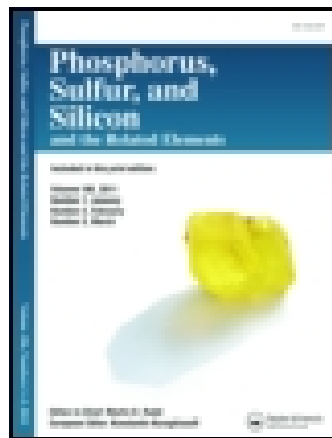


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### Zeolite H-SDUSY Powder (CBV720) as a Recyclable Catalyst for an Efficient Thiocyanation of aromatic and heteroaromatic compounds in acetonitrile

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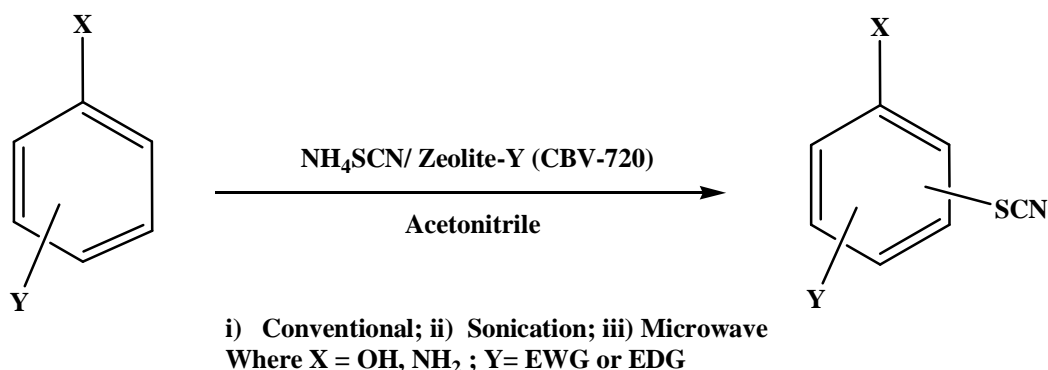
**Zeolite H-SDUSY Powder (CBV720) as a Recyclable Catalyst for an Efficient Thiocyanation of aromatic and heteroaromatic compounds in acetonitrile**

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**Abstract:** Thiocyanation of various aromatic and heteroaromatic compounds has been studied using Zeolite H-SDUSY Powder (CBV720) as catalyst and ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ) as thiocyanation reagent in acetonitrile medium. Reactions afforded good yields of products under stirred conditions at reflux temperature. Reactions underwent enhancements under sonication (using an ultrasonic probe of 24 kHz frequency) and microwave irradiation. The use of ultrasound decreased the reaction times from (7 -12h) to few minutes (70-150 min). The use of microwave irradiation much more effectively enhanced the reaction rates than sonicated and conventional protocols used in this study.



**Keywords:** Zeolite H-SDUSY(CBV720); ammonium thiocyanate; thiocyanation; organic compounds; sonication; microwave irradiation; rate accelerations

## INTRODUCTION

The electrophilic thiocyanation of aromatic and heteroaromatic compounds is an important carbon-heteroatom bond formation reaction in organic synthesis <sup>1</sup>. Thiocyanate is a versatile synthon that can be readily transferred to pharmaceutically important functional groups<sup>2</sup> such as sulfide, aryl nitrile, thiocarbamate, and thionitrile to produce drugs. This versatile nature of thiocyanate group attracted several groups of workers, who embarked on taking up thiocyanation of aromatic and heteroaromatic compounds <sup>3-5</sup> under varied conditions in order to explore user-friendly methodologies. Oxidizing catalysts such as ceric ammonium nitrate (CAN) <sup>6a</sup>, ferric chloride (FeCl<sub>3</sub>)<sup>6b</sup>, Oxone<sup>6c</sup>, 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ)<sup>6d</sup>, (HIO<sub>3</sub>)<sup>6e</sup>, iodine pentoxide (I<sub>2</sub>O<sub>5</sub>)<sup>6f</sup>, *p*-Toluenesulfonic acid (*p*-TSA)<sup>6g</sup>, have been recently employed for efficient and regioselective thiocyanation of aromatic and heteroaromatic compounds in presence of ammonium thiocyanate as reagent. Sajjadifar et al recently reported simple and highly efficient catalytic protocols<sup>7</sup> for thiocyanation of aromatic compounds using H<sub>2</sub>O<sub>2</sub> /KSCN and H<sub>5</sub>IO<sub>6</sub>/KSCN in aqueous media.

