

# Silica-supported Preyssler Nanoparticles as New Catalysts in the Synthesis of 4(3H)-Quinazolinones

Majid M. Heravi<sup>a\*</sup>, Samaher Sadjadi<sup>a</sup>, Sodeh Sadjadi<sup>a</sup>, Hossein A. Oskooie<sup>a</sup>,  
R. Hekmat Shoar<sup>a</sup> and Fatemeh F. Bamoharram<sup>b</sup>

<sup>a</sup>Department of Chemistry, School of Sciences, Azzahra University, Vanak, Tehran, Iran.

<sup>b</sup>Department of Chemistry, School of Sciences, Islamic Azad University, Mashad Branch, Mashad, Iran.

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## ABSTRACT

A new and efficient method for the preparation of 4(3H)-quinazolinones from the condensation of anthranilic acid, orthoester and substituted anilines, in the presence of catalytic amounts of silica-supported Preyssler nanoparticles is reported. The catalyst performs very well in comparison with other catalysts reported before. An important advantage of this catalyst is the ease of separating it from the reaction mixture, as well as the fact that it could be recycled a number of times.

## KEYWORDS

4(3H)-Quinazolinones, recyclable catalyst, silica-supported Preyssler nanoparticles, heteropolyacids.

## 1. Introduction

The quinazolinone core and its derivatives form an important class of bioactive molecules, with useful therapeutic and pharmacological properties, such as anti-inflammatory, anti-convulsant, antihypertensive and antimalarial activity.<sup>1</sup> Several bio-active natural products including febrifugine and iso-febrifugine contain a quinazolinone moiety and possess antimalarial activity.<sup>2,3</sup>

Many reagents have been reported in the literature<sup>4</sup> for the synthesis of 4(3H)-quinazolinone derivatives. However, many of these methodologies are associated with several shortcomings such as multi-step procedures, long reaction times, expensive reagents, harsh conditions, low product yields, occurrence of several side-products and difficulty in recovery and reusability of the catalysts.

The demand for increasingly clean and efficient chemical synthesis is important from both the economic and environmental points of view,<sup>5</sup> so more attempts to find green and economical synthetic methods are necessary.

The catalytic function of heteropolyacids (HPAs) and related polyoxometalate compounds has attracted much attention, particularly in the last two decades.<sup>6</sup> Polyoxometalates (POMs) are a class of molecularly defined organic metal-oxide clusters; they possess intriguing structures and diverse properties.<sup>7</sup> These compounds exhibit high activity in acid-base type catalytic reactions, hence they are used in many catalytic areas as homogeneous and heterogeneous catalysts.

Numerous attempts to modify the catalytic performance of heteropolyacids, such as supporting them on mobile composition of matter (MCM), silica gel and others have been reported.<sup>8</sup>

The application of Preyssler catalysts is highly limited and only a few examples of catalytic activity have been reported.<sup>9</sup> The important advantages of this heteropolyacid are: strong Brønsted acidity with 14 acidic protons, high thermal stability, high hydrolytic stability (pH 0–12), reusability, safety, quantity of waste, ease of separation, corrosiveness, high oxidation potential, and application as a green reagent along with an exclusive

structure. All these characteristics have attracted much attention in the recent literature.<sup>10,11</sup>

Over the last decade, due to the unique properties of nanoparticles along with their novel properties and potential applications in different fields,<sup>12</sup> the synthesis and characterization of catalysts with lower dimension has become an active topic of research. As the particle size decreases, the relative number of surface atoms increases, and thus activity increases. Moreover, due to quantum size effects, nanometre-sized particles may exhibit unique properties for a wide range of applications.<sup>13</sup>

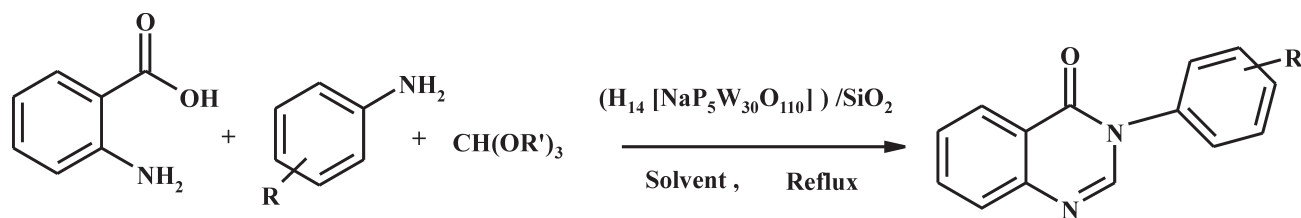
In spite of extensive investigations on Keggin-type nanocatalysts,<sup>14</sup> the synthesis of Preyssler-type nanocatalysts has been largely overlooked. Recently we have explored the application of a Preyssler catalyst in various organic reactions.

