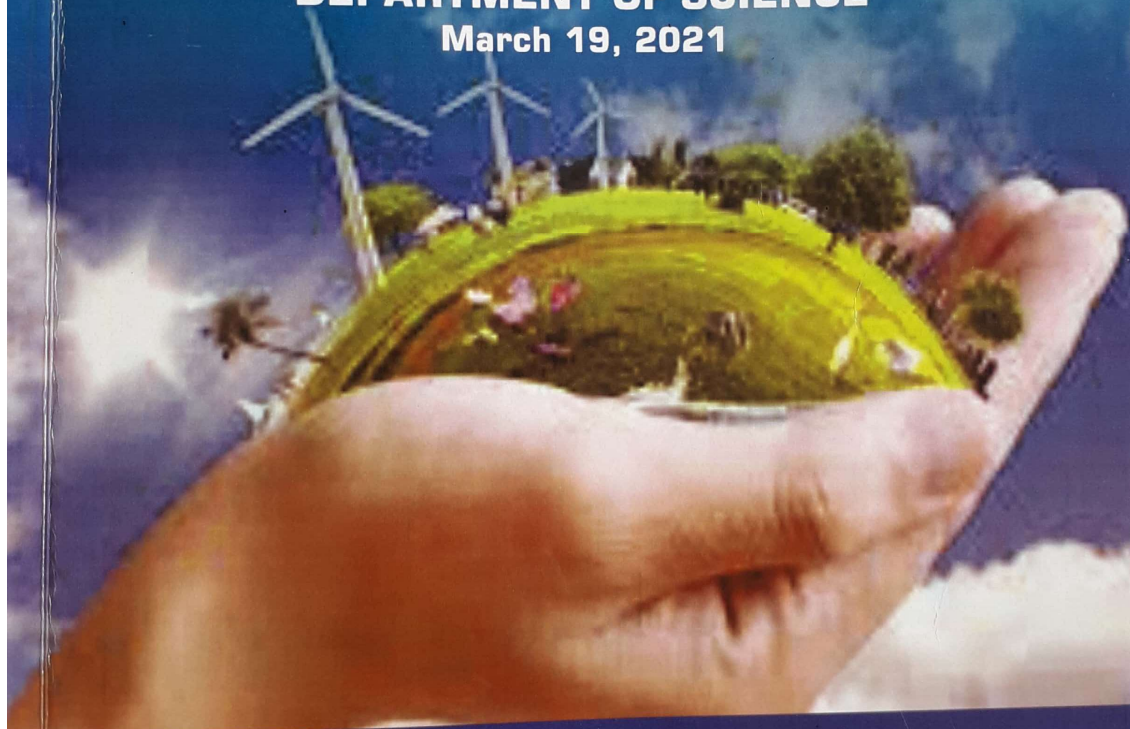




**Proceedings  
of  
DGHE Approved One Day National Science  
Webinar  
on  
New Frontiers in Science & Technology**

**DEPARTMENT OF SCIENCE  
March 19, 2021**



**MARKANDA NATIONAL COLLEGE  
Shahabad Markanda  
NAAC Re-Accredited Institute with Grade B**

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**Markanda National College**

Ladwa Road, HUDA 1, Shahabad Markanda, Kurukshetra, Haryana-136135  
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## Synthesis and Characterization of Polystyrene-anchored Iron (III) Complex

Amit Kumar

Department of Chemistry, Indira Gandhi National College, Ladwa, Kurukshetra

### ABSTRACT

The reaction between polystyrene 3-formylsalicylate and 2-Aminophenol in DMF in the presence of ethyl acetate results in the formation of polystyrene N-(2-hydroxyphenyl)-2'-hydroxybenzylideneimine-3'-carboxylate (**I**). A benzene suspension of **I** reacts with mercaptoacetic acid undergoes cyclization and forms polystyrene N-(2-hydroxyphenyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one,  $\text{PSCH}_2\text{-LH}_2$  (**II**). A DMF suspension of **II** reacts with Fe (III) and forms the corresponding polystyrene-anchored coordination compound,  $[\text{PSCH}_2\text{-LH.FeCl}_2(\text{DMF})_2]$  (**III**). The polystyrene-anchored coordination compound has been characterized on the basis of elemental analyses, spectral (IR, reflectance) studies and magnetic susceptibility measurements. **II** acts as a monobasic bidentate OS donor ligand in this compound. An octahedral structure for **III** are suggested.