An increasing demand for the design of catalysts, which can be easily separated from products and recycled, is still the subject of investigation from green chemistry point of view. Micro and mesoporous materials offer unique opportunities for heterogeneous catalysis by their large surface area. Zeolites are a special class of aluminosilicates that contain silicate and aluminate ions as backbones along with a few vacant positions in their structures <sup>8,9</sup>. Because of the large vacant spaces in three-dimensional structures, Zeolites give room for cations such as sodium and calcium and molecules such as water. These spaces are interconnected and form long channels and pores, which are of different sizes in different Zeolites. Even though several Zeolites have

found their use as efficient catalysts, not much work appears to have been reported on Zeolite H-SDUSY Powder (CBV720). These reasons together with the versatility of SCN functionality stimulated us to take up the Zeolite H-SDUSY Powder (CBV720)/ ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ) triggered thiocyanation of aromatic and hetero aromatic compounds. The control experiments of thiocyanation did not proceed even at elevated temperatures and after 24 h of reaction time in dichloromethane, dichloroethane, and toluene. Even in acetonitrile medium, also the reactions were sluggish. Therefore, the authors took up the present study under sonication and microwave irradiation for effective thiocyanation encouraged by earlier success reports and advantages of ultrasound assisted<sup>10-12</sup>, and microwave assisted chemical reactions<sup>13-15</sup>.

## RESULTS AND DISCUSSION

Aromatic compounds upon treatment with (0.10 mol) ammonium thiocyanate in presence of Zeolite-Y (CBV-720) as a catalyst in acetonitrile medium afforded thiocyanate derivatives in very good yield (Scheme-1). Nature of solvent plays an important role in altering the rate of a reaction, therefore, we have studied the thiocyanation of phenol using it as a model substrate in various solvents such as chloroform, dichloromethane (DCM), dichloroethane (DCE), n-hexane, tetra hydro furan (THF) and acetonitrile (MeCN) at room temperature. Another important aspect is to optimize the reaction conditions i.e., the concentrations of catalyst (Zeolite-Y), substrate and  $\text{NH}_4\text{SCN}$ . Practically no trace of product was observed in chloroform, DCM, DCE, THF and n-hexane media, even though a wide concentration of Zeolite-Y (5 to 100 mg i.e., 0.26-5.20 mol %) is used as catalyst. Nevertheless, the reaction went much more smoothly in ACN with small amount of catalyst (0.26-mol percentage Zeolite-Y), 1.0 millimolar substrate and 2.0 millimolar of  $\text{NH}_4\text{SCN}$ . Therefore, reactions with other substrates were studied using 0.26 mol percentage

(or 5.0 mg) of Zeolite-Y. A wide range of active, moderately active, and non-active aromatic and heterocyclic compounds were taken for Zeolite-Y triggered thiocyanation under conventional and non-conventional conditions and the corresponding results are presented in Table 1.

**[Insert Table 1]**

Even though the presented data reflects that thiocyanation reactions require longer reaction times are (7 to 12 h), it is of interest to note that *para* substituted compounds underwent thiocyanation only at the *ortho* position, while *ortho* substituted compounds afforded the *para* derivative in very good yield. It is also of interest to note that aromatic hydrocarbons such as  $\alpha$ -methoxynaphthalene underwent smooth thiocyanation with good yields. Since the reactions required longer reaction times (7-12h) under convention conditions, we have tried ultrasonically assisted and microwave assisted methods to enhance the rates of the reactions. In the recent past ultrasonically assisted (USAR) and microwave-assisted (MWAR) reactions have received considerable attention because of their immense importance to promote and enhance a broad spectrum of synthetic organic reactions<sup>10-15</sup>. These techniques have become helpful to chemists to overcome many of the difficulties associated with conventional reactions related to their environmental shortcomings. All the reactions in the present study underwent dramatic rate enhancements, and maintained their desirable selectivity and ease of experimental manipulation. The reaction times were reduced from 7-12 h (under conventional conditions) to about 70 – 90 min in USA reactions and about 1-4 min in MWA reactions.

Rate accelerations of the ultrasonically assisted thiocyanation reactions (USNR) in the present study are due to cavitation, a physical process that creates, enlarges, and implodes gaseous and

vaporous cavities in an irradiated liquid<sup>10-13</sup>. Cavitation is a process in which mechanical activation destroys the attractive forces of molecules in the liquid phase. Applying ultrasound, compression of the liquid is followed by rarefaction (expansion), in which a sudden pressure drop forms small, oscillating bubbles of gaseous substances. These bubbles expand with each cycle of the applied ultrasonic energy until they reach an unstable size; they can then collide and/or violently collapse. Cavitation induces very high local temperatures in the liquid and enhances mass transfer.

