Preparation of a novel nanocatalyst based on rice husk-derived nano-silica and its application for the synthesis of pyrano[2,3-d]pyrimidine-2,4-diones and pyrano[2,3-d]pyrimidine-4-one-2-thiones

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Abstract

A novel nanomaterial was prepared via grafting an organic moiety containing 4,4′-bipyridine scaffold on rice husk-derived nano-silica (RH-SiO$_2$). The nanomaterial namely 1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$ (**SPBHRS**) was characterized by FT-IR, XRD, EDX, FE-SEM and TGA analyses. Afterward, the synthesis of pyrano[2,3-d]pyrimidine-2,4-diones and pyrano[2,3-d]pyrimidine-4-one-2-thiones was performed through the one-pot multicomponent reaction of aromatic aldehyde, malononitrile and barbituric acid (or 2-thiobarbituric acid) using **SPBHRS** as an efficient and recyclable catalyst.

Graphical abstract
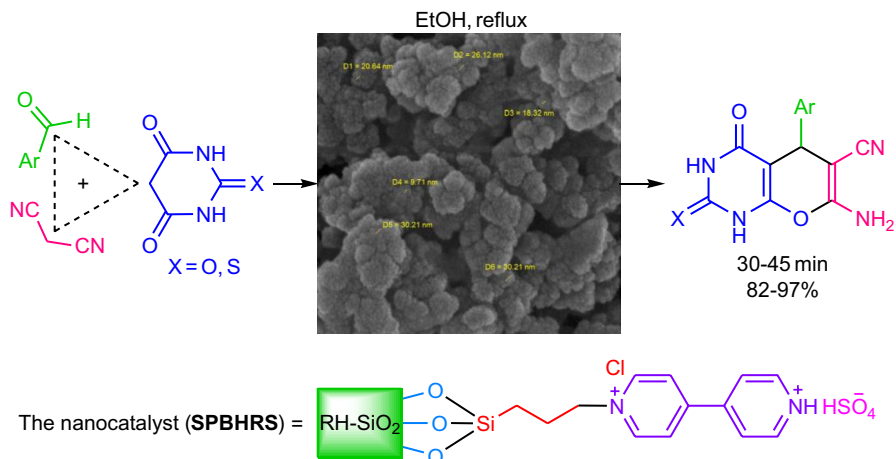
Extended author information available on the last page of the article

Introduction

Considering the green chemistry goals, production of ecofriendly and recyclable catalysts from sustainable resources is of great importance. Among the different heterogeneous catalytic supports, silica is usually preferable, due to high surface area, environmentally friendly nature, durability, low cost, easy recycling and simple functionalization by organic components. Silica is the major inorganic constituent of rice husk; extraction of silica from rice husk is a smart and inexpensive alternative to commercial silica, and meantime aids solving rice husk disposal problem faced by rice milling industry. In most rice producing countries, rice husk is usually burnt away causing environmental and health problems [1–6].

Nanotechnology allows scientists, physicians, chemists and engineers to work at the molecular levels, and create significant advances in the life sciences to improve human life. The widespread applications of nanomaterials cause the necessity to design new ones with diverse structures [7–11].

Nanomaterials based on silica (as support) not only have been used in catalysis processes [1, 7, 12–16], but also have extensive applications in different industries; e.g., they have been applied as multimodal imaging agent [17], multifunctional additive for enhanced lubrication [18], drug delivery tools for skin cancer (melanoma) treatment [19] and adsorbent [20], and for upcycling waste plastics to fabricate lightweight, waterproof and carbonation resistant cementitious materials [21], degradation of antibiotic in aqueous solution [22] and targeted cancer therapy [23].

Because of the advantages of multi-component reactions, such as high atom economy, flexibility, saving energy and time, selectivity and agreement with green chemistry protocols, they have been utilized as a practical and useful technique for production of complex molecules from simple reactants through a one-pot process [24–29].

