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# Efficient synthesis of 2*H*-indazolo[2,1-b]phthalazine-trione derivatives using succinimidinium *N*-sulfonic acid hydrogen sulfate as a new ionic liquid catalyst

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ABSTRACT

Succinimidinium *N*-sulfonic acid hydrogen sulfate is prepared as a new ionic liquid and characterized with a variety of techniques including FT-IR, $^{1}H$ and $^{13}C$ NMR, SEM, mass spectra method as well as Hammett acidity function. After identification, this reagent was used as an efficient catalyst for the multi-component synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives. The simple work-up, mild reaction conditions, excellent yields and relatively short reaction times are the notable advantages of this protocol. In addition, the ionic liquid could be recycled several times without appreciable reduction in its catalytic activity.



## 1. Introduction

Ionic liquids (ILs) have received considerable interest as eco-friendly solvents, catalysts and reagents in green synthesis. This interest can be attributed to their unique properties such as negligible vapor pressure, nonflammability, nomiscibility with nonpolar solvents, reasonable thermal and chemical stability and reusability for many times without considerable decrease in their activity [1]. Among these types of compounds, acidic ionic liquids are the most important ones which have been successfully used in different types of organic transformations [2].

In the past decade, other types of acidic ionic liquids based on *N*-substituted reagents are prepared and successfully used for the acceleration of some of the organic reactions including functional group transformation reactions and synthesis of the various types of organic compounds via the multi-component reactions [3].

Synthesis of heterocycles containing phthalazine is of interest because of their very important pharmacological and biological activities such as anti-inflammatory [4], anti-convulsant [5], anti-microbial [6], anti-fungal [7] and anti-cancer [8] activities. In addition, these compounds can be used as new luminescent materials or fluorescence probes [9].

Among these compounds, the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives is so attracted the attention of many organic chemists that in the past decade, several reports on the synthesis of them using different types of catalysts have been reported in the literature [10–20]. However, some of these methods suffer from limitations such as the use of expensive catalysts, harsh reaction conditions, the use of toxic solvents, low yields and long reaction times. Therefore, it is important to find more efficient catalysts and methods for the synthesis of these types of compounds.

In recent years, the use of green catalysts or conditions in synthetic reactions attracted the attention of many organic chemists. This attention can be attributed to the reduction of environmental pollution and the cost of the applied methods. Therefore, preparation of phthalazine derivatives in the presence of green catalysts such as ionic liquids or heterogeneous compounds in the absence of solvent is a useful direction in green chemistry [21–25].

In 2011, and on the basis of our previous reports using ionic liquids in organic transformations [26], we have introduced succinimide-*N*-sulfonic acid [SuSA] for the promotion of different types of organic reactions [27].

Herein and in continuation of these studies we wish to report the preparation, characterization and application of succinimidinium *N*-sulfonic acid hydrogen sulfate {[SuSA-H]$HSO_4$} in the promotion of the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives.

## 2. Experimental

### 2.1. General

Chemicals were purchased from Fluka, Merck, and Aldrich chemical companies. All yields refer to the isolated products. Products were

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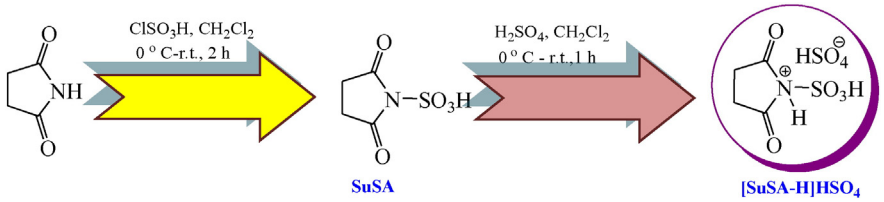