On the other hand the observed rate and yield enhancements in MWA reactions<sup>14,15</sup> is probably due to a combination of thermal effects, arising from the heating rate, superheating or “hot spots” and the selective absorption of radiation by polar substances which ultimately causes bulk activation of molecules. It is important to note that the rate of the reaction has a direct dependence on the fraction of activated/energized species. We have tried to compare the results obtained in the present study with earlier reports on thiocyanation and presented the data in table-2. These results are found to be in comparable range with earlier reports. More particularly the reaction times under microwave conditions are lesser than earlier protocols indicating that the present methodology is also a sincere effort in the development of new eco-friendly protocol for thiocyanation of aromatic compounds.

### **Conclusions**

In summary, the authors have developed Zeolite H-SDUSY Powder (CBV720) /ammonium thiocyanate triggered electrophilic thiocyanation of aromatic and heteroaromatic compounds in acetonitrile medium under conventional, sonication and micro wave irradiation. These newly developed methods have several advantages such as reusability of the catalyst, simple workup

procedure, good selectivity, short reaction times and high product yields. The longer reaction times of conventional methods (7-12 h) were remarkably reduced to about (70 – 150) min in sonicated and (1-4) min in MW assisted reactions.

## EXPERIMENTAL

### Materials and methods

Chemicals were obtained from S.D fine Chemicals (India), Avra fine Chemicals (India), and Aldrich Chemical Company and used without further purification. CBV720 (Si/Al bulk = 13) was purchased from Zeolyst International. It was supplied in H-form. A detailed characterization of this catalyst is well documented in literature and effectively used as a catalyst<sup>9</sup>.

### A typical procedure for thiocyanation of organic compounds

In a typical experiment, zeolite catalyst (0.26-0.52 mol percentage), substrate (0.1 mol), and ammonium thiocyanate (0.2 mol) in MeCN (20 mL) were taken in a reaction flask and constantly stirred for about 10 -12 h at reflux temperature, depending on the nature of the substrate. The progress of the reaction was monitored by TLC (ethyl acetate: n-hexane 1:9). After completion of the reaction, the reaction mixture was treated with DCE, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated to obtain the product. The products were identified by IR, <sup>1</sup>H NMR and MS spectra. IR spectra showed the characteristic peaks of the –SCN group between 2120 and 2150 cm<sup>-1</sup> and the C–S stretching between 650 and 750 cm<sup>-1</sup>. The results agreed well with literature reports<sup>3-7</sup>.

### Typical Procedure for ultrasonic assisted (USAR) thiocyanation of organic compounds

Method of thiocyanation under sonication is similar to the one described in the preceding section. The reaction vessel containing zeolite catalyst (0.26-0.52 mol percentage), substrate

(0.1 mol), and ammonium thiocyanate (0.2 mol) in MeCN (20 mL) was placed in a Sonicator and the progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was filtered, washed with water and worked up as described in the conventional approach. All products are characterized by comparison of their IR, <sup>1</sup>H-NMR and Mass spectra and with their physical data.

#### **Procedure for Microwave Assisted (MWAR) thiocyanation of organic compounds**

A CEM microwave reactor was used, which was equipped with temperature, pressure, and power control units. An oven-dried microwave vial was charged with a mixture containing zeolite catalyst (0.26-0.52 mol percentage), substrate (0.1 mol), and ammonium thiocyanate (0.2 mol) in silica gel slurry, and irradiated in a microwave (power input 140 W) at 150°C for few minutes. After completion of the reaction, as ascertained by TLC, the reaction mixture was treated with sodium bicarbonate; the organic layer was diluted with dichloromethane (DCM), and separated from the aqueous layer. The crude product mixture was purified by recrystallization with an ethyl acetate -DCM mixture in 2:3 ratios. The purity was checked with TLC. The products were identified by characteristic spectroscopic data.