The compounds containing pyrano-pyrimidine moiety are an attractive group of heterocycles, which have different biological and pharmaceutical activities, e.g., antioxidant [30], antidiabetic [30], antiviral [30], antihypertensive [30], antibacterial [31], antifungal [31], anticancer [32], anti-inflammatory [33], antimicrobial [34] and inhibitor of SARS-CoV-2 [35]. Pyrano[2,3-*d*]pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones could be synthesized through the one-pot multi-component reaction of aromatic aldehyde, malononitrile and barbituric acid (or 2-thiobarbituric acid) using a catalyst [34–43].

Considering the above issues, a novel nanomaterial based on rice husk-derived nano-silica (RH-SiO$_2$) namely 1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$ (**SPBHRS**) was prepared, and characterized by FT-IR, X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), field emission scanning electron microscopy (FE-SEM), thermal gravimetric (TG) and derivative thermal gravimetric (DTG) analyses. It was utilized as an efficient and recoverable catalyst for the synthesis
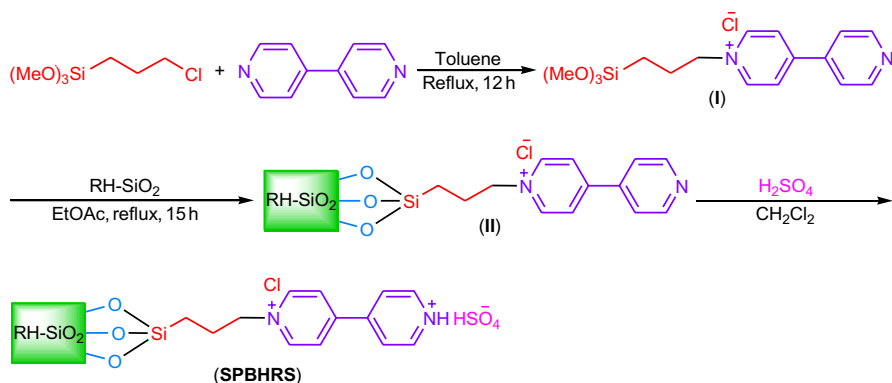
Scheme 1 The preparation of SPBHRS

of pyrano[2,3-*d*]pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones from aromatic aldehyde, malononitrile and barbituric acid (or 2-thiobarbituric acid).

Experimental

Materials and instruments

Information of materials and instruments have been given in supplementary material.

Preparation of SPBHRS (Scheme 1)

A mixture of (3-chloropropyl)trimethoxysilane (0.497 g, 2.5 mmol) and 4,4′-bipyridine (0.390 g, 2.5 mmol) in dry toluene (8 mL) was stirred under reflux conditions for 12 h; then, the solvent was distilled under vacuum at 95 °C to provide **I**. RH-SiO$_2$ (prepared via the reported procedure [44–46]) (0.300 g, 5 mmol) and EtOAc (10 mL) were added to compound **I**, refluxed accompanied with stirring for 15 h, centrifuged, and decanted; the residue was washed by EtOAc (2×5 mL), and dried under vacuum at 70 °C to furnish compound **II**. Finally, **II** was added slowly to a stirring solution of sulfuric acid (0.245 g, 2.5 mmol) in dry CH$_2$Cl$_2$ (8 mL) at room temperature; the resulting mixture was stirred for 5 h at room temperature and 1 h under reflux conditions; then, it was centrifuged, decanted, washed by CH$_2$Cl$_2$ (3×5 mL), and dried under vacuum at 90 °C to afford **SPBHRS**.

General procedure for the synthesis of pyrano[2,3-d]pyrimidine-2,4-diones and pyrano[2,3-d]pyrimidine-4-one-2-thiones

A mixture of aldehyde (0.5 mmol), malononitrile (0.036 g, 0.55 mmol), barbituric acid or thiobarbituric acid (0.5 mmol) and **SPBHRS** (0.015 g) in EtOH (5 mL) was

stirred under reflux conditions. After completion of the reaction (as observed by TLC), the solvent was distilled, EtOAc (20 mL) was added, stirred for 2 min under reflux conditions, centrifuged, and decanted to isolate the unsolvable **SPBHRS** (this work was performed two times). The catalyst was washed by EtOAc (2×3 mL), dried and used for next run. The resulted EtOAc from the two times decanting was distilled, and the remainder precipitate was recrystallized from EtOH (96%) to afford the pure product.

