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Green and Efficient Method for Thiocyanation of Aromatic and Heteroaromatic Compounds Using Crosslinked Poly (4-Vinylpyridine) Supported Thiocyanate Ion as Versatile Reagent and Oxone as Mild Oxidant

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GREEN AND EFFICIENT METHOD FOR THIOCYANATION OF AROMATIC AND HETEROAROMATIC COMPOUNDS USING CROSS-LINKED POLY (4-VINYLPYRIDINE) SUPPORTED THIOCYANATE ION AS VERSATILE REAGENT AND OXONE AS MILD OXIDANT

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GRAPHICAL ABSTRACT



Abstract A green and efficient regioselective thiocyanation of indoles, anilines and pyrrole has been achieved via a simple protocol using cross-linked poly (4-vinylpyridine) supported thiocyanate ion, [P₄-VP]SCN, as a versatile polymeric reagent and oxone as an environmentally friendly and mild oxidant. Various indoles, anilines, pyrroles, and carbazoles were transformed into their corresponding aryl thiocyanates in high to excellent yields in a short reaction time. The present procedure offers advantages such as simple reaction work-up, and the polymeric reagents can also be regenerated and reused several times without significant loss of their activity.

Keywords Thiocyanation; oxone; regioselectivity; polymeric reagent; aryl thiocyanate

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INTRODUCTION

Arylthiocyanates are well known in the area of organosulfur chemistry¹ and have found widespread applications such as insecticides, biocidal, antiasthmatic, vulcanization accelerators, and starting materials for the preparation of heterocycles.^{2–5} The thiocyanation of aromatics and heteroaromatics is an important carbon-hetero atom bond formation in organic synthesis and constitutes an interesting group, which could be readily transformed into other sulfur-bearing functionalities,^{6,7} especially for producing drugs and pharmaceuticals.^{8,9} Also, thiocyanate is a versatile synthon which can be readily transferred to other functional groups such as sulfide,^{10–13} nitrile,¹⁴ thiocarbamate,^{15,16} thionitrile,¹⁷and arylthioesters.¹⁸ Therefore, it is important to find new methods for the thiocyanation systems.

Several methods for the thiocyanation of aromatic systems using a variety of reagents such as bromine/potassium thiocyanate (only for indoles),¹⁹ N-thiocyanatosuccinimide (only for 5-methoxy-2-methylindole and accompanied by two bisthiocyanates²⁰ trichloroisocyanuric acid/NH₄SCN/wet SiO₂,²¹ cerium(IV) ammonium nitrate/NH₄SCN,²² acidic montmorillonite K10 clay/NH₄SCN,²³ iodine/methanol/NH₄SCN,²⁴ silica boron sulfonic acid/H₂O₂/NH₄SCN,²⁵ sodium pertborate/NH₄SCN,²⁶ oxone/NH₄SCN,²⁷ diethyl azodicarboxylate,²⁸ diphenylphosphinite ionic liquid,²⁹ potassium peroxydisulfate-copper(II),³⁰ ferric(III) chloride/NH₄SCN,³⁵ and para-toluene sulfuric acid/NH₄SCN³⁶ have been explored. However, these methodologies suffer from one or more drawbacks such as the less availability or hard preparation of starting materials,^{19,20} the requirement for a large excess of oxidizing reagents, low yields for some compounds,^{22,27} and performances under certain special conditions.²³

Potassium peroxymonosulfate is a cheap and readily accessible oxidizing reagent. It is commonly referred to as oxone (2KHSO₅.KHSO₄.K₂SO₄) and is a versatile and mild oxidant for the transformation of a wide range of functional groups.³⁷

Recent developments in polymer-supported reactions have led to the propagation of combinatorial chemistry as a method for the rapid and efficient preparation of novel functionalized molecules.³⁸ An interesting and fast growing branch of this area is polymer-supported reagents.³⁹ Although polymeric reagents and scavengers have been used in organic synthesis for decades, the development of combinatorial and parallel high throughput synthesis techniques brought this class of reagents to a wider attention. The first compound collections were based on peptides and oligonucleotides, which were stepwise assembled on a solid support,⁴⁰ following the concept developed by Merrifield.⁴¹ In recent years, polymeric reagents, especially anion exchange resins, have been widely applied in organic transformations.^{42–61} The advantages of this technique over conventional classical methods are mild reaction conditions, safe handling, rapid, and very simple work-up. On the other hand the spent polymeric reagents can usually be regenerated and reused several times without significant loss of their activity. In addition, many ion-exchange resins, and indeed reagents supported on them, are commercially available and are relatively inexpensive.

