxygen molecules). Under this condition, the reaction was carried out as well as air atmosphere.

The recyclability of the catalyst was also studied in the model reaction of benzaldehyde (1 mmol, 0.106 g), 3-methyl-1-phenyl-1*H*-pyrazol-5-amine (1 mmol, 0.173) and 3-cyanoacetylindole (1 mmol, 0.184) under above-mentioned optimal conditions. As indicated in the Fig. 11, the applied MHMHPA could be recycled (see experimental for details) and efficiently reused up to five reaction cycles with a marginal decreasing its catalytic activity.

To compare the efficiency of described catalyst for the synthesis of 6-(1*H*-indol-3-yl)-3-methyl-1,4-diphenyl-1*H*-pyrazolo[3,4-*b*]pyridine-5carbonitrile, we have used various organic and inorganic acid catalysts for condensation reaction between banzaldehyde (1 mmol, 0.106 g), 3methyl-1-phenyl-1*H*-pyrazol-5-amine (1 mmol, 0.173) and 3-cyanoacetylindole (1 mmol, 0.184) under refluxing ethanol conditions. As Table 2 indicated, MHMHPA is the best of choice for the synthesis of (3'-indolyl)pyrazolo[3,4-*b*]pyridines; due to the shorter reaction times, higher yields and amount of applied catalyst.

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#### Table 2

Evaluation of various catalyst for the synthesis of (3'-indolyl)pyrazolo[3,4-b]pyridines in comparison with MHMHPA in ethanol and under reflux conditions.

Entry	Catalyst	Reaction condition	Time (min)	Yield (%)	Ref.
1	MHMHPA (5 mol%)	EtOH, reflux	120	75	а
2	[Bu <sub>4</sub> N]OH (10 mol%)	EtOH, reflux	120	54	а
3	Fe <sub>3</sub> O <sub>4</sub> @SiO <sub>2</sub> @ PrNH <sub>2</sub> (5 mg)	EtOH, reflux	120	49	а
4	ZnCl <sub>2</sub> (10 mol%)	EtOH, reflux	120	30	а
5	[Msim]ZnCl <sub>3</sub> (10 mol%)	EtOH, reflux	120	57	а
6	TrCl (10 mol%)	EtOH, reflux	120	35	а
7	p-TSA (10 mol%)	EtOH, reflux	120	53	а
8	Al(HSO <sub>4</sub> ) <sub>3</sub> (10 mol%)	EtOH, reflux	120	56	а
9	[Py-SO <sub>3</sub> H]Cl (10 mol%)	EtOH, reflux	120	51	а
10	SSA (10 mg)	EtOH, reflux	120	55	а
11	H <sub>3</sub> PO <sub>4</sub> (10 mol%)	EtOH, reflux	120	48	а
12	HCl (10 mol%)	EtOH, reflux	120	41	а
13	H <sub>2</sub> SO <sub>4</sub> (10 mol%)	EtOH, reflux	120	43	а
14	Oxalic acid (10 mol%)	EtOH, reflux	120	37	а
15	HNO <sub>3</sub> (10 mol%)	EtOH, reflux	120	46	а
16	NaHSO <sub>4</sub> (10 mol%)	EtOH, reflux	120	38	а
17	_	Glycol, MW, 250 w,	10	82	[37]
		150 °C			
18	-	Glycol, 150 °C	11 h	70	[37]
19		MW, 300 w, 200 °C	9	62	[38]

<sup>a</sup> This work.

#### 4. Conclusion

In summary, we have synthesized melamine hexakis(methylene) hexakis(phosphonic acid) (MHMHPA) as novel nano-heterogeneous catalyst and characterized by several techniques such as FT-IR, Uv–vis, solid <sup>31</sup>P NMR, Mass spectra, SEM, EDX, TEM, TG and DTG analysis. The described catalyst was applied for the synthesis of (3'-indolyl) pyrazolo[3,4-*b*]pyridines *via* a vinylogous anomeric based oxidation mechanism. Short reaction time, mild and clean profile of reaction, recyclability of the catalyst and the use of green solvent are the salient features of the described method. So the present catalyst can open up a promising insight in the course of rational design, synthesis and applications of novel and task-specific melamine-based polymers with phosphonate linkages.

### **Declaration of Competing Interest**

There are no conflicts to declare.

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### Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.mcat.2019.110666.

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