Note: Selected spectral data and original spectrums of the products have been reported in supplementary material.

Results and discussion

Characterization of SPBHRS

1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$ (**SPBHRS**) was synthesized via the nucleophilic substitution reaction of 4,4′-bipyridine and (3-chloropropyl)trimethoxysilane, then, grafting the obtained compound on rice husk-derived nano-silica, and finally, addition of H$_2$SO$_4$ (Scheme 1). It was characterized by FT-IR, XRD, EDX, FE-SEM, TG and DTG analyses.

The FT-IR spectrum of **SPBHRS** (Fig. S1) confirmed presence of the expected bonds and functional groups in its organic and inorganic components. The peaks appeared at 469, 802 and 1094 cm^{-1} are related to bending modes, symmetric stretching and asymmetric stretching of Si–O bonds in SiO$_2$. The adsorbed H$_2$O molecules on the silica surface gave a peak at 1639 cm^{-1}. The peaks observed at 1416 and 1570 cm^{-1} are assigned to C=C bonds of the 4,4′-bipyridine moiety. The broad peak at ∼2781–3733 cm^{-1} is belong to OH groups of hydrogen sulfate and the adsorbed water on the silica surface. In the FT-IR spectrum of compound **I** (Fig. S2), the peaks pertained to Si–O bonds of Si–OCH$_3$ moiety (464, 798 and 1100 cm^{-1}), C–O of SiO–CH$_3$ component (1171 cm^{-1}), C=C bond of the 4,4′-bipyridine constituent (1580 cm^{-1}), aliphatic C–H (2927 cm^{-1}) and Si–OH produced by hydrolysis of Si–OCH$_3$ (∼3315–3691 cm^{-1}) were seen. In the spectrum of compound **II** (Fig. S3), the bands related to Si–O bonds in SiO$_2$ (470, 803 and 1092 cm^{-1}), C=C bond of the 4,4′-bipyridine component (1417 and 1574 cm^{-1}), aliphatic C–H (2930 cm^{-1}) and OH groups of the adsorbed water on the silica surface (∼3285–3720 cm^{-1}) were observed. The peak belong to C–O of SiO–CH$_3$ was not seen in the spectrum of compound **II**, because of bonding **I** to silica surface and leaving OCH$_3$ (compare the spectrums of **I** and **II**). The band related to OH groups of hydrogen sulfate can be seen in the spectrum of **SPBHRS**, but the spectrum of **II** did not show this band; this subject confirmed successful performing the reaction of sulfuric acid with compound **II** to produce **SPBHRS**.

The XRD patterns of **SPBHRS** (the catalyst) and the reactants used for its synthesis (i.e., RH-SiO$_2$, compound **I** and compound **II**) at a range of 2θ=2–70º are represented in Fig. 1 (the catalyst synthesis is shown in Scheme 1). In the XRD pattern of RH-SiO$_2$, there is a broad peak at 2θ ≈ 15–38º, which is related to amorphous form

of SiO_2. There are some diffraction lines in the XRD pattern of compound **I**, which are assigned to its crystalline forms. In the pattern of compound **II** (which have been synthesized by reacting RH-SiO$_2$ and compound **I**), observation of the broad peak of amorphous silica and some diffraction lines verified successful grafting compound **I** on RH-SiO$_2$; this subject was also observed in the catalyst XRD (reducing the diffraction lines in the XRD pattern of **SPBHRS** in comparison with compound **II** can be attributed to addition of H_2SO_4 to compound **II** to afford **SPBHRS**). The above explanation approved successful production of **SPBHRS**.

The EDX spectrum (Fig. 2) showed the peak related to silicon, which is belong to RH-SiO$_2$ and Si-pr constituents of 1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$. In the spectrum, the peak pertained to oxygen was observed, which is ascribed to RH-SiO$_2$ and the HSO_4^- moieties of **SPBHRS**. The EDX analysis confirmed existence of carbon in the nanomaterial that is attributed to Si-pr and [4,4′-bipyridine]-1,1′-diium components. The presence of the nitrogen related to [4,4′-bipyridine]-1,1′-diium was also approved by the EDX analysis. Existing HSO_4^- in the **SPBHRS** structure was verified by observing the peak related to sulfur in the EDX spectrum. Thus, the analysis confirmed presence of all expected elements in the structure of **SPBHRS**. Moreover, some peaks with low intensity were observed in the spectrum, these can be attributed to the impurities in the rice husk-derived nano-silica, which mentioned in the literature [45, 47].