In our attempt to use heteropolyacids as catalysts in organic reactions, we reported that Preyssler-type heteropolyacids,  $H_{14}[NaP_5W_{30}O_{110}]$ , show good catalytic reactivity.<sup>15</sup> Considering many reports on the modification of heteropolyacids by supporting them on silica gel,<sup>8</sup> and due to the unique properties of nanoparticles along with their novel properties and potential applications in different fields,<sup>12</sup> we decided to immobilize  $H_{14}[NaP_5W_{30}O_{110}]$  onto the  $SiO_2$  nanoparticles. It was hoped that this would modify the catalytic activity of the Preyssler-type heteropolyacid,  $H_{14}[NaP_5W_{30}O_{110}]$ . This would hopefully enable us to investigate the catalytic behaviour of this proposed new catalyst in the synthesis of 4(3H)-quinazolinones. The proposed reaction is a one-pot reaction of anthranilic acid, orthoester and substituted anilines, in the presence of silica-supported Preyssler nanoparticles as a new and efficient catalyst (Scheme 1).

## 2. Results and Discussion

Silica-supported Preyssler nanostructures were obtained through a microemulsion method. Although some authors have used this procedure, this method has never been reported for the synthesis of Preyssler nanostructures with different morphologies. In addition, in the same reactions, only spherical nanoparticles have been obtained.

\* To whom correspondence should be addressed. E-mail: mmh1331@yahoo.com



Scheme 1

**Table 1** Synthesis of 4(3H)-quinazolinones using 0.03 mmol silica-supported Preyssler nanoparticles under refluxing conditions in 5 mmol CH<sub>3</sub>CN at 82 °C.

Entry	R	R'	Time/min	Yield/% <sup>a</sup>	M.p./°C	Lit. m.p./°C <sup>17</sup>
1	H	C <sub>2</sub> H <sub>5</sub>	15	98	138	139
2	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>	15	98	147	147
3	4-Cl	C <sub>2</sub> H <sub>5</sub>	25	94	181	182
4	4-Br	C <sub>2</sub> H <sub>5</sub>	25	91	185	186
5	H	CH <sub>3</sub>	25	96	139	139
6	CH <sub>3</sub>	CH <sub>3</sub>	25	97	147	147
7	4-Cl	CH <sub>3</sub>	35	92	182	182
8	4-Br	CH <sub>3</sub>	35	90	186	186

<sup>a</sup> Yields refer to isolated products.

The morphology of the Preyssler nanostructures was found to depend strongly on the reaction conditions, such as concentration and time. The sizes and morphology of the products were controlled by changing the water:sodium bis(2-ethylhexyl) sulphosuccinate molar ratio/s and the reaction times. For short reaction times, the tubular structure was found to prevail, whereas spherical shapes dominated for longer times.

Spherical particles of about 20 nm diameter were obtained at a molar ratio (water to sulphosuccinate) of 3:1 after 30 h, while the tubular morphology was obtained at a molar ratio of 3:1 and various times ranging from 12 h up to about 30 h. The molar ratio has been studied in various ranges and the results showed that higher molar ratios are unfavourable. The samples were analysed by tunneling electron microscopy (TEM). A mixture of nanowire (tubular shape) and nanospherical structures was obtained at ratio 3:1 and 12 h. The fraction of tubular shapes increased up to about 18 h.

The reason is not yet clear, but this is not surprising. A shape change of the particles has been observed in other synthetic methods of nanoparticle preparation.<sup>16</sup> The reason for this can be attributed to metastable states, which could spontaneously change under equilibrium reaction conditions, which is in agreement with previous observations.<sup>16</sup> Scanning electron microscopy (SEM) pictures of samples and X-ray diffraction (XRD) patterns of the synthesized samples were taken. The patterns of the spherical synthesized products contain a broad peak centered at 52 Å. Analogous diffraction patterns have been observed for other synthesized samples.