As far as we know there are a few reports in the literature on the application of polymer-supported thiocyanate ion^{42–45} but, to the best of our knowledge, there are no reports on polymer-supported thiocyanate ion for thiocyanation of aromatic or heteroaromatic rings. We have recently reported an efficient method for preparation of cross-linked poly (4-vinylpyridine) supported thiocyanate ion, $[P_4-VP]SCN$, and applied for synthesis of alkyl thiocyanates from alkyl halides⁴⁴ and for synthesis of aryl thiocyanates via dediazoniation-thiocyanation of arylamines.⁴⁵

Entry	Solvent	[P ₄ -VP]SCN (mmol of SCN ion)	Oxone (mmol of KHSO ₅)	Time (min)	Yield ^a (%)
1	CCl ₄	2.00	1.50	120	0.0
2	H ₂ O	2.00	1.50	30	45
3	C ₂ H ₅ OH	2.00	1.50	65	93
4	CH ₃ CN	2.00	1.50	30	96
5	CH ₃ COCH ₃	2.00	1.50	70	95
6	CH ₃ OH	2.00	1.50	10	96
7	CH_2Cl_2	2.00	1.50	120	0.0
8	CH ₃ OH	2.00	2.00	10	95
9	CH ₃ OH	2.00	1.00	10	74
10	CH ₃ OH	2.50	1.50	10	96
11	CH ₃ OH	1.50	1.50	10	61

 Table 1 Optimization of the reaction conditions for thiocyanation of indole (1mmol) in different solvents and different molar ratio of [P₄-VP] SCN/oxone

^aIsolated yields.

In this report, we wish to disclose a simple, convenient, and efficient protocol for the thiocyanation of indoles, anilines, pyrrole, carbazole, and aryl amines using $[P_4-VP]SCN$ as versatile polymeric reagent and oxone as a mild and environmentally friendly oxidant.

RESULTS AND DISCUSSION

In this paper, we report the first procedure for facile and rapid thiocyanation of indoles, anilines, pyrrole, carbazole, and aryl amines using $[P_4-VP]SCN$ as versatile polymeric reagent and oxone as a mild and environmentally friendly mild oxidant.

Cross-linked poly (4-vinylpyridinium) thiocyanate ion, [P₄-VP]SCN, was prepared according to our previously reported method⁵² via the reaction of quarternized crosslinked poly (N-methyl-4-vinylpyridinium) iodide, [P₄-VP]I, with an aqueous solution of KSCN. A variety of activated aromatics such as anilines and heterocyclic aromatics such as indoles, pyrrole, and carbazole were also subjected to thiocyanation reaction in methanol.

Since the nature of the solvent influences the rate of reaction, the thiocyanation of indole (1 mmol) as a model substrate was performed in various solvents such as H_2O , ethanol, methanol, acetone, acetonitrile, CCl_4 , and CH_2Cl_2 at room temperature. According to the data presented in Table 1, methanol has selected as the best solvent (entry 6). One reason for the observed different reaction times could be the solubility of the starting materials in the solvents used in this study. The next step has the optimization of $[P_4-VP]SCN/oxone$ molar ratio in the thiocyanation reaction in methanol were tested. The results presented in Table 1 showed that the optimized molar ratio of $[P_4-VP]SCN/oxone$ was 2/1.5 (entry 6).

Under the optimized reaction conditions, the thiocyanation of different aromatic and heteroaromatic compounds such as anilines, indoles, pyrrole, carbazole, and aryl amines were investigated and the results are summarized in Table 2. The reaction of indole at room temperature yielded the desired product 3-thiocyanato-1H-indole in 96% yield (Table 2, entry 1). The same reaction using N-methyl indole as the starting material gave 3-thiocyanato-N-methyl indole in 98% yield (Table 2, entry 6). The reaction was further extended to include other substituted indoles. It was found that 2-methylindole gave

Ģ	Mp.	Reported	73-76 ³³ , 105-106 ³⁴	73–76 ³³ , 105–106 ³⁴	73–76 ³³ , 105–106 ³⁴	73-76 ³³ , 105-106 ³⁴	(Continued on next page)
t room temperatur		Found	75-76	75–76	75–76	75-76	
l/oxone in methanol a		Yield (%) ^a	96	96	95	96	
unds with [P4-VP]SCN		Time (min)	0	0	=	13	
n of aromatic and heteroaromatic compo		Product	sc.	N N N N N N N N N N N N N N N N N N N			:
Table 2 Thiocyanatio		Substrate		ZI			
		Entry	_	d D	ę	4	