**Scheme 1.** Preparation of [SuSA-H]$HSO_4$.

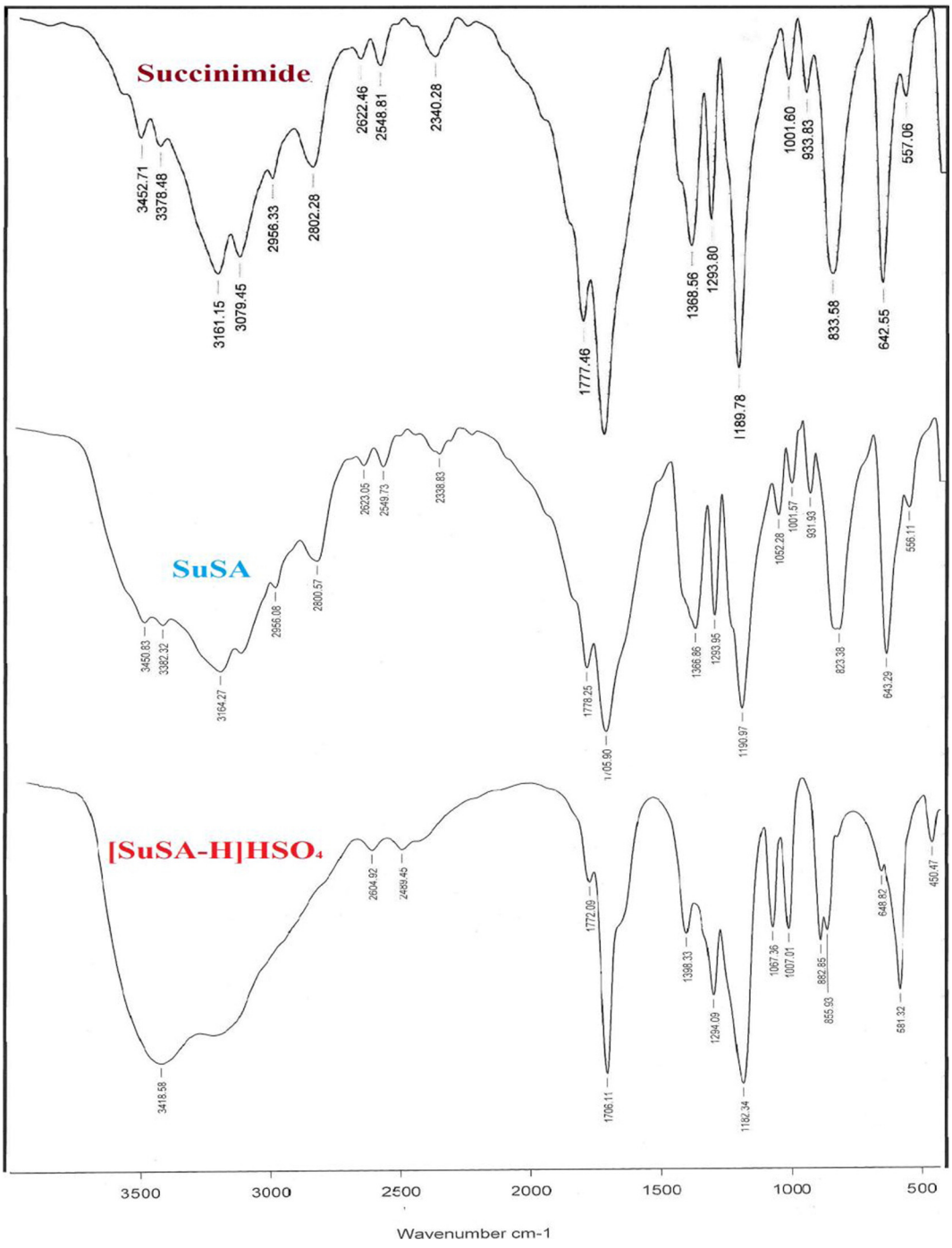



**Fig. 1.** FT-IR spectra of succinimide, SuSA and [SuSA-H]$HSO_4$.

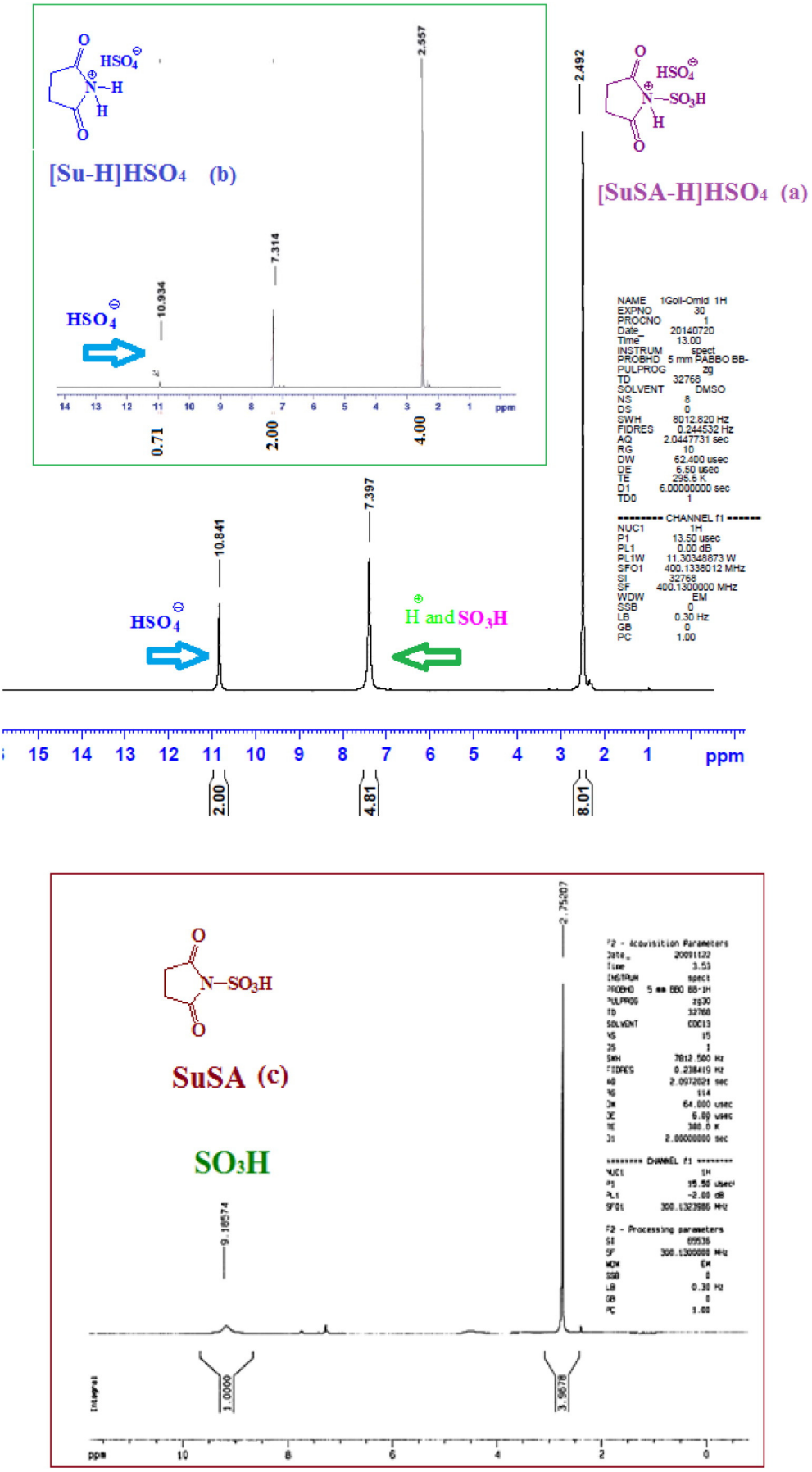



**Fig. 2.** $^{1}H$ NMR spectra of $[SuSA\text{-}H]HSO_4$ (a), $[Suc\text{-}H]HSO_4$ (b) and SuSA (c).

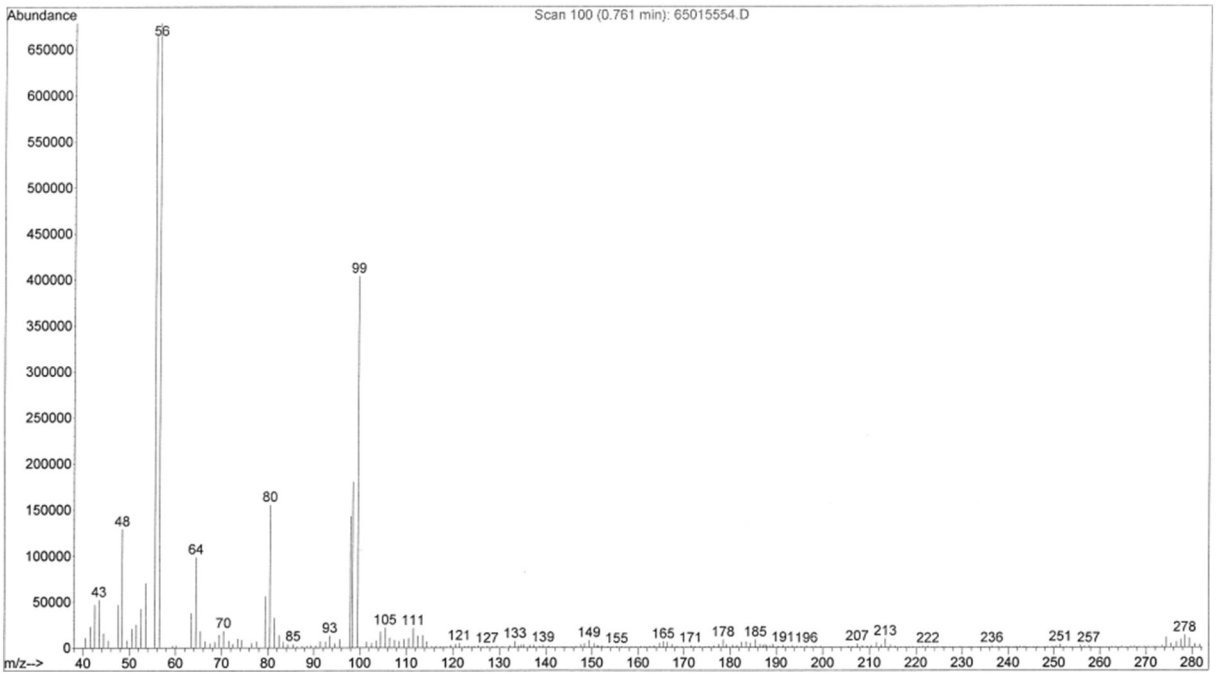



**Fig. 3.** Mass spectra of [SuSA-H]$HSO_4$.

characterized by comparison of their physical constants and also their IR and NMR spectra with authentic samples and those reported in the literature. The purity determination of the substrate and reaction monitoring were accompanied by TLC on silicagel polygram SILG/UV 254 plates.

### *2.2. Preparation of succinimidinium N-sulfonic acid hydrogen sulfate ([SuSA-H]$HSO_4$)*

Succinimide-*N*-sulfonic acid [SuSA] is prepared according to the reported method in the literature [27]. Then, sulfuric acid 98% (0.5 mL) was added drop wise to a mixture of SuSA (1.68 g) in dry $CH_2Cl_2$ (10 mL) over a period of 2 min in an ice bath. The resulting mixture was stirred for 1 h and then the solvent was decanted. The solid compound was washed with dry diethylether (2 × 5 mL) and dried under vacuum to give [SuSA-H]$HSO_4$ as a white gel in 98% (Scheme 1).