A sample of **SPBHRS** was analyzed by FE-SEM to determine the size distribution, particles shapes and surface morphology. The obtained micrographs (Fig. 3) indicated that **SPBHRS** has porous and irregular shapes, and the particles sizes are
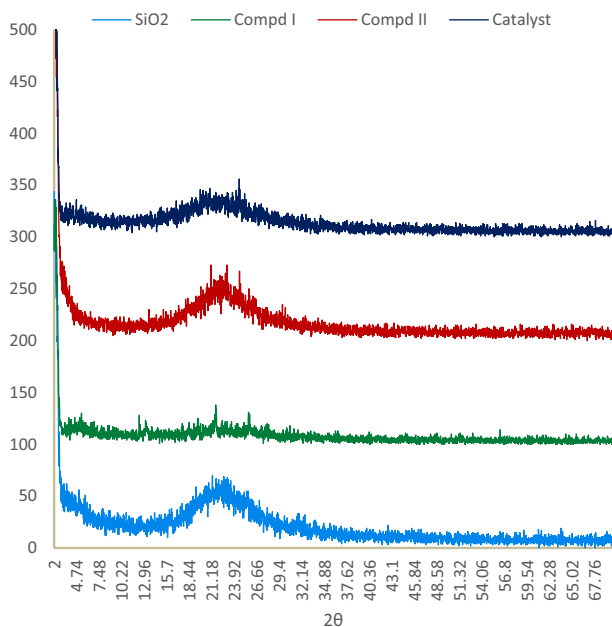

Fig. 1 The XRD patterns of RH-SiO$_2$, compound **I**, compound **II** and the catalyst
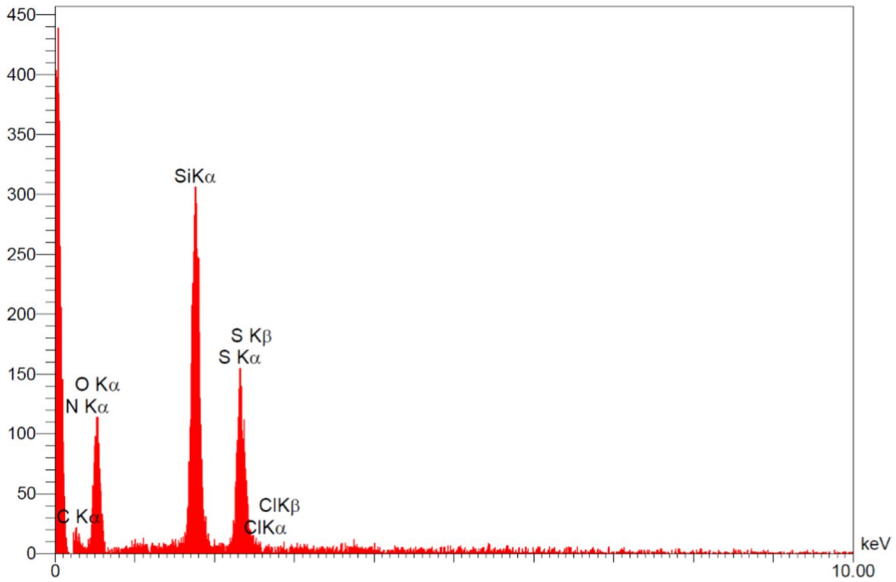
Fig. 2 The EDX spectrum of **SPBHRS**

less than 100 nm (e.g., 9.71, 14.38, 18.32, 18.52, 20.24, 20.64, 21.06, 24.10, 24.48, 26.12 and 30.21 nm).