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	Table 2 Thiocyanation of a	romatic and heteroaromatic compounds with $[P_4]$	-VPJSCN/oxone in n	nethanol at room te	mperature (<i>Con</i>	inued)
						Mp.
Entry	Substrate	Product	Time (min)	${ m Yield}~(\%)^{ m a}$	Found	Reported
Sb		sov	15	94	75–76	$73-76^{33}$, 105-106 ³⁴
	XH					
9		NON HIS	10	86	82–84	83-84 ³⁴ , 76-78 ²⁵
L	GH ²	CH ₃ SCN	10	94	100-102	99–101 ³⁴ , 104–106 ²⁵
	CH3	-CH3				
8	HN	XH	20	85	51-52	52–53 ³³ , 96–98 ²⁶
		H ₂ N—SCN				



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	Table 2 Thiocyanation	of aromatic and heteroaromatic compounds with [P4-VP]SCN/o	xone in methanol	at room temperat	ure (<i>Continue</i>	<i>(p</i>
						Mp.
Entry	Substrate	Product	Time (min)	Yield (%) ^a	Found	Reported
13	HaHN	PhHN-SCN 60% +	20	85	60–62	58-60 ³⁵ , 62-64 ³⁴
		25%				
14		SCN	10	82	0il	01134.35 01134.35
15	H	H	120	70	79–81	8081 ³⁴
a rool are	od vields	H				

brough years. ^bThe entries 2–5, refer to the use of the [P4-VP]SCN that is recycled first, second, third, and fourth time, respectively, under identical conditions. ^cIn this procedure, the melting point was not reported.

94% yield of 3-thiocyanato-2-methylindole (Table 2, entry 7). The lower yield is probably attributed to the steric hindrance of 2-substituted indole.

Indole was chosen as a model substrate and reusability of the polymer was tested by using $[P_4-VP]SCN$ that is recycled first, second, third, and fourth time, respectively, under identical conditions and the results are summarized in Table 2 (entries 2–5).

After successfully developed the thiocyanation reactions of indoles, we then explored the reactions of aniline and its derivatives. Aniline is an electron-rich system bearing a free-amino group. The reaction of [P₄-VP]SCN/oxone with aniline performed under the same condition described above afforded desired product (4-thiocyanatoanilline) in 85% vield (Table 2, entry 8). Different mono and di-N-substituted anilines were also reacted with [P₄-VP]SCN/oxone, and they all gave high yields (85–88%) of the corresponding products (Table 2, entries 9–12). As Table 2 reveals, aromatic amino compounds were readily converted to the mono thiocyanated products with high para-selectivity (Scheme 1 and Table 2, entries 8-12) and the thiocyanato group is selectively added to the para position of the amino group. These observations are also supported by other reported methods.^{21,25–27,33–36} One exception was observed when diphenylamine was subjected to this approach because mono and di thiocyanated products could be produced (60% mono and 25% di thiocyanated products were separated) (Table 2, entry 13). The same result has been reported by Nair et al. when used cerium(IV) ammonium nitrate for thiocyanation of diphenylamine by ammonium thiocyanate (32% mono and 6% dithiocyanated products were separated).²² As Table 2 reveals, using indoles (entries 1–7) and carbazole (entry 15) as substrates, the reaction gave unique 3-thiocyano substituted indoles in high yields (94–98%) and unique 3-thiocyano substituted carbazole in good yield (70%) but, unique 2-thiocyano substituted pyrrole was obtained in 82% isolated yield when, thiocyanation of pyrrole was treated (entry 14).

The regiochemistry of substitution was achieved by the interpretation of ¹H NMR spectra and their comparison with the spectra and physical data of authentic samples.

On the other hand, one of the disadvantages of polymeric reagents is their expense, but in this case, the spent polymeric reagents can in principle be recycled (Scheme 1, step 8) and reused many times (Table 2, entries 2–5). Preparation of the $[P_4-VP]SCN$ (steps 1 and 2), regioselective thiocyanation of aromatic and heteroaromatic compounds (steps 3–7) and regeneration of the polymer (step 7) are shown in Scheme 1.

Entry	Time (min)	Yield (%)	Mp.	Ref.
1	120	83	73–76	33
2	20	97	105-106	34
3	15	95	78	26
4	45	88	_	36
5	50	85	_	24
6	43	98	72-73	27
7	20	93	70-72	35
9	10	96	73–74	А

Table 3Comparison of the result of the present work for the model reaction with the previous reported methodin literature $^{21,25-27,33-36}$

A: Present method Table 2 (entry 1).



 R^1 and $R^2=H$, Me, Et or Ph; R^3 and $R^4=H$ or M

[P4-VP] 2% DVB : Poly (4-vinylpyridine) cross-linked with 2% divinyl benzene



The arylthiocyanate products were characterized by FT-IR; and ¹H- and ¹³C-NMR spectroscopy and physical properties were compared known compounds. IR spectrum showed the characteristic peak of –SCN between 2145–2160 cm⁻¹ and the –C–S stretching at 642–755 cm⁻¹. The characteristic spectral data of some arylthiocyanate products are presented as supplemental materials (Table S_h1).