The heteropolyacid (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>]) on the SiO<sub>2</sub> nanoparticles was confirmed by infrared (IR) spectroscopy. IR spectroscopy demonstrates that (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>]) is preserved in the HPA/SiO<sub>2</sub> nanoparticles. The antisymmetric stretching wavenumber of the terminal oxygen-containing group is observed at 960 cm<sup>-1</sup> and the antisymmetric P-O stretching wavenumber is noted at 1080 and 1165 cm<sup>-1</sup>. The prominent P-O bands at 960, 1080 and 1165 cm<sup>-1</sup> are consistent with a C<sub>5v</sub> symmetry anion. It could therefore be confirmed that the heteropolyacid (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>]) was successfully immobilized onto the SiO<sub>2</sub> nanoparticles.

TEM and IR studies showed that the heteropolyacid stayed

intact on the nanoparticles after it was recycled several times in the reaction reported below. Bleeding of the heteropolyacid was found to be negligible by weighing the catalyst again after it was recycled five times.

The results of the synthesis of 4(3H)-quinazolinones in the presence of silica-supported Preyssler nanoparticles are reported in Table 1. Lower yields and longer reaction times were obtained for anilines with electron-withdrawing groups, namely 4-bromo-aniline and 4-chloro-aniline. It is presumed that the electron-withdrawing groups on aniline may reduce the nucleophilicity of aniline.

To compare the catalytic effect of the normal Preyssler catalyst with the silica-supported Preyssler nanoparticles in the synthesis of 4(3H)-quinazolinones a control experiment was carried out for both catalysts under the same conditions. The results of this comparison are reported in Table 2.

It is clear that in these reactions the efficiency of (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>])/SiO<sub>2</sub> is slightly higher than that of the conventional Preyssler catalyst.

Preyssler's anion has the formula [NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>]<sup>14-</sup> and has an unusual 5-fold symmetry. It is formed by fusion of five {PW<sub>6</sub>O<sub>22</sub>} groups. The central sodium ion lies not on the equator of the anion but in a plane roughly defined by the oxygen atoms of the phosphate groups. The sodium cation is non-labile on the NMR

**Table 2** Comparison of the efficiency of Preyssler and (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>])/SiO<sub>2</sub> catalysts (0.3 mol%) in the synthesis of 4(3H)-quinazolinones under refluxing conditions in 5 mmol CH<sub>3</sub>CN at 82 °C.

Entry	R	R'	Catalyst	Yield/% <sup>a</sup>
1	H	C <sub>2</sub> H <sub>5</sub>	(H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ])/SiO <sub>2</sub>	98
2	H	C <sub>2</sub> H <sub>5</sub>	Preyssler	95
3	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>	(H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ])/SiO <sub>2</sub>	98
4	CH <sub>3</sub>	C <sub>2</sub> H <sub>5</sub>	Preyssler	94
5	4-Cl	C <sub>2</sub> H <sub>5</sub>	(H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ])/SiO <sub>2</sub>	94
6	4-Cl	C <sub>2</sub> H <sub>5</sub>	Preyssler	90
7	4-Br	C <sub>2</sub> H <sub>5</sub>	(H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ])/SiO <sub>2</sub>	91
8	4-Br	C <sub>2</sub> H <sub>5</sub>	Preyssler	88

<sup>a</sup> Yields refer to isolated products.

time scale, and appears to be essential for the anion synthesis. The presence of the sodium cation reduces the overall anion symmetry from  $D_{3h}$  to  $C_{3v}$ .<sup>18</sup> Infrared spectroscopy shows that in  $(H_{14}[NaP_5W_{30}O_{110}])/SiO_2$  particles, the heteropolyacid structure  $(H_{14}[NaP_5W_{30}O_{110}])$  is preserved. It is therefore expected that  $(H_{14}[NaP_5W_{30}O_{110}])/SiO_2$  will exhibit the same catalytic characteristics of classical Preyssler catalysts. As the particle size of the nanomaterial decreases, the relative number of surface atoms increases, and thus activity increases. Moreover, due to quantum size effects, nanometre-sized particles can exhibit unique properties.

To obtain the optimum amount of catalyst for this reaction, various amounts of catalysts for the reaction were used. In Table 3 the results of various amounts of catalysts are summarized.

The reusability of the catalyst was also investigated. At the end of the reaction, the catalyst was recovered by a simple filtration. The recycled catalyst was washed with dichloromethane and subjected to a second run of the reaction process. To ensure that the catalyst did not dissolve in solvent the filtered catalysts were weighed before reusing. The results show that this catalyst is not soluble in solvent, and that bleeding at most is minimal.

In Table 4 a comparison of the efficiency of this catalyst in the synthesis of 4(3H)-quinazolinones after recycling five times is reported. As shown in Table 4 the yields of the reaction after using this catalyst five times show only a slight reduction.