Thermal gravimetric analysis of **SPBHRS** was carried out at a range of 25–600 °C; the corresponding diagrams are illustrated in Fig. 4. According to the diagrams, **SPBHRS** showed good thermal stability, and decomposed gradually at a range of ~210–600 °C with low-speed weight loss. The weight loss below ~110 °C
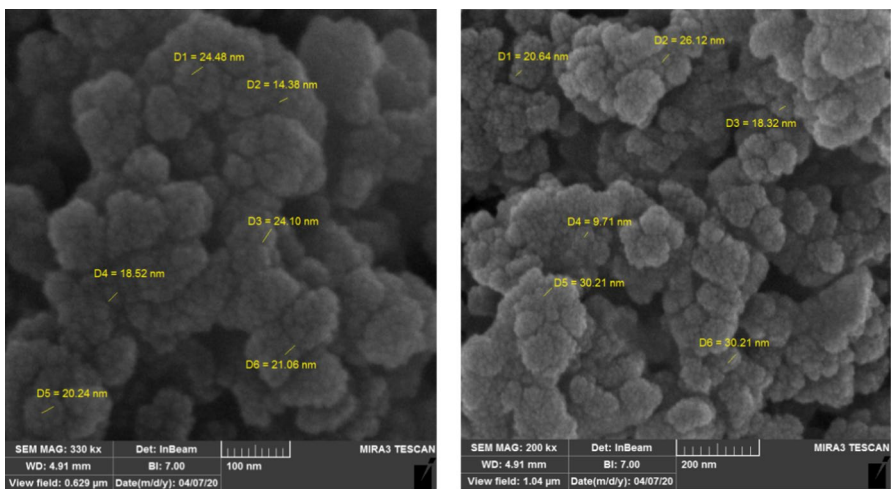

Fig. 3 The FE-SEM images of **SPBHRS**

can be attributed to the loss of physically adsorbed water and other solvents on the nanomaterial surface. The weight losses at ~ 210–600 °C can be related to the decomposition of 1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$ surface and the condensation of the silanol groups. These data confirmed successful grafting the organic component on RH-SiO$_2$ surface, and stability of the nanomaterial up to 210 °C.

Catalytic activity of SPBHRS

Catalytic activity of **SPBHRS** was tested for the preparation of pyrano[2,3-*d*]pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones. For this purpose, effect of the catalyst loading, solvent and temperature was investigated on the condensation of 4-chlorobenzaldehyde (0.5 mmol), malononitrile (0.55 mmol) and barbituric acid (0.5 mmol), as a model reaction (Scheme 2); the results are summarized in Table 1. The optimal catalyst quantity was found to be 0.015 g (Table 1, entry 3). According to the data acquired from examining the reaction under solvent-free conditions, and in EtOH, EtOH/H$_2$O (1/1), H$_2$O, MeCN and CH$_2$Cl$_2$ (5 mL), the best solvent was EtOH (Table 1, entry 3). Furthermore, the best reaction time and yield were obtained when the reaction was carried out under reflux conditions (Table 1, entry 3).

