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An efficient one-pot three-component synthesis of pyrido[2,3-d]pyrimidine derivatives in the presence of nano silica-supported Preyssler $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ as a green and reusable catalyst

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One-pot reaction.

Abstract. An efficient and mild condensation reaction of 6-aminouracil derivatives with various aromatic aldehydes and ethyl or methyl acetoacetate was performed in the presence of catalytic amounts of $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ as a recyclable, eco-friendly, and green nano-catalyst to afford the desired pyrido[2,3-d]pyrimidine derivatives in good to excellent yields.

1. Introduction

Green chemistry is the key developing field providing us with a promising path for the future technologies. It also carries out chemical processes in a way that significantly reduces, or eliminates, all the hazardous substances [1]. In recent years, using water in chemical reactions has been considered as a desirable, safe, inexpensive, and environmentally benign solvent rather than organic solvents [2].

Pyrido[2,3-*d*]pyrimidine derivatives are classified as an important category of heterocyclic compounds with a diverse range of biological properties [3-5] such as being antiviral, calcium channel antagonism [6], adenosine kinase inhibition [7], and tyrosine phosphatase inhibition [8]. Moreover, these key heterocyclic cores are applied as Fibroblast Growth Factor Receptor (FGFR3) tyrosine kinase inhibitors [9]. The aforementioned admirable characteristics have attracted much attention of chemists towards the synthesis of pyrido[2,3-*d*]pyrimidine derivatives. As a result, some methods for the preparation of these key compounds using different types of catalysts, such as nano-Fe_3O_4 [10] and thiourea dioxide [11], or in different conditions without catalyst [12-14] have been reported. Multi-step procedures, high reaction times, toxic solvents, and the use of homogeneous catalysts are some drawbacks of the previously reported pathways. Thus, the development of novel methods based upon eco-friendly and recyclable

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catalytic systems is an important task for organic chemists.

Over the last few years, there has been tremendous growth in the use of heteropolyacids, *HPAs*, as environmentally friendly catalysts due to their unique properties such as excellent acidity, ease of handling, high thermal stability, low cost, simple work-up, and recyclability [15]. Among the solid heteropolyacids, Preyssler with 14 acidic protons and unique hydrolytic stability is an efficient and effective supper acid catalyst for organic reactions [16-18]. Also, immobilization of Preyssler on the appropriate nano-SiO_2 support can develop its catalytic properties by enhancing the specific surface area of the catalyst [16]. Hence, the synthesis and catalytic activities of the nano-silica supported Preyssler, *HPA*, catalysts have been largely overlooked [19-25]. In continuation of our previous efforts to develop environmentally benign protocols using solid acid catalysts [26-31], here, we wish to report the catalytic activity of $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ as a Preyssler type *HPA* in the one-pot synthesis of pyrido[2,3-*d*]pyrimidine derivatives **1** by condensation of 6-aminouracil derivatives **2** with aromatic aldehydes **3** and appropriate 1,3-diketone **4** in aqueous media under reflux conditions (Scheme 1).

2. Results and discussion

2.1. Synthesis and characterization of nano $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$

The nano silica-supported Preyssler was prepared by micro emulsion method and its structure has been well characterized by Transmission Electron Microscopy (TEM) as illustrated in Figure 1 [19].

The immobilization of HPA, $H_{14}[NaP_5W_{30}O_{110}]$, onto the SiO_2 nano particles was proven using infrared spectroscopy technique (Figure 2) [19]. As it can be seen, the asymmetric stretching frequency of all oxygens is identical with that of C_5V symmetry anion. The presence of these bands confirm that $H_{14}[NaP_5W_{30}O_{110}]$ is actually present in the HPA/SiO_2 nano particles. Furthermore, the protonated water of $H_{14}[NaP_5W_{30}O_{110}]$ has also been remained in the nano particles proven by the presence of a band at 1730 cm^{-1}. Thus, the immobilization of the heteropolyacid $H_{14}[NaP_5W_{30}O_{110}]$ onto the SiO_2 nano particles looks unprecedented. Due to un-reactivity of

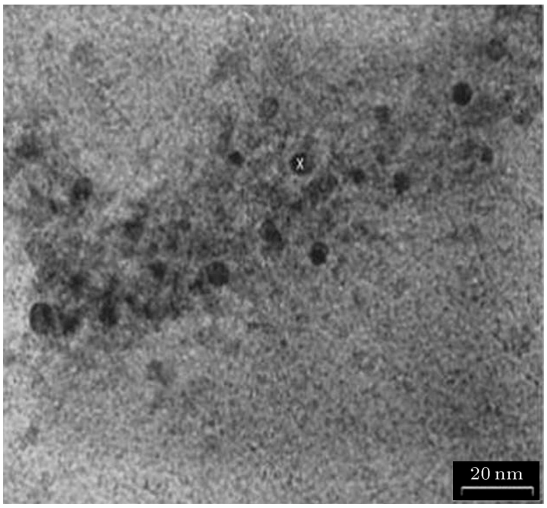


Figure 1. TEM image of the synthesized nano $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$.