Spectroscopic data for [SuSA-H]$HSO_4$ are as follows:

FT-IR (KBr, $cm^{-1}$) $\upsilon_{max}$: 3418, 1706, 1294, 1182, 855, 581; $^1$H NMR (400 MHz, DMSO-$d_6$): δ = 2.49 (4H, s), 7.39 (2H, s), 10.84 (1H, s, OH) ppm; $^{13}$C NMR (100 MHz, DMSO-$d_6$) δ = 29.78, 180 ppm; MS: 56, 99, 278 m/z;

### *2.3. Catalyst characterization*

#### *2.3.1. Instrumentation*

The FT-IR spectra were recorded on a Perkin Elmer 781 Spectrophotometer. The $^1$H NMR spectra were recorded with Bruker Avance 400 instruments. In all the cases the chemical shifts are quoted in parts per

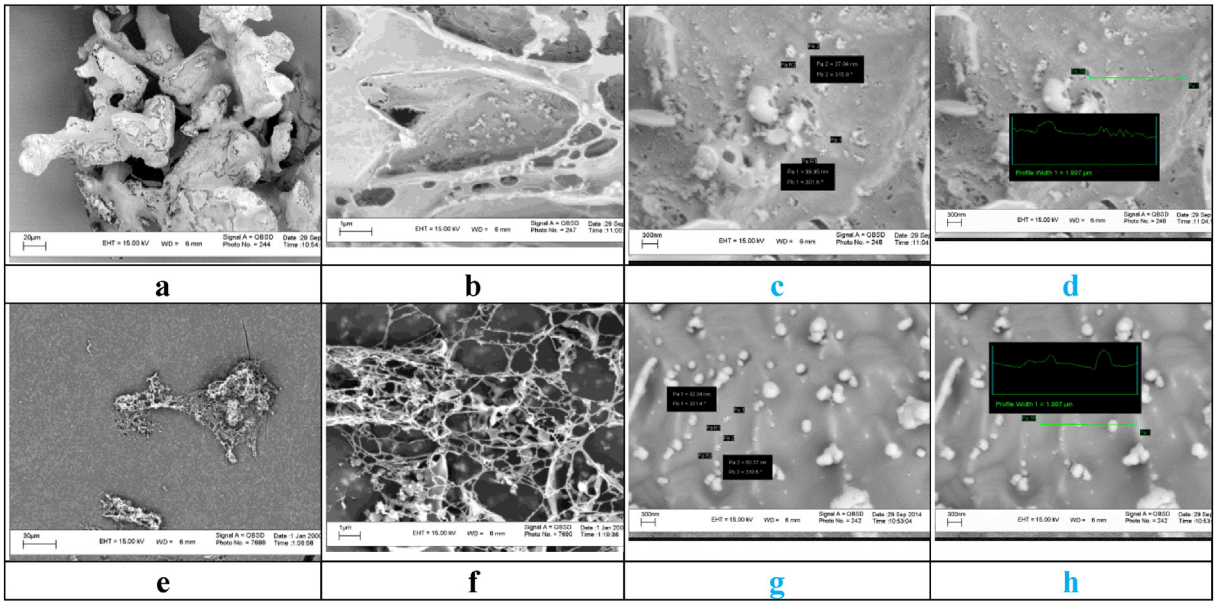



**Fig. 4.** SEM micrographs of [SuSA-H]$HSO_4$ (a–c) and succinimide (d–f).

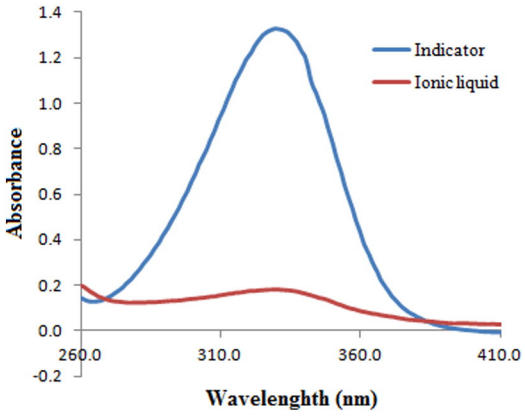



**Fig. 5.** Absorption spectra of 4-nitroaniline (indicator) and [SuSA-H]$HSO_4$ (catalyst) in $CCl_4$.

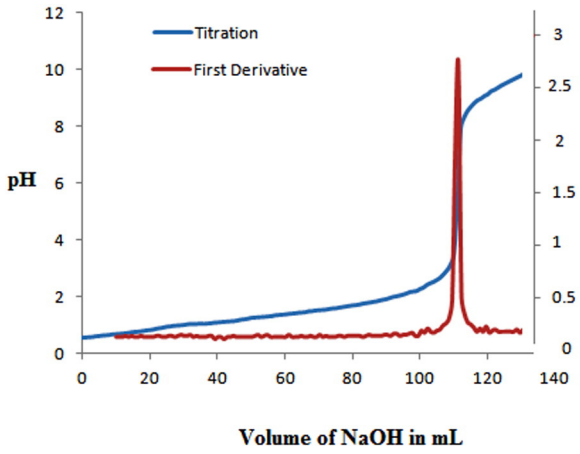



**Fig. 6.** Titration and its first derivative curves of the ionic liquid with NaOH.

million (ppm) relative to TMS using deuterated solvent. The $^{13}C$ NMR data were collected on Bruker Avance 100 MHz instrument. MS studies were performed using 5973 network mass selective detector, Agilent Technology (HP) company (ion source: electronic (EI) 70 eV; ion source temperature: 230 °C; analyzer: quadrupole). Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

*2.3.2. IR analysis*

The infrared spectra of succinimide, SuSA and [SuSA-H]$HSO_4$ are shown in Fig. 1. The IR spectrum of the ionic liquid shows a broad peak at 3000–3600 $cm^{-1}$ which can be related to the OH stretching of the $SO_3H$ groups. Moreover, the strong peaks observed at 581, 882 and 1182 $cm^{-1}$ correspond to the S–O symmetric and asymmetric stretchings, respectively. It should be noted that the two peaks of the carbonyl groups in succinimide which are observed at 1698 and 1777 $cm^{-1}$, converted to one peak (1706 $cm^{-1}$) in the catalyst [27].

*2.3.3. $^1H$ NMR analysis*

The $^1H$ NMR spectrum of succinimidinium hydrogen sulfate [Su-H]$HSO_4$, SuSA and [SuSA-H]$HSO_4$ is compared in Fig. 2. In the $^1H$ NMR spectrum of [SuSA-H]$HSO_4$ (Fig. 2A), in addition to the other protons, the acidic hydrogen of $HSO_4$ appeared at 10.84 ppm [28]. This observation clarifies that [SuSA-H]$HSO_4$ is exactly synthesized (Fig. 2A).