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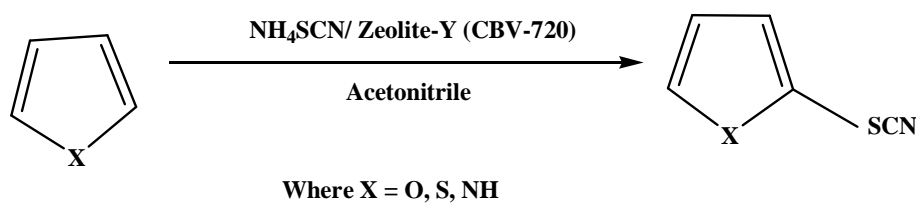
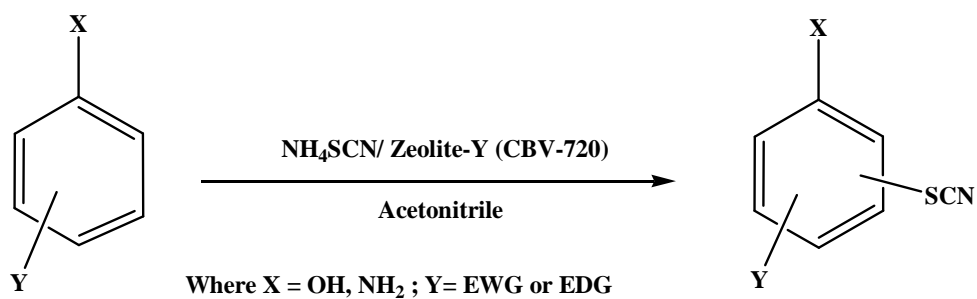
**Table 1:** Ultrasonic and microwave assisted thiocyanation of certain aromatic and heteroaromatic compounds using (Zeolite H-SDUSY Powder (CBV720) /NH<sub>4</sub>SCN) in acetonitrile

S. N	Substrate	Product <sup>3-7</sup>	Conventional		Sonication		MWAR	
			R. T (hrs)	Yield (%)	R. T (min)	Yield (%)	R. T (sec)	Yield (%)
1	Phenol	2-Thiocyanato phenol	10	65	90	68	55	72
2	<i>o</i> -Cresol	2-Methyl-4-thiocyanato phenol	8	67	70	72	50	75
3	<i>m</i> -Cresol	3-Methyl-4-thiocyanato phenol	9	70	75	72	60	78
4	4-Bromo phenol	4-Bromo-2-thiocyanato-phenol	9	66	70	70	50	72
5	Aniline	4-Thiocyanato aniline	10	75	90	78	70	80
6	<i>o</i> -Chloro aniline	2-Chloro- 4-thiocyanato aniline	9	68	70	71	60	74
7	<i>m</i> -Methoxy-aniline	3-Methoxy-4-thiocyanato-aniline	10	60	100	65	90	70
8	N-Methyl aniline	4-Thiocyanato-N-methyl aniline	11	68	120	72	120	78
9	N,N-Dimethyl aniline	4-Thiocyanato-N,N-dimethyl aniline	13	66	150	72	240	80
10	Furan	2-Thiocyanato furan	7	74	50	78	40	80
11	Diphenylamine	4-Thiocyanato diphenylamine	8	65	80	72	60	75

12	Indole	3-thiocyanato-1H-indole	8	70	70	72	60	76
13	1-Methyl-indole	1-Methyl-3-thiocyanatoindole	8	75	70	74	60	77
14	5-Bromo-indole	5-Bromo-3-thiocyanatoindole	8	85	70	86	60	86
15	2-Methyl-Indole	2-methyl 3-thiocyanato-indole	8	80	70	78	60	79
16	Pyrrole	2-thiocyanato-1H-pyrrole	8	72	70	75	50	78
17	Thiophene	2-Thiocyanato thiophene	10	66	90	70	70	74
18	1-Methoxy naphthalene	1-Methoxy-4-thiocyanato naphthalene	12	68	70	72	60	75

**Table 2:** Comparison of the isolated yields for the thiocyanation of Aniline to 4-thiocyanato aniline with reported classical methods

Catalyst	Reaction Time & Conditions	Yield (%)	Reference
CBV720/MW assisted reaction	70 (sec)	80	Present work
P4VP-SCN	8( min) 0-5°C	85	4(a)
Aryldiazonium salt	--	84	3(h)
Copper powder	--	56	3(i)
1-methyl-3-(2-(sulfooxy)ethyl)-1H-imidazol-3-ium thiocyanate.	7 (min)	90	3(j)
NH <sub>4</sub> VO <sub>3</sub> / MW assisted reaction	5 -7 min	80	5(f)



i) Conventional; ii) Sonication; iii) Microwave

Scheme - 1: Zeolite-Y (CBV-720)/ NH<sub>4</sub>SCN triggered Thiocyanation of Aromatic and Heterocyclic Compounds