**KEYWORDS:** Thiazolidin-4-one, Polystyrene-anchored coordination compounds, magnetically dilute.

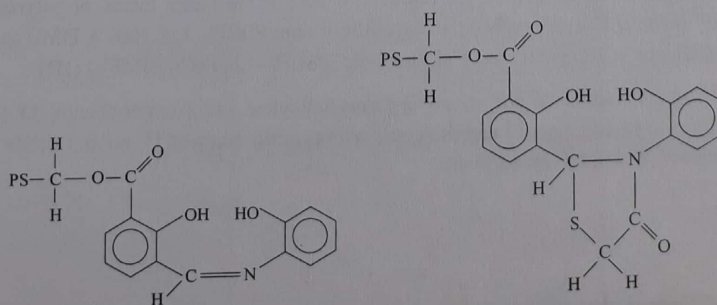
### INTRODUCTION

Among transition metal based polymers, the complexes of polymeric Schiff base and its derivatives are considered as a very important class of coordination compounds which have been extensively studied [1] owing to their wide applications in many biological, clinical, analytical and industrial activities, in addition to their important roles in catalysis and organic synthesis [2].

Thiazolidin-4-ones belong to an important group of heterocyclic compounds with carbonyl group at fourth position [3]. They show broad spectrum of biological activities due to their ready accessibility and diverse chemical reactivity [4].

These facts prompted us to explore the coordination behavior of polystyrene-anchored thiazolidin-4-one (**II**) derived from the Schiff base (**I**) (obtained from the condensation of polystyrene 3-formylsalicylate and 2-Aminophenol) towards Fe(III) ions.

In this manuscript, we describe the syntheses and characterization of polystyrene-anchored thiazolidin-4-one,  $\text{PSCH}_2\text{-LH}_2$  (**II**) and its coordination compound (**III**).



I

II

## EXPERIMENTAL

### MATERIALS

Chloromethylated polystyrene, PSCH<sub>2</sub>-Cl (containing 1.17 mmol of Cl per g of resin and 1% crosslinked with divinylbenzene) [Sigma Chemical Co (USA)]. Iron (III) chloride (anhydrous) [Sarabhai]; 2-Aminophenol [Loba-Chemie (Mumbai)] were used as supplied for the syntheses. Polystyrene 3-formylsalicylate and 3-formylsalicylic acid were synthesized by following the reported procedures [5]. The elemental analyses, IR, reflectance spectral studies and magnetic susceptibility measurements were carried out as described in our previous report [5].

### Synthesis of polystyrene N-(2-hydroxyphenyl)-2'-hydroxybenzylideneimine-3'-carboxylate (I)

Polystyrene 3-formylsalicylate (1.0 g) was allowed to suspend and swell in DMF (100 mL) for 45 min. To this suspension, a DMF solution (60 mL) of 2-Aminophenol (.510 g, 4.68 mmol) and ethyl acetate (100 mL) were added, while stirring magnetically. The mixture was refluxed for 8 h and then cooled to room temperature. The polystyrene-anchored Schiff base, I obtained was suction filtered, washed with DMF and ethyl acetate. It was dried *in vacuo* at room temperature.

### Synthesis of polystyrene N-(2-hydroxyphenyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one, PSCH<sub>2</sub>-LH<sub>2</sub> (II)

Mercaptoacetic acid (0.32 g, 3.51 mmol) was added to the swollen suspension of I (1.0 g) in benzene (100 mL). The mixture was refluxed for 12 h on a water bath and then cooled to room temperature. The solid product was filtered and washed with 10% sodium bicarbonate solution followed by chilled distilled water. The product was dried as mentioned above. IR bands (KBr): 1690 cm<sup>-1</sup> [ $\nu$ (C=O)(thiazolidinone ring)], 1575 cm<sup>-1</sup> [ $\nu$ (C-N)(thiazolidinone ring)], 1530 cm<sup>-1</sup> [ $\nu$ (C-O)(phenolic)] and 830 cm<sup>-1</sup> [ $\nu$ (C-S)(thiazolidinone ring)]