In continue, to assess the efficiency and generality of **SPBHRS** for the synthesis of miscellaneous derivatives of pyrano[2,3-*d*]pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones, the reaction of diverse arylaldehydes
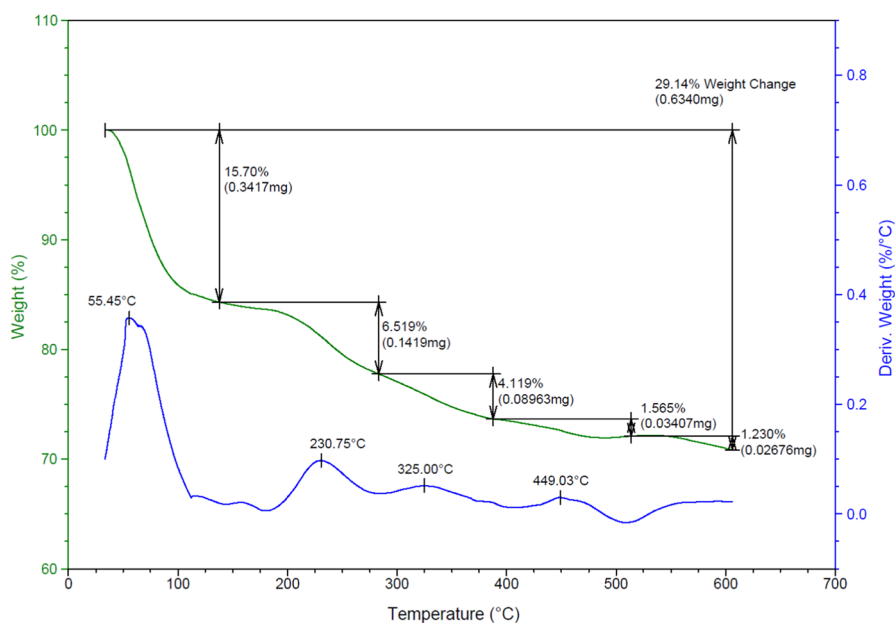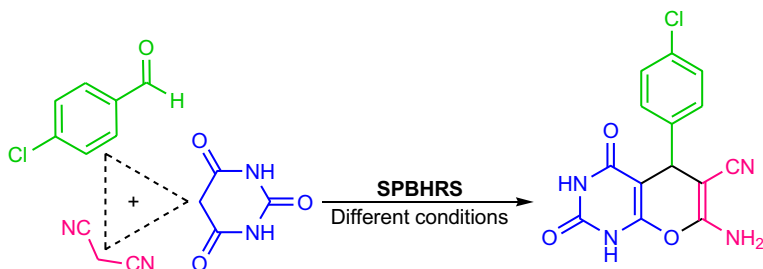

Fig. 4 The TG and DTG diagrams of **SPBHRS**

Scheme 2 The model reaction

**Table 1** Investigating influence of the catalyst quantity, solvent and temperature on the model reaction

Entry	Catalyst quantity (g)	Solvent	Temp. (°C)	Time (min)	Yield (%)
1	0.015	–	110	90	65
2	0.013	EtOH	Reflux	40	89
3	0.015	EtOH	Reflux	30	97
4	0.017	EtOH	Reflux	28	97
5	0.015	EtOH/H_2O (1/1)	Reflux	50	83
6	0.015	H_2O	Reflux	60	54
7	0.015	MeCN	Reflux	60	68
8	0.015	CH_2Cl_2	Reflux	70	29

with malononitrile and barbituric acid (or 2-thiobarbituric acid) was tested in the optimal conditions; the results are given in Table 2. As it can be seen in Table 2, arylaldehydes bearing halogen, electron-releasing and electron-withdrawing substituents on ortho-, meta- and para-positions afforded the relevant products in high yields and relatively short times. The results were satisfactory for both barbituric acid and 2-thiobarbituric acid. According to the results, it can be said **SPBHRS** was an efficacious and general catalyst for the preparation of the mentioned compounds.

To complete our study on the synthesis and catalytic applicability of 1-(Si-pr)-[4,4′-bipyridine]-1,1′-diium hydrogen sulfate grafted on RH-SiO$_2$ for the preparation of the pyrano-pyrimidine derivatives, its recyclability and reusability were investigated for the preparation of compound **1i** (Fig. 5). **SPBHRS** was reusable for four times with negligible decrement in its catalytic performance.