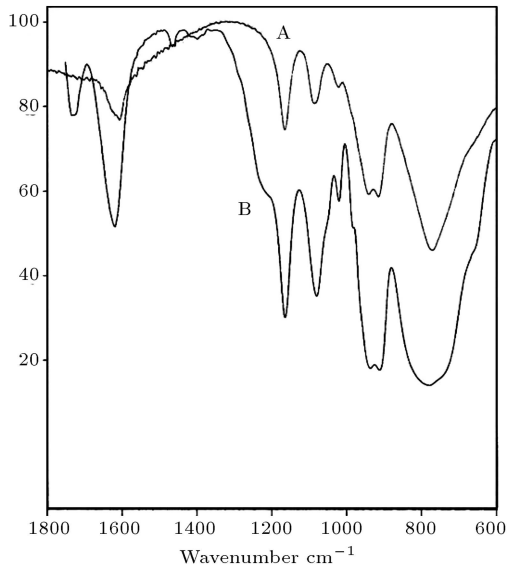
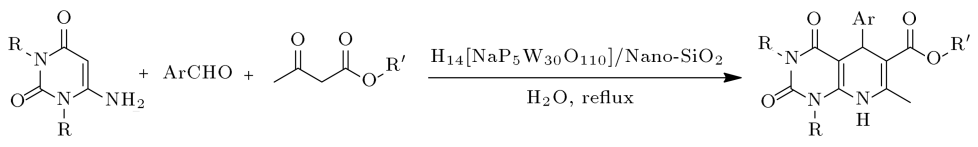


Figure 2. IR spectrum of Preyssler catalyst in nano form (A) and bulk form (B).

heteropolyacid towards tetraethoxysilane or with water in the micro emulsion, the structure of silica nano particles is not affected.

2.2. Synthesis of pyrido[2,3-d]pyrimidine derivatives

For the synthesis of pyrido[2,3-*d*]pyrimidine derivative compounds **1**, the reaction of 6-amino-1,3-



Scheme 1. Synthesis of pyrido[2,3-*d*]pyrimidine derivatives **1** via a three-component reaction.

Table 1. Effect of various amounts of $H_{14}[NaP_5W_{30}O_{110}]$/Nano-$SiO_2$, solvents, and temperature on the synthesis of **1a**.

Entry	Catalyst amount (g)	Solvent	Temperature	Time (min)	Yield (%)[a]
1	0.01	H_2O	Reflux	35	87
2	0.02	H_2O	Reflux	20	94
3	0.03	H_2O	Reflux	20	94
4	0.02	—	80	180	Trace
5	0.02	H_2O	50	140	56
6	0.02	EtOH	Reflux	35	85
7	0.02	CH_2Cl_2	Reflux	130	Trace
8	0.02	CH_3CN	Reflux	60	79
9	0.02	H_2O/EtOH (1:1)	Reflux	45	81

[a]: Yields of isolated pure products.

Table 2. $H_{14}[NaP_5W_{30}O_{110}]$/Nano-$SiO_2$ catalyzed three-component one-pot reaction for the preparation of pyrido[2,3-d]pyrimidine derivatives.

Entry	Ar	R	R′	Product	Time (min)	Yield (%)[a]	Found M.p.	Reported M.p.
1	C_6H_5	Me	Et	1a	20	94	299-300	298-299 [8]
2	4-$OMeC_6H_4$	Me	Et	1b	25	89	268-269	267-269 [8]
3	4-OHC_6H_4	Me	Et	1c	30	90	240-242	240-242 [11]
4	3-$O_2NC_6H_4$	Me	Et	1d	10	86	258-259	256-258 [10]
5	4-FC_6H_4	Me	Et	1e	35	91	240-241	240-241 [10]
6	4-OHC_6H_4	Me	Me	1f	45	85	245-247	244-246 [11]
7	3-$O_2NC_6H_4$	Me	Me	1g	30	80	275-277	273-274 [10]
8	C_6H_5	H	Et	1h	60	87	>320	New

[a]: Yields of isolated pure products.

dimethyluracil **2a**, benzaldehyde **3a**, and ethyl acetoacetate **4a** in the presence of nano $H_{14}[NaP_5W_{30}O_{110}]$ /SiO_2 was firstly chosen as a model reaction. In order to obtain the best reaction condition, we examined the effect of the catalyst by using different amounts of $H_{14}[NaP_5W_{30}O_{110}]$/$SiO_2$ (0.01, 0.02, and 0.03 g), as summarized in Table 1. Based on the results in entries 1-3, the optimum yield of the product **1a** was achieved when 0.02 g of HPA was used (entry 2). Among the tested solvents such as water, ethanol, dichloromethane, acetonitrile, and mixture of water/ethanol (1:1), and the solvent-free system (Table 1, entries 4-9), the best result was obtained in water under reflux condition.