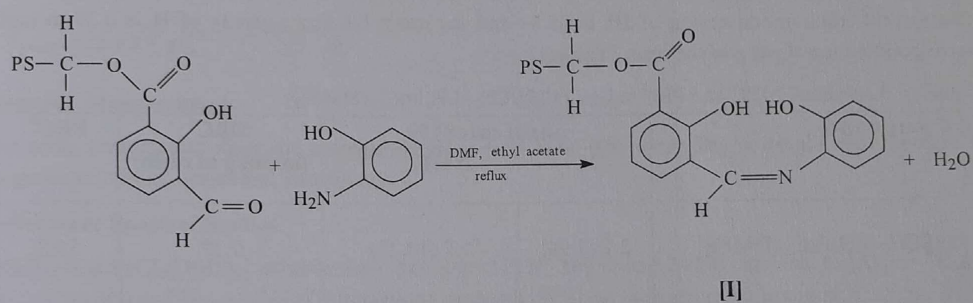
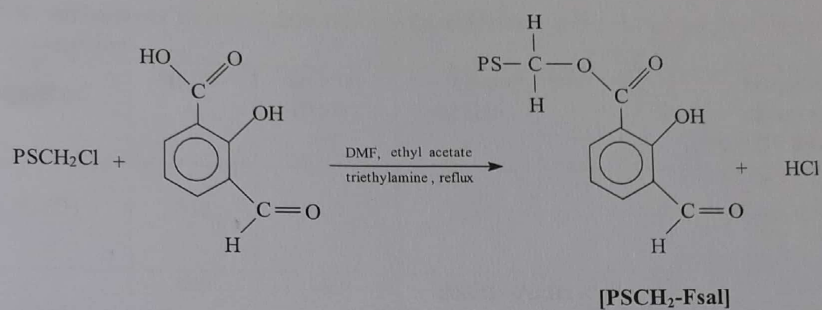
### Syntheses of coordination compounds of II

1.0 g of II was allowed to suspend and swell in DMF (100 mL) for 1 h. A DMF solution of appropriate metal salt (2.34 mmol) was added to the above suspension. The mixture was refluxed on water bath for 8-10 h and the products obtained were suction filtered, washed several times with ethyl acetate and DMF. The products were then dried as mentioned above.

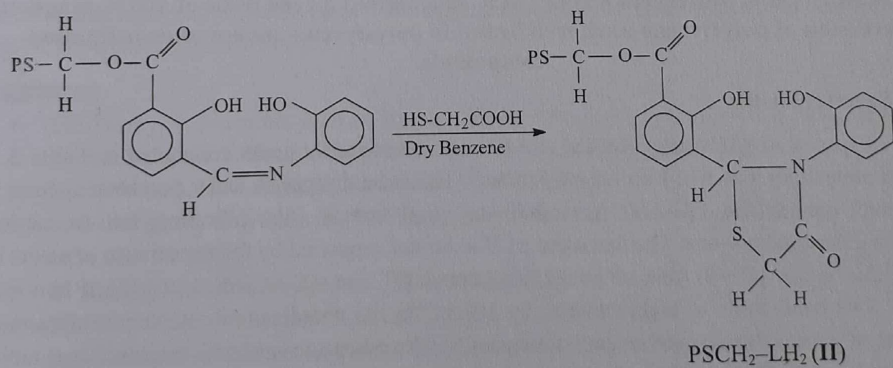
## RESULTS AND DISCUSSION

The reaction between polystyrene 3-formylsalicylate and 2-Aminophenol in DMF in the presence of ethyl acetate results in the formation of polystyrene N-(2-hydroxyphenyl)-2'-hydroxybenzylideneimine-3'-carboxylate (I). The cyclization of I with mercaptoacetic acid in benzene forms polystyrene N-(2-hydroxyphenyl)-C-(3'-carboxy-2'-hydroxyphenyl) thiazolidin-4-one, PSCH<sub>2</sub>-LH<sub>2</sub> (II). A DMF suspension of II reacts with Fe (III) ions in 1:2 molar ratio and forms the [PSCH<sub>2</sub>-LH.FeCl<sub>2</sub>(DMF)<sub>2</sub>] (III).

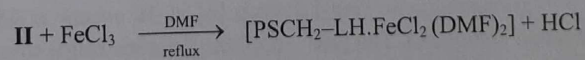
The formations of I (by the reaction of polystyrene 3-formylsalicylate and 2-aminophenol, II (by the cyclization of I with mercaptoacetic acid) and the coordination compounds of II with Fe(III) ions are depicted as per Schemes I, II and III respectively.



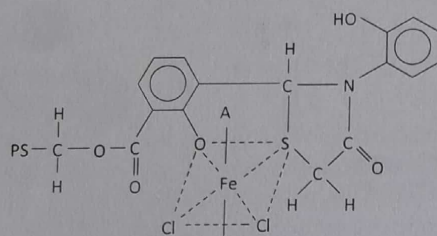
**Scheme-I**



**Scheme-II**



**III**



III (A= DMF)

### Scheme-III

The percent reaction conversion of **III** is 55.84 and the metal binding capacity of **II** is 0.39 m mol of corresponding metal per g of the resin (**Table 1**).