As it is shown in the reaction mechanism (Scheme 3), hydrogen sulfate of **SPBHRS** catalyzes the reaction through activation of electrophiles and nucleophiles (steps 1, 3 and 4), assisting removal of H_2O (step 2), and helping tautomerization (steps 1, 3 and 5).
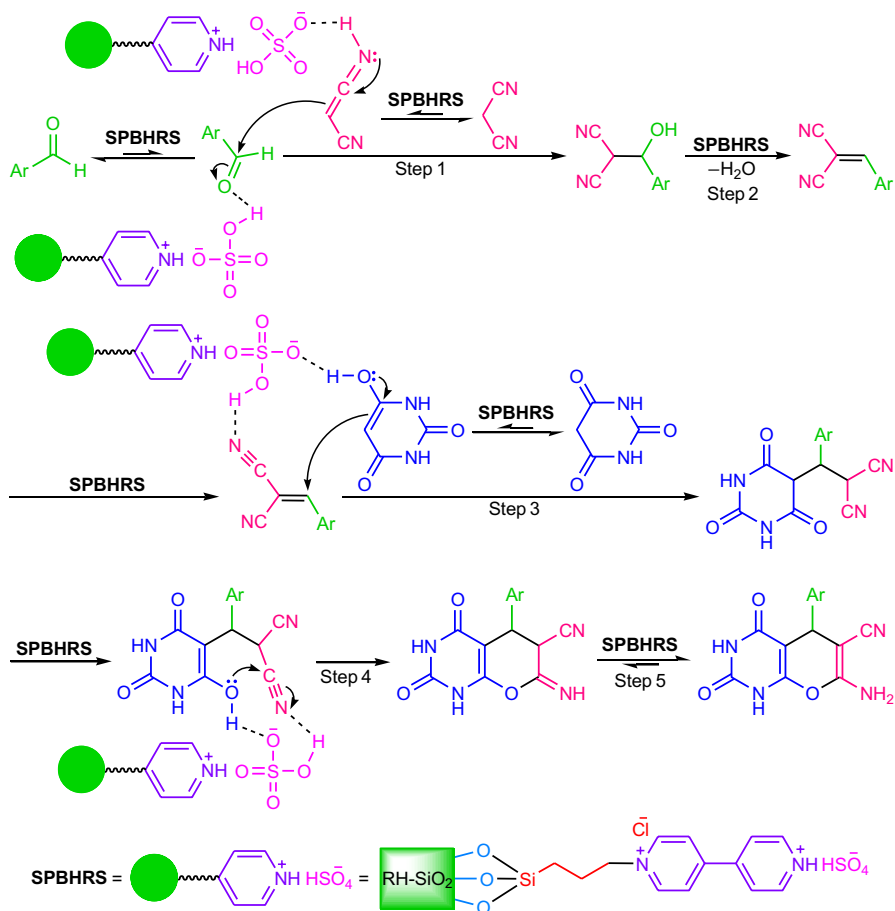
Comparison of the reaction conditions and the results of **SPBHRS** with those of some reported catalysts in the construction of compounds **1a** and **1i** is illustrated in Table 3. This comparison confirmed superiority of our catalyst with respect to these reported catalysts in at least two of these factors: the reaction temperature, time and yield. Furthermore, in our work, the construction of pyrano[2,3-*d*]

Table 2 The construction of pyrano[2,3-*d*]pyrimidine-2,4-dione and pyrano[2,3-*d*]pyrimidine-4-one-2-thione derivatives using **SPBHRS**
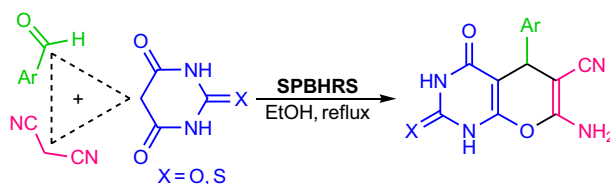
Product No.	Ar	X	Time (min)	Yield [a] (%)	M.p. (°C) [lit.]
1a	C_6H_5	O	30	93	219–221 (219–221) [36]
1b	2-ClC$_6$H$_4$	O	35	87	210–212 (212–215) [35]
1c	3,5-Cl$_2$C$_6$H$_3$	O	45	89	228–230
1d	3,5-Cl$_2$C$_6$H$_3$	S	45	87	257–259
1e	2,6-Cl$_2$C$_6$H$_3$	O	40	91	224–226 (227–228) [38]
1f	2,6-Cl$_2$C$_6$H$_3$	S	45	87	252–254
1 g	2,4-Cl$_2$C$_6$H$_3$	O	40	94	242–244 (241–242) [37]
1 h	2,4-Cl$_2$C$_6$H$_3$	S	45	92	237–239 (238–240) [37]
1i	4-ClC$_6$H$_4$	O	30	97	238–240 (240–242) [36]
1j	2-BrC$_6$H$_4$	S	40	93	217–219
1 k	4-BrC$_6$H$_4$	O	30	84	231–233 (228–232) [36]
1 l	3-CH$_3$OC$_6$H$_4$	O	35	82	202–204 (200–202) [43]
1 m	2-O$_2$NC$_6$H$_4$	O	35	88	251–253 (254–256) [42]
1n	2-Naphthyl	S	40	90	235–237