In order to establish the scope and generality of this procedure, various aromatic aldehydes and 1,3-diketone were examined under the optimized reaction condition (Table 2). As illustrated in Table 2, both electron donating and electron withdrawing substituents on the aromatic aldehydes afforded the corresponding pyrido[2,3-*d*]pyrimidines. In all cases, the desired product was isolated after a simple filtration and recrystallization from ethanol in 80-94 yields.

To study reusability of the catalysts, it was separated by filtration in recrystallization step and thoroughly washed with diethyl ether, and then dried at 100°C under reduced pressure. The recycled catalyst can be reused in three consecutive model reactions without any significant loss of activity (Table 3).

Table 3. Reusability of the catalyst in the synthesis of **1a**.

Entry	Number of recycle	Time (min)	Yield (%)[a]
1	Fresh	20	94
2	1	20	94
3	2	20	91

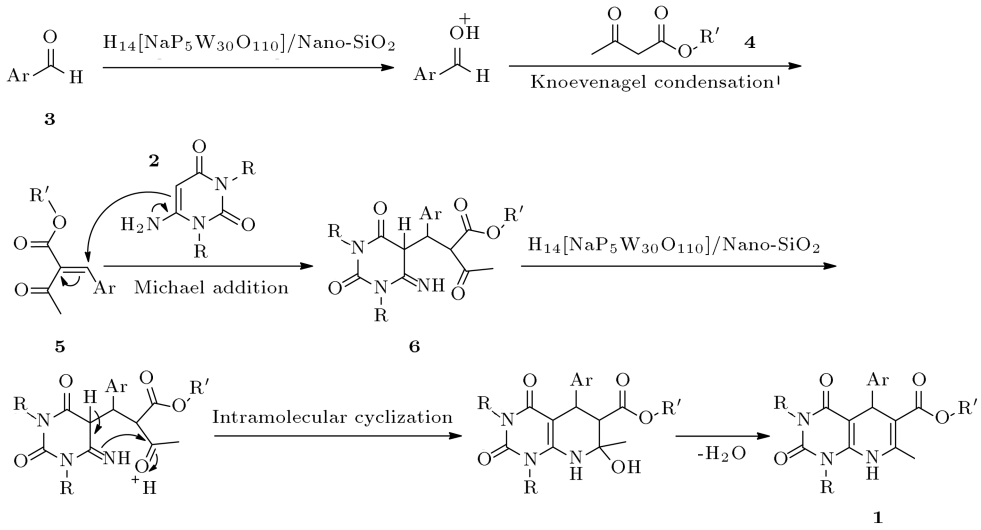
[a]: Yields of isolated pure products.

The synthesis of pyrido[2,3-*d*]pyrimidine **1a** has been investigated in several conditions in literature, as shown in Table 4. In comparison with other existing methods, the present methodology has several advantages such as a simple procedure, short reaction time, and excellent yields with high purity of products.

The plausible mechanism for this reaction is illustrated in Scheme 2. First, the Knoevenagel condensation of **3** and **4** in the presence of nano catalyst $H_{14}[NaP_5W_{30}O_{110}]$/$SiO_2$ produces the intermediate **5**,

Table 4. Comparison of different conditions in the synthesis of **1a**.

Entry	Catalyst	Solvent	Condition	Time (h)	Yield (%)	Year
1	Nano $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$	H_2O	Reflux	0.33	94	This work
2	Thiourea dioxide (TUD)	H_2O	50°C, N_2	8	92	2012 [11]
3	Nano Fe_3O_4@SiO_2–SO_3H	H_2O	70°C	0.5	92	2013 [10]



Scheme 2. The plausible mechanism for the synthesis of pyrido[2,3-d]pyrimidines **1**.

which reacts with compound **2** by Michael addition to obtain the intermediate **6**. Subsequently, intramolecular cyclization and dehydration on intermediate **6** yield the final product **1**.

3. Conclusion

In summary, herein, we reported a green, simple, and efficient one-pot synthesis of pyrido[2,3-*d*]pyrimidine derivatives by readily available starting materials. Furthermore, using nano-silica supported Preyssler offers several advantages including the use of recyclable catalyst, simple procedure, high yields, and easy work-up.