To show the efficiency of this method for the synthesis of

**Table 3** The results of using different amounts of silica-supported Preyssler nanoparticles in the synthesis of derivatives of 4(3H)-quinazolinones under refluxing condition in 5 mmol  $CH_3CN$  at 82 °C.

Entry	R	R'	Time/min	Catalyst amount/ mol%	Yield/% <sup>a</sup>
1	H	$C_2H_5$	25	0.1	95
2	H	$C_2H_5$	15	0.3	98
3	H	$C_2H_5$	15	0.5	98
4	$CH_3$	$C_2H_5$	20	0.1	94
5	$CH_3$	$C_2H_5$	15	0.3	98
6	$CH_3$	$C_2H_5$	15	0.5	98
7	4-Cl	$C_2H_5$	35	0.1	91
8	4-Cl	$C_2H_5$	25	0.3	94
9	4-Cl	$C_2H_5$	25	0.5	94.3
10	4-Br	$C_2H_5$	25	0.1	88
11	4-Br	$C_2H_5$	25	0.3	91
12	4-Br	$C_2H_5$	25	0.5	91

<sup>a</sup> Yields refer to isolated products.

4(3H)-quinazolinones, a comparison of the yields of different catalysts and conditions for the synthesis of 3-phenyl-4(3H)-quinazolinone from the reaction of anthranilic acid, orthoesters and aniline from the literature was done. The results are reported in Tables 5 and 6. As shown in these tables various kinds of heteropolyacids and Lewis acids have been reported for synthesis of 3-phenyl-4(3H)-quinazolinone. It is clear that this silica-

**Table 4** A comparison of the efficiency of silica-supported Preyssler nanoparticles (0.03 mmol) in the synthesis of 4(3H)-quinazolinones under refluxing condition in 5 mmol  $CH_3CN$  at 82 °C upon recycling of the catalyst.

Entry	R	R'	Yield after different times of recycling/% <sup>a</sup>				
			First	Second	Third	Fourth	Fifth
1	H	$C_2H_5$	98	94	92	90	88
2	$CH_3$	$C_2H_5$	98	97	95	93	92
3	4-Cl	$C_2H_5$	94	92	90	87.3	85
4	4-Br	$C_2H_5$	91	89	87.6	85	84
5	H	$CH_3$	96	95	93.7	92	91
6	$CH_3$	$CH_3$	97	95	93	92	90
7	4-Cl	$CH_3$	92	90	88	87	85.2
8	4-Br	$CH_3$	90	88	86	84.5	83

<sup>a</sup> Yields refer to isolated products.

**Table 5** Comparison of other catalysts used in the synthesis of 3-phenyl-4(3H)-quinazolinone from anthranilic acid,  $CH(OCH_3)_3$  and aniline with silica-supported Preyssler nanoparticles as catalyst.

Entry	Condition	Catalyst	Time/min	Temperature/°C	Solvent	Yield/%
1	reflux	$(H_{14}[NaP_5W_{30}O_{110}])/SiO_2$	15	82	$CH_3CN$	96
2	stirring	$Bi(TFA)_3[nbp]FeCl_4$	10	60	no solvent	94 <sup>5</sup>
3	stirring	$La(NO_3)_3 \cdot 6H_2O$	5	25	no solvent	98 <sup>3</sup>
4	stirring	Silica gel/ $FeCl_3$	5	20	no solvent	98 <sup>19</sup>
5	reflux	$PW_{12}$	120	110	toluene	70 <sup>17</sup>
6	microwave	$PW_{12}$	13	–	no solvent	57 <sup>17</sup>
7	microwave	$PW_{12}$	13	–	2-ethoxyethanol	70 <sup>17</sup>
8	reflux	$SiW_{12}$	120	110	toluene	63 <sup>17</sup>
9	microwave	$SiW_{12}$	13	–	no solvent	48 <sup>17</sup>
10	microwave	$SiW_{12}$	13	–	2-ethoxyethanol	65 <sup>17</sup>
11	reflux	$PMo_{12}$	120	110	toluene	54 <sup>17</sup>
12	microwave	$PMo_{12}$	13	–	no solvent	43 <sup>17</sup>
13	microwave	$PMo_{12}$	13	–	2-ethoxyethanol	65 <sup>17</sup>
14	reflux	$SiMo_{12}$	120	110	toluene	43 <sup>17</sup>
15	microwave	$SiMo_{12}$	13	–	no solvent	47 <sup>17</sup>
16	microwave	$SiMo_{12}$	13	–	2-ethoxyethanol	60 <sup>17</sup>

**Table 6** Comparison of yields of other catalysts used in the synthesis of 3-phenyl-4(3*H*)-quinazolinone from reaction of anthranilic acid, CH(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub> and aniline with silica-supported Preyssler nanoparticles as catalyst.