[a]Isolated yield
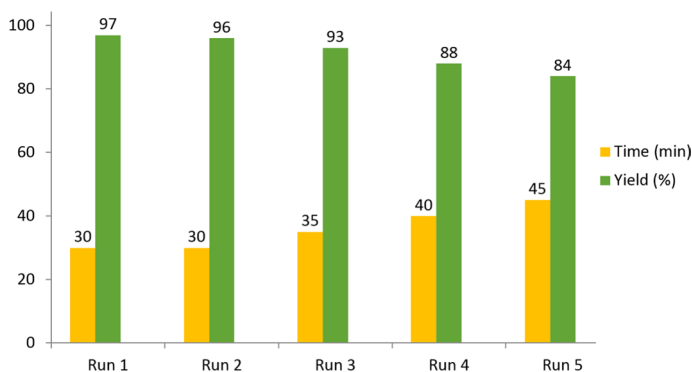
Fig. 5 Reusability of **SPBHRS**

pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones has been performed; however, in many of these works, pyrano[2,3-*d*]pyrimidine-4-one-2-thiones have not been synthesized. The production of nano-silica (as a precursor for the

Scheme 3 The reaction mechanism

catalyst synthesis) from a sustainable and inexpensive resource (rice husk) is another advantage of our research.

Conclusions

A novel nanomaterial has been prepared based on nano-silica derived from a sustainable and natural resource (i.e., rice husk). It could catalyze the synthesis of pyrano[2,3-*d*]pyrimidine-2,4-diones and pyrano[2,3-*d*]pyrimidine-4-one-2-thiones; efficacy, generality, high yields, relatively short reaction times and reusability of the catalyst are some benefits of this catalytic process.

Table 3 Comparing the reaction conditions and the results of **SPBHRS** with those of the reported catalysts in the construction of compounds **1a** and **1i**

Catalyst	Conditions	Time (min) of products 1a/1i	Yield (%) of products 1a/1i	Refs.
SPBHRS	EtOH, reflux	30/30	93/97	–
β-Cyclodextrin[a]	H_2O, 80 °C	37/48	90/75	[34]
WELPSA[a,b]	H_2O, r.t	540/540	86/85	[35]
WELPSA[a]	H_2O, MW, 100 W, 100 °C	2/3	95/93	[35]
Taurine/choline chloride	H_2O, 90 °C	23/19	91/93	[36]
$(NH_4)_2HPO_4$	H_2O/ EtOH, r.t	120	71-90[c]	[37]
SBA-Pr-SO_3H[a]	Solvent-free, 140 °C	-[d]/45	-[d]/30	[38]
Er-folic acid-coated $CoFe_2O_4$[a]	Solvent-free, 100 °C	20/25	96/93	[39]
Er-folic acid-coated $CoFe_2O_4$[a]	H_2O, 80 °C, US irradiation	10/12	92/89	[39]
Bis-imidazolium hydrogen sulfate[a]	H_2O, reflux	25/12	70/94	[40]
Bis-imidazolium perchlorate[a]	H_2O, reflux	30/15	65/92	[40]
Agar-entrapped [DABCO] $(SO_3H)_2(Cl)_2$	H_2O, reflux	-[d]/9	-[d]/98.7	[41]
Choline chloride/ urea/thiourea	H_2O, reflux	21/17	91/93	[42]
CuO/ZnO[a]	H_2O, reflux	20/12	92/91	[43]

[a]In this work, the synthesis of pyrano[2,3-*d*]pyrimidine-4-one-2-thiones has not been performed

[b]A natural catalyst of alkali and alkaline earth metals

[c]The yields range of all synthesized compounds have been given, because, in this work, compounds **1a** and **1i** have not been constructed

[d]In this research, the construction of this compound has not been reported

Declarations

Conflict of interest This declaration is not applicable.

Ethical approval This declaration is not applicable.

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