*2.3.4. Mass analysis*

The mass spectrum of [SuSA-H]$HSO_4$ is shown in Fig. 3. In this spectrum the correct molecular ion peak appears at 278. Another ion peak is also observed at 99 ($M^+$-$SO_3H$ and $HSO_4$) [28].

*2.3.5. SEM analysis*

The samples of succinimide and [SuSA-H]$HSO_4$ were also analyzed by scanning electron microscopy (SEM) with various magnifications for determining the particle shape, size distribution and surface morphology (Fig. 4). These images show that with chemical modification the primary morphology of succinimide is completely changed and the particles are aggregated in the product. This increased the surface area of the catalyst and finally its catalytic activity. This aggregation can be caused by hydrogen bonding sites and nearby the positive and negative sides [28].

*2.3.6. Acidity of the catalyst*

The Hammett acidity method is an effective way to identify the acidity strength of an acid in organic solvents, using UV–vis technique [29]. The Hammett function is defined as:

$$H_0 = \mathrm{pK(I)_{aq}} + \log\left([\mathrm{I}]_s / [\mathrm{IH}^+]_s\right)$$

where the $pK(I)_{aq}$ is the $pK_a$ value of aqueous solution of indicator, $[IH^+]_s$ and $[I]_s$ are the molar concentrations of protonated and unprotonated forms of the indicator in the solvent, respectively. According to Lambert–Beer's Law, the value of $[I]_s/[IH^+]_s$ can be determined and calculated through UV–visible spectrum.

For this purpose, 4-nitroaniline ($pK(I)aq = 0.99$) as the basic indicator and $CCl_4$ as the solvent were chosen. As can be seen in Fig. 5, the maximal absorbance of the unprotonated form of the indicator was observed at 330 nm in $CCl_4$. When [SuSA-H]$HSO_4$ as the ionic liquid catalyst was added to the indicator solution, the absorbance of the unprotonated form of the indicator decreased, which indicated that the indicator was partially in the form of $[IH^+]$. These results that have been listed in Table 1, show the acidity strength of [SuSA-H]$HSO_4$.

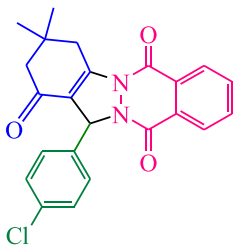
A comparison of the Hammett acidity of SuSA (Table 1, entry 2) with [SuSA-H]$HSO_4$, shows that the prepared ionic liquid is more acidic than

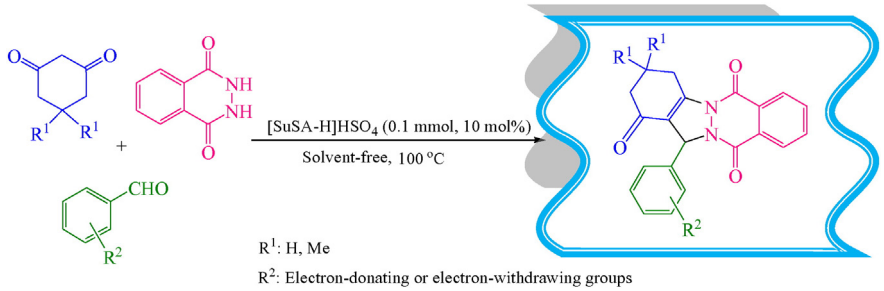
**Table 1**
Calculation of the Hammett acidity function ($H_0$) for [SuSA-H]$HSO_4$.