4. Experimental

4.1. Materials and methods

All the solvents and reagents were purchased from Aldrich and Merck companies with high-grade quality, and used without further purification. All products were characterized by their spectra and physical data in comparison with those of the authentic samples already reported in literatures. Melting points were determined in capillary tubes on a Barnstead Electrothermal 9200 apparatus. IR spectra were recorded using KBr disks on a FT-IR Bruker Tensor 27 instrument. ^{1}H NMR and ^{13}C NMR spectra were obtained on a Bruker AQS AVANCE-400 MHz spectrometer, using DMSO as the solvent and TMS as an internal standard. Mass spectra were recorded using a 5973 Network Mass Selective Detector operating at ionization potential of 70 eV. Elemental analyses were performed on a ThermoFinigan Flash EA 1112 series elemental analyzer.

4.2. Preparation of the catalyst

Nano catalyst $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ was prepared in accordance with our procedure, previously reported [19], as follows: to a solution of sodium bis(2-ethylhexyl)sulfosuccinate (being used as surfactant) in cyclohexane (0.2 M), a solution of Preyssler heteropoly-acid in a specified volume of water was gradually added. The optimized molar ratios of water to surfactant had already been found to be 3, 5, and 7. Then, tetraethoxysilane was added into this microemulsion phase. The reaction mixture was stirred for different times (8, 12, 18, 25, and 30 h) at ambient temperature. Next, the obtained dispersed Preyssler acid/SiO_2 nano structures were centrifuged (15,000 rpm) and the nano particles were dipped in acetone, filtered and dried in a vacuum oven at 80°C.

4.3. General procedure for the synthesis of pyrido[2,3-d]pyrimidine derivatives (1a-h)

A mixture of aromatic aldehyde **3** (1 mmol), 1,3-diketone **4** (1 mmol), 6-aminouracil derivatives **2** (1 mmol), and $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ (0.02 g) in H_2O (4 ml) was stirred under reflux condition for the appropriate time. The progress of the reaction was monitored by TLC using *n*-hexane/ethyl acetate (1:5)

as eluent. After completion of the reaction, the mixture was cooled to room temperature. The solid product was collected by filtration, washed with water and ethanol, and purified by recrystallization from ethanol.

4.4. Recycling of the catalyst

Nano silica-supported Preyssler $H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ is not soluble in hot ethanol and could be recycled in the recrystallization step by filtration. The recovered catalyst was washed with diethyl ether, and then dried at 100°C under reduced pressure. The catalyst was reused in three consecutive model reactions and the yield of the products showed no significant change.

Ethyl 7-methyl-2,4-dioxo-5-phenyl-1,2,3,4,5, 8-hexahydropyrido[2,3-*d*]pyrimidine-6-carboxylate (1h):

White Solid, **M.p.** > 320°C; **IR (KBr):** $\nu_{max} = 3267$, 3226, 3089, 3023, 2816, 1714, 1660, 1527, 1209 cm^{-1}; **^{1}HNMR (DMSO-d_6, 400 MHz):** δ_H = 1.109 (t, J = 7 Hz, 3H, CH_3), 2.313 (s, 3H, CH_3), 3.972 (q, J = 7 Hz, 2H, CH_2), 4.794 (s, 1H, CH), 7.091-7.236 (m, 5H, H_{Ar}), 8.526 (s, 1H, NH), 10.009 (s, 1H, NH), 10.716 (s, 1H, NH) ppm; **^{13}CNMR (DMSO-d_6, 400 MHz)**: δ = 14.6, 18.9, 36.8, 59.6, 88.9, 103.5, 126.4, 127.9, 128.3, 128.8, 129.7, 147.8, 163.3, 163.4, 167.0 ppm; **EIMS m/z**: 327 (M^+); **Anal. Calcd (%) for $C_{17}H_{17}N_3O_4$:** C, 62.38; H, 5.23; N, 12.84. Found: C, 62.35; H, 5.27; N, 12.88.

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Majid Momahed Heravi was born in 1952 in Mashhad, Iran. He received his BSc degree from the National University of Iran in 1975 and his MSc and PhD degrees from Salford University, England, in 1977 and 1980. He completed his doctoral thesis under supervision of late Jim Clarck in Salford University, England. He started his career as a research fellow in Daroupakhsh (a pharmaceutical company) in 1981, Tehran, Iran, and joined as an Assistant Professor to Ferdowsi University of Mashhad, Iran, in 1983 and was promoted to Associate Professor in 1993 and Full Professor in 1997 in the aforementioned university. In 1999, he moved to Alzahra University of Tehran, Iran, as Professor of Chemistry where he is still working in. He has previously been a visiting professor at UC

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