In Table 3, the result of the present work for the model reaction was compared with previously reported methods.^{25–27,33–36} The reaction time in the present method is shorter than that in the previously reported methods (Table 3, entry 9). This can probably be attributed to the local concentration of thiocyanate ion species inside the pores of the polymer.

The reduction potential of oxone and the oxidation potential of indole and ammonium thiocyanate in anhydrous methanol were determined by Wu and coworkers.²⁷ The reduction and oxidation potentials of oxone and indole were estimated to be +0.325 V and -1.050 V, respectively. Yet, the thiocyanate ion exhibited no oxidation potential. So, it might be assumed that oxone oxidized indole rather than the thiocyanate ion. The oxidation occurs at the C–C double bond on the hetero-, five-membered ring of indole, giving a cation radical of indole which was stabilized by a resonance effect. It was followed by nucleophilic attack of -SCN at the three position. The radical intermediate so formed then undergoes a 3-H-abstraction by OH, that generated from oxone ⁶⁴ during its reduction, affording the end product, as depicted in Scheme 2.



The advantages of this method over conventional classical methods are mild reaction conditions, safe handling, rapid, and very simple work-up. In addition, there is current research and general interest in heterogeneous systems because such systems are important in industry and developing technologies.⁶⁵

CONCLUSIONS

We have developed an efficient, rapid, experimentally simple method for regioselective thiocyanation of indoles, pyrrole, anilines, and carbazole via a green and simple protocol using cross-linked poly (4-vinylpyridine) supported thiocyanate ion as versatile polymeric reagent and oxone as a mild and environmentally friendly oxidant. The spent polymeric reagent can be easily separated by filtration and can be easily regenerated by treated with aqueous solution of KSCN and reused for several cycles without significant loss of their activity.

EXPERIMENTAL

Materials and Instruments

Chemicals were either prepared in our laboratory or were purchased from Fluka (Buchs, Switzerland), Aldrich (Milwaukee, WI), and Merck chemical companies. Poly (4-vinylpyridine) cross-linked with 2% divinyl benzene (DVB), (white powder, and 100–200 mesh), [P₄-VP] 2% DVB, was purchased from Fluka (Buchs, Switzerland). Cross-linked poly (N-methyl-4-vinylpyridinium) iodide, [P₄-VP]I, and cross-linked Poly (N-methyl-4-vinylpyridine) thiocyanate, [P₄-VP]SCN, were synthesized according to our reported procedures.⁴⁴ Progress of the reaction was monitored by thin layer chromatography (TLC) using silica gel Poly Gram SIL G/UV 254 plates (Fluka). All products were characterized by comparison of their melting point, FT-IR, and in some cases 1H-NMR spectral data, with those of known samples and all yields refer to the isolated pure products. Melting points were determined with a Buchi melting point B-540 B.V. CHI apparatus. The Supplemental Materials file contains characterization data for the previously known compounds as products of this reaction.

Preparation of [P₄-VP]SCN

Cross-linked poly (N-methyl-4-vinylpyridinium) thiocyanate, [P₄-VP]SCN, was synthesized and its capacity was determined according to our reported procedure (Scheme 1).⁴⁴

The obtained capacity of the polymer was 3.3 mmol of thiocyanate ion per gram of polymer.

General Procedure for Thiocyanation of Aromatic or Heteroaromatic Compounds Using [P₄-VP]SCN/Oxone

To a suspension of $[P_4-VP]SCN$ (625 mg, 2.00 mmol of SCN ion) and an aromatic or heteroaromatic compound (1 mmol) in methanol (10 mL) was added 924 mg of oxone (1.5 mmol) and the reaction mixture was stirred at room temperature for the appropriate time according to Table 2. The progress of the reaction was monitored by TCL [eluent: nhexane/ethyl acetate (8/2)]. Then the polymer was separated by filtration and the filtrate was diluted with water (15 mL) and extracted with dichloromethane (4 × 8 mL). The combined organic layer was dried over anhydrous Na₂SO₄ and the solvent was evaporated under reduced pressure. The resulting crude product was purified by column chromatography on silica gel (eluted with n-hexane/ethylacetate: 8/2) to afford the corresponding thiocyanated products.

Regeneration of [P₄-VP]SCN

The spent polymer (1.00 g) was added to an excess aqueous solution of KSCN and was stirred for 24 h at room temperature. The mixture was filtered and washed several times with distilled water and ethanol and dried overnight under vacuum in the presence of P_2O_5 at 40 °C (Scheme 1, step 8). The regenerated polymer can be reused several cycles without losing significantly its activity (Table 2, entries 2–5).

SUPPLEMENTAL MATERIAL

Supplementary data for this article can be accessed on the publisher's website, www.tandfonline.com/gpss

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