| Entry | Catalyst | $A_{max}$ | $[I]_s$ % | $[IH^+]_s$ % | $H_0$ Ref. |
|---|---|---|---|---|---|
| 1 | – | 1.329 | 100 | 0 | – |
| 2 | SuSA | 0.412 | 25.4 | 74.6 | 0.52 [27] |
| 3 | [SuSA-H]$HSO_4$ | 0.182 | 13.69 | 86.31 | 0.191 |

Condition for UV–visible spectrum measurement: solvent: $CCl_4$, indicator: 4-nitroaniline ($pK(I)_{aq} = 0.99$), $1.44 \times 10^{-4}$ mol/L (10 mL); Catalyst: SuSA or [SuSA-H]$HSO_4$ (10 mg), 25 °C.

**Table 2**
Optimization of the reaction conditions for the synthesis of phthalazine-triones derivative of 4-chlorobenzaldehyde.

| Entry | Product | Catalyst (mmol) | Time (min) | Isolated yields (%) | Temperature (° C) |
|---|---|---|---|---|---|
| 1 |  | 0.1 | 60 | 50 | r.t. |
| 2 | | 0.2 | 60 | 80 | 60 |
| 3 | | 0.2 | 60 | 85 | 80 |
| 4 | | 0.4 | 15 | 96 | 80 |
| **5** | | **0.1** | **7** | 97 | **100** |
| 6 | | 0.2 | 7 | 97 | 100 |
| 7 | | 0.4 | 4 | 96 | 100 |
| 8 | | 0.1 | 30 | 98 | 120 |




**Scheme 2.** Synthesis of phthalazine-triones derivatives using [SuSA-H] $HSO_4$.

SuSA, which may cause it more efficient catalyst for the requested reactions.

*2.3.7. Titration curve*

The titration method is used to determine the number of protic protons of the prepared catalyst. In this experiment a solution of the ionic liquid was titrated with NaOH. The titration curve for the reaction of 20.5 mL of 0.09 M ionic liquid with 0.05 M NaOH is given in Fig. 6. This figure clearly shows that, when 110.7 mL of the basic solution is added, all the acidic protons are neutralized. On the other hand, Eq. (1) shows that for the neutralization of each of the acidic protons 36.9 mL of the basic solution is needed. On the basis of these studies it can be concluded that this ionic liquid has three protic protons with almost the same acidic power.

$$M(\text{acid}) \times V(\text{acid}) = M(\text{base}) \times V(\text{base})$$

$$0.09(\text{molar}) \times 20.5(\text{mL}) = 0.05(\text{molar}) \times V(\text{base}) \quad (1)$$

$$V(\text{base}) = 36.9 \text{ mL}$$

*2.4. General procedure for the synthesis of 2H-indazolo[1,2-b]phthalazine-triones*

A mixture of aldehyde (1 mmol), 1,3-cyclic diketone (1 mmol), phthalhydrazide (1 mmol) and [SuSA-H]$HSO_4$ (27.7 mg, 0.1 mmol, 10 mol%) was stirred in an oil-bath at 100 °C under solvent-free conditions. After completion of the reaction [monitored by TLC: *n*-hexane:ethyl acetate (8:2)], the reaction mixture was cooled, $H_2O$ (5 mL) was added to it and filtered to separate the catalyst. The solid residue was recrystallized from ethanol to give the pure product.

## 3. Results and discussion

On the basis of the obtained information, we anticipated that [SuSA-H]$HSO_4$ can be used as an efficient catalyst for the acceleration of the reactions which need the use of an acidic catalyst to speed-up. So we were interested to investigate the applicability of this ionic liquid in the promotion of the synthesis of 2*H*-indazolo[1,2-b]phthalazine-triones derivatives.

At first, and in order to find the optimized reaction conditions, the reaction of 4-chlorobenzaldehyde with phthalhydrazide and dimedone was studied as a model reaction in the absence of solvent at different temperatures using different amounts of [SuSA-H] $HSO_4$ as the catalyst. The results are tabulated in Table 2.

Also, the model reaction was examined in various solvents ($H_2O$, EtOH, $CH_3CN$ and $CH_2Cl_2$) and the obtained results showed that even after a long time (>1 h) the reactions were not completed. Therefore the solvent-free condition was selected as the best choice (Scheme 2).

After optimization of the reaction conditions and in order to show the efficiency of this method, different types of aldehydes were subjected to the same reaction under the determined conditions.