Entry	Condition	Catalyst	Time/min	Temperature/°C	Solvent	Yield/%
1	reflux	(H <sub>14</sub> [NaP <sub>5</sub> W <sub>30</sub> O <sub>110</sub> ])/SiO <sub>2</sub>	15	82	CH <sub>3</sub> CN	98
2	reflux	PW <sub>12</sub>	120	110	toluene	75 <sup>17</sup>
3	microwave	PW <sub>12</sub>	13	–	no solvent	70 <sup>17</sup>
4	microwave	PW <sub>12</sub>	13	–	2-ethoxyethanol	75 <sup>17</sup>
5	reflux	SiW <sub>12</sub>	120	110	toluene	68 <sup>17</sup>
6	microwave	SiW <sub>12</sub>	13	–	no solvent	55 <sup>17</sup>
7	microwave	SiW <sub>12</sub>	13	–	2-ethoxyethanol	68 <sup>17</sup>
8	reflux	PMo <sub>12</sub>	120	110	toluene	56 <sup>17</sup>
9	microwave	PMo <sub>12</sub>	13	–	no solvent	55 <sup>17</sup>
10	microwave	PMo <sub>12</sub>	13	–	2-ethoxyethanol	65 <sup>17</sup>
11	reflux	SiMo <sub>12</sub>	120	110	toluene	49 <sup>17</sup>
12	microwave	SiMo <sub>12</sub>	13	–	no solvent	50 <sup>17</sup>
13	microwave	SiMo <sub>12</sub>	13	–	2-ethoxyethanol	60 <sup>17</sup>
14	stirring	La(NO <sub>3</sub> ) <sub>3</sub> ·6H <sub>2</sub> O	5	–	no solvent	97 <sup>3</sup>
15	stirring	Silica gel/FeCl <sub>3</sub>	5	25	no solvent	97 <sup>19</sup>

supported heteropolyacid shows better efficiency with lower reaction times and temperatures. The other kinds of heteropolyacids were used under refluxing conditions. The Lewis acids used in this reaction showed almost the same or slightly lower efficiency, compared with (H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>])/SiO<sub>2</sub>. There have been no reports about the reusability of La(NO<sub>3</sub>)<sub>3</sub>·6H<sub>2</sub>O.<sup>3</sup> Bi(TFA)<sub>3</sub>[nbp]FeCl<sub>4</sub> and silica gel/FeCl<sub>3</sub> was reused three and four times respectively.<sup>5,19</sup> This method can therefore be rightly considered as an efficient and easy route for the synthesis of 4(3*H*)-quinazolinones.

### 3. Experimental

#### 3.1. Chemicals and Apparatus

All the chemicals were obtained from Merck (Darmstadt, Germany) and used as received. Melting points (uncorrected) were measured using Electro thermal IA 9100 digital melting point apparatus. Yields are based on GC/mass analysis using an Agilent (Denver, CO, USA) 6890 GC system Hp-5 capillary 30 m × 530 μm × 1.5 μm nominal.

#### 3.2. Catalyst Synthesis Procedure

To a solution of the surfactant, sodium bis(2-ethylhexyl) sulphosuccinate, in cyclohexane (0.2 mol L<sup>-1</sup>), a solution of Preyssler acid in a specified amount of water was added. The molar ratio of water to surfactant was selected to be 3, 5 and 7. Tetraethoxysilane was then added to the micro-emulsion phase. After mixing for various times (8, 12, 18, 25 and 30 h) at room temperature, dispersed Preyssler acid/SiO<sub>2</sub> nanostructures were centrifuged (1500 rpm) and the particles were rinsed with acetone (4 times) and dried in a vacuum oven. The optimum ratio of water to surfactant was 3:1 and the optimum time was 30 h.

#### 3.3. General Procedure

To a mixture of anthranilic acid (10 mmol), orthoester (10 mmol) and substituted aniline (10 mmol), a catalytic amount of silica-supported Preyssler nanoparticles (0.03 mmol) was added and the mixture was refluxed in acetonitrile (5 mL) for 15 min. The progress of the reaction was monitored by TLC using EtOAc:hexane (1:4) as eluent (R<sub>f</sub>: 70%). The obtained solid

was crystallized from ethanol after washing with water to eliminate any catalyst residue.

All products were identified by comparison of their physical and spectroscopic data with those reported for authentic samples.<sup>17</sup>

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