Table 1: Analytical, MBC and PRC values of [PSCH<sub>2</sub>-LH.FeCl<sub>2</sub>(DMF)<sub>2</sub>]

Compound	obsd(calcd)%		MBC <sup>a</sup> (mmol/g of resin)	PRC <sup>b</sup>
	M	DMF		
[PSCH <sub>2</sub> -LH.FeCl <sub>2</sub> (DMF) <sub>2</sub> ]	2.2 (3.94)	5.7 (10.27)	0.39	55.8

$$^a\text{MBC} = [\text{M}\% (\text{observed}) \times 10] / (\text{atomic weight of metal})$$

$$^b\text{PRC} = [\text{M}\% (\text{observed}) \times 100] / \text{M}\% (\text{calculated}) \text{ on the basis of } 100\% \text{ reaction conversion of polystyrene-anchored ligand to polystyrene-anchored coordination compounds.}$$

### Infrared Spectral Studies

The infrared spectra of **I-III** were recorded in KBr and the prominent peaks are shown in **Table 2**. The  $\nu(\text{C}=\text{N})$  (azomethine) stretch of **I** occurs at  $1625 \text{ cm}^{-1}$ . This band disappears and a new band appears in **II** at  $1575 \text{ cm}^{-1}$  due to the  $\nu(\text{C}-\text{N})$  (thiazolidinone ring) stretch [6], indicating the formation of corresponding thiazolidin-4-one. The formation of **II** is further supported by the appearance of a new band at  $830 \text{ cm}^{-1}$  due to the  $\nu(\text{C}-\text{S})$  (thiazolidinone ring) stretch [7]. The  $\nu(\text{C}-\text{O})$   $\phi$  stretch [8] of **II** occurs at  $1530 \text{ cm}^{-1}$ . This band shifts to higher energy by  $10 \text{ cm}^{-1}$  in the coordination compound indicating the involvement of phenolic O atom of either 3-aldehyde-2-hydroxybenzoic acid or 2-aminophenol moieties towards coordination. On the basis of steric grounds, we suggest the non-involvement of phenolic (2-aminophenol moiety) O atom towards coordination. The  $\nu(\text{C}=\text{O})$ (thiazolidinone) stretch [9] of **II** occurs at  $1690 \text{ cm}^{-1}$ . This band remains unchanged in the coordination compound showing its non-involvement in coordination. The  $[\nu(\text{C}-\text{N})$ (thiazolidinone ring)] stretch [6] of **II** occurs at  $1575 \text{ cm}^{-1}$  also remains unchanged in the coordination compound. The  $[\nu(\text{C}-\text{S})$ (thiazolidinone ring)] stretch [7] of **II** occurring at  $830 \text{ cm}^{-1}$  shifts to lower energy by  $30 \text{ cm}^{-1}$  in the coordination compound. DMF shows a band at  $1680 \text{ cm}^{-1}$  due to the  $\nu(\text{C}=\text{O})$  stretch [10]. This band shifts to lower energy by  $25 \text{ cm}^{-1}$  in **III** indicating the involvement of O atom towards coordination [10].



Table 2: IR, reflectance spectral data ( $\text{cm}^{-1}$ ) and magnetic moment of polystyrene-anchored coordination compound

Compound	$\nu(\text{C-S})$	$\nu(\text{C=O})$ (DMF)	$\nu(\text{C-O})$ (phenolic)	$\nu_{\text{max}}$	Magnetic moment <sup>a</sup> (B. M.)
PSCH <sub>2</sub> -LH <sub>2</sub> (II)	830	–	1530	–	Diamagnetic
[PSCH <sub>2</sub> -LH.FeCl <sub>2</sub> (DMF) <sub>2</sub> ]	800	1655	1540	12720, 16950, 24900	5.85

$$^a \mu_{\text{eff.}} = 2.83 (\chi_{\text{M}}^{\text{corr}} \times T)^{1/2} \text{ B. M.}$$

#### Magnetic Measurements

The room temperature magnetic moment of **III** is 5.85 B.M. The value lies in usual range reported for magnetically dilute octahedral compounds.

#### Reflectance Spectral Studies

[PSCH<sub>2</sub>-LH.FeCl<sub>2</sub>(DMF)<sub>2</sub>] exhibits three bands at 12720, 16950 and 24900  $\text{cm}^{-1}$  due to  $^6\text{A}_{1g} \rightarrow ^4\text{T}_{1g}(\text{G})$ ,  $^6\text{A}_{1g} \rightarrow ^4\text{T}_{2g}(\text{G})$  and  $^6\text{A}_{1g} \rightarrow ^4\text{A}_{1g}(\text{G})$  transitions, respectively, in an octahedral environment.

#### CONCLUSION

The elemental analyses, IR, reflectance and magnetic susceptibility measurements an octahedral structure for [PSCH<sub>2</sub>-LH.FeCl<sub>2</sub>(DMF)<sub>2</sub>] (**III**).

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