As shown in Table 3, a series of aromatic aldehydes containing either electron-donating or electron-withdrawing substituents successfully

**Table 3**
Synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives in the presence of [SuSA-H] $HSO_4$.[a].

| Entry | Aldehyde | $R^1$ | Time (min) | Yield (%) | Melting point (°C) | |
|---|---|---|---|---|---|---|
| | | | | | Found | Reported[Ref] |
| 1 | $C_6H_5CHO$ | $CH_3$ | 15 | 93 | 202–204 | 202–204 [21] |
| 2 | 4-$ClC_6H_4CHO$ | $CH_3$ | 7 | 97 | 265–269 | 263–265 [21] |
| 3 | 3-$ClC_6H_4CHO$ | $CH_3$ | 25 | 92 | 204–208 | 207–209 [30] |
| 4 | 2-$ClC_6H_4CHO$ | $CH_3$ | 10 | 94 | 260–262 | 266–268 [30] |
| 5 | 4-$NO_2C_6H_4CHO$ | $CH_3$ | 10 | 96 | 218–220 | 224–226 [21] |
| 6 | 3-$NO_2C_6H_4CHO$ | $CH_3$ | 20 | 94 | 267–269 | 269–271 [21] |
| 7 | 2-$NO_2C_6H_4CHO$ | $CH_3$ | 30 | 92 | 233–236 | 236–238 [22] |
| 8 | 4-$BrC_6H_4CHO$ | $CH_3$ | 20 | 89 | 260–265 | 266–268 [30] |
| 9 | 4-$CH_3OC_6H_4CHO$ | $CH_3$ | 30 | 96 | 218–220 | 217–219 [21] |
| 10 | 3-$CH_3OC_6H_4CHO$ | $CH_3$ | 15 | 95 | 206–208 | 206–208 [22] |
| 11 | 2-$CH_3OC_6H_4CHO$ | $CH_3$ | 70 | 91 | 238–241 | 242–243 [19] |
| 12 | 2-$HOC_6H_4CHO$ | $CH_3$ | 20 | 97 | 182–185 | 185–187 [18] |
| 13 | 4-$FC_6H_4CHO$ | $CH_3$ | 30 | 88 | 217–220 | 219–221 [27] |
| 14 | 4-$CH_3C_6H_4CHO$ | $CH_3$ | 5 | 95 | 224–226 | 226–228 [25] |
| 15 | 2-$CH_3C_6H_4CHO$ | $CH_3$ | 15 | 89 | 238–241 | 242–244 [22] |
| 16 | 4-$BrC_6H_4CHO$ | H | 23 | 92 | 285–287 | 279–282 [18] |
| 17 | 4-$NO_2C_6H_4CHO$ | H | 50 | 91 | 252–254 | 252–254 [22] |
| 18 | 4-$HOC_6H_4CHO$ | H | 40 | 97 | 258–260 | 258–260 [31] |
| 19 | 4-$(CH_3)_2NC_6H_4CHO$ | H | 15 | 95 | 254–256 | 256–258 [31] |

[a]Isolated yields.

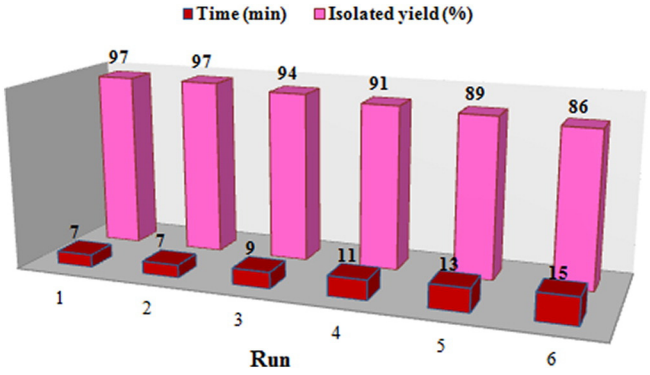



**Fig. 7.** Recyclability of the catalyst in the reaction reported in Table 3, entry 2.

reacted and afforded high to excellent yields of the pure products under the selected conditions. The nature and electronic properties of the substituents had no obvious effect on the rate and reaction yields.

It is important to note that some of the reactions were also tested on a larger scale without any difficulty by using only 0.1 mmol (10 mol%) of [SuSA-H] $HSO_4$. For example, the reaction of 5 mmol 4-chlorobenzaldehyde (Table 3, entry 2) was investigated in the presence of 0.1 mmol of the catalyst. The reaction was completed within 10 min and the desired product was obtained in 97% yield. This result indicates that a large scale reaction is also feasible using a lesser amount of the ionic liquid catalyst without significant loss of the yields.

To check the reusability of the catalyst, the reaction of 4-chlorobenzaldehyde (Table 3, entry 2) under the optimized reaction conditions was studied. After separation of the product, the catalyst was recovered by evaporation of water, washed with $Et_2O$, dried at 50 °C under vacuum for 1 h and reused for the same reaction. This process was carried out over six runs and all reactions led to the desired product with high efficiency (Fig. 7). It should also be noted that the FT-IR of the recovered catalyst clearly was similar to the fresh catalyst. This result suggests that the catalyst remains intact during the course of the reaction (Fig 8).

Table 4 compares our results with the results reported in the literature using some of the other catalysts in the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives.

The presented results show that when the same reactions are carried out in the presence of sulfuric acid in addition to the necessity of the larger amounts of the catalyst and the use of [bmim]$BF_4$ as the solvent, the reaction times are longer compared with the present method (Table 4, entry 6). On the other hand although in some of the reported cases smaller amounts of the catalysts are used but the use of an ionic liquid as the solvent is needed (Table 4, entries 4, 5).

To illustrate the efficiency of our method, the preparation of 13-(4-chlorophenyl)-3,3-dimethyl-3,4-dihydro-1*H*-indazolo[2,1-b]phthalazine-1,6,11(2*H*,13*H*)-trione was also studied under the optimized conditions in the presence of SuSA. The obtained results clarified that in this situation the reaction time is longer compared with [SuSA-H]$HSO_4$ (Table 4, entry 8).

## 4. Conclusion

In this study, succinimidinium *N*-sulfonic acid hydrogen sulfate, is simply prepared from the commercially available starting materials and characterized with a variety of techniques. This new ionic liquid can be used as a catalyst in the preparation of 2*H*-indazolo[2,1-*b*]phthalazine-trione derivatives under thermal solvent-free conditions. This method is more secure than the other methods which in them the mineral acids like $H_2SO_4$ are used as the catalysts. Mild reaction conditions, easy work-up, short reaction times and high yields of the products are significant advantages of this method. Also, this ionic liquid could be successfully recovered and recycled at least for six runs without significant loss in its activity.

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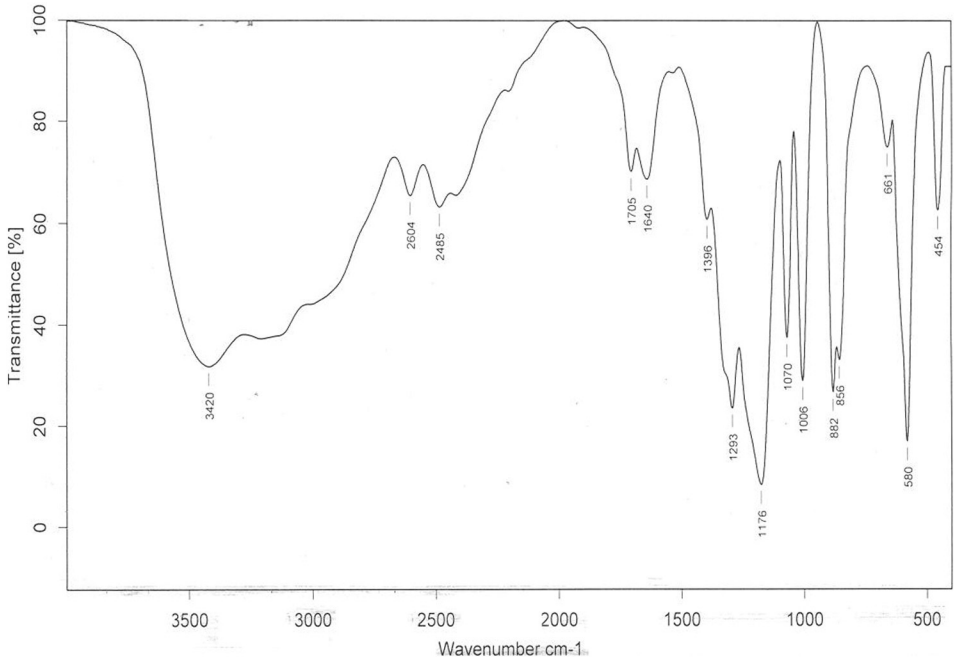


**Fig. 8.** FT-IR recovered catalyst after six run.

**Table 4**
Comparison of the results obtained from the synthesis of 13-(4-chlorophenyl)-3,3-dimethyl-3,4-dihydro-1*H*-indazolo[2,1-b]phthalazine-1,6,11(2*H*,13*H*)-trione using [SuSA-H] $HSO_4$ with those obtained using other catalysts.

| Entry | Catalyst loading (mol %) | Conditions | Time (min) | Yield (%) | Reference |
|---|---|---|---|---|---|
| 1 | SSA (6.5) | Solvent-free, 100 °C | 7 | 91 | [11] |
| 2 | Nano-alumina sulfuric acid(23.2) | Solvent-free, 110 °C | 12 | 98 | [20] |
| 3 | MTSA (15) | Solvent-free, 100 °C | 15 | 80 | [32] |
| 4 | Tungstosilicic acid (1) | [bmim] [$PF_6$], 100 °C | 60 | 75 | [33] |
| 5 | Phosphotungstic acid (3) | [bmim]$BF_4$ (2 mL), r.t. | 8 | 92 | [34] |
| 6 | $H_2SO_4$ (15) | [bmim]$BF_4$(0.5 mL), 80 °C | 30 | 88 | [35] |
| 7 | [Simp]$HSO_4$ (10) | Solvent-free, 100 °C | 17 | 82 | [36] |
| 8 | SuSA (10) | Solvent-free, 100 °C | 15 | 90 | This work |
| 9 | [SuSA-H] $HSO_4$ (10) | Solvent-free, 100 °C | 7 | 97 | This work |

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