

## AC impedance, FTIR studies of Biopolymer Electrolyte Potato starch: NH<sub>4</sub>SCN

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### Abstract

Biopolymer - an environmentally friendly and harmless polymeric material continues to show good growth potential. Several factors such as soaring oil prices, worldwide interest in renewable resources, growing concern regarding greenhouse gas emissions and a new emphasis on waste management have created renewed interest in biopolymers such as corn starch, Potato starch and arrow root starch etc. Focusing on these interests, a new solid biopolymer electrolytes based on the biopolymer Potato starch doped with different molar concentrations of Ammonium thiocyanate (NH<sub>4</sub>SCN) have been prepared by Solution Casting Technique. The complex formation between the polymers and the salt has been confirmed by FTIR analysis. From the conduction graph the Polymer electrolyte having 40 mol% Potato starch doped with 60 mol% NH<sub>4</sub>SCN has low bulk resistance and high ionic conductivity of  $3.93 \times 10^{-4}$  S/cm at 303 K. Moisture content of the prepared biopolymer electrolytes have been studied by using moisture analysis.

**Keywords:** FTIR, AC Impedance, Moisture studies

### 1. Introduction

Biopolymers play a significant role both in the natural world and in modern industrial economies. Biopolymers such as Corn starch, Potato starch, Arrow root starch etc have a variety of environmental benefits rather than the synthetic polymers such as PMMA, PVDF etc [1]. Starch attracts scientists due to its rich variety and abundance in nature [2]. Also it is one of the most popular renewable and biodegradable polymers. Tuhina Tiwari et al reported that Polymer electrolyte based on Potato starch has the ionic conductivity  $1.3 \times 10^{-4}$  S cm<sup>-1</sup> [3]. In the Present work, Potato starch doped with different molar concentrations of Ammonium thiocyanate polymer electrolytes have been prepared and characterized by various experimental techniques such as FTIR Analysis, Ac impedance spectroscopy and Moisture analysis.

### 2. Experimental Procedure

#### 2.1 Sample Preparation

In the present study, Potato starch with molecular weight= 162.14 g/mol (LOBA CHEMIE) and NH<sub>4</sub>SCN with molecular weight 76.122 g/mol (REACHEM) are used as starting material with Water as solvent. Different molar ratios of Potato starch: NH<sub>4</sub>SCN as (100:0), (50:50), (40:60) and (30:70) have been prepared by Solution Casting Technique. In this technique, appropriate weight of Potato starch and NH<sub>4</sub>SCN have been dissolved individually in Water and these solutions have been mixed together and stirred well by using magnetic stirrer to obtain a homogeneous mixture. The obtained mixture is casted in Propylene petridish and is subjected to vacuum dried at 40°C for 1 day. Mechanically strong, transparent and flexible films have been obtained.

#### 2.2 Characterization

##### Vibrational study

FTIR spectra have been recorded for the polymer electrolyte films using a SHIMADZU- IR Affinity-1 Spectrometer in the

range of 400cm<sup>-1</sup> to 4000cm<sup>-1</sup> at room temperature.

##### AC impedance study

Conductivity measurements have been carried out by using a HIOKI – 3532 LCZ meter in the frequency range of 42 Hz – 1MHz over the temperature range of 303K – 323K.

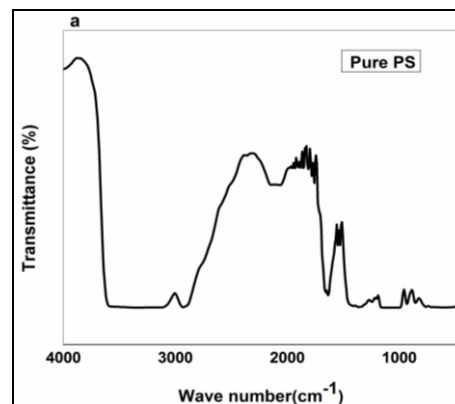
##### Moisture study

An accurate measurement of moisture contents in the polymer electrolytes has been done using MOC63U Shimadzu moisture analyzer.

### 3. Results and Discussion

#### 3.1 Fourier Transform Infrared analysis

The purpose of measurement of Fourier transform infrared spectroscopy (FTIR) of pure Potato starch and Potato starch-doped ammonium thiocyanate (NH<sub>4</sub>SCN) polymer electrolytes is to confirm the complex formation of the ammonium salt with polymer. Figure 1 represents the FTIR spectra of 40Potato starch: 60 NH<sub>4</sub>SCN Polymer electrolyte. The vibrational frequencies observed in the FTIR spectra of Potato starch – NH<sub>4</sub>SCN polymer electrolytes are given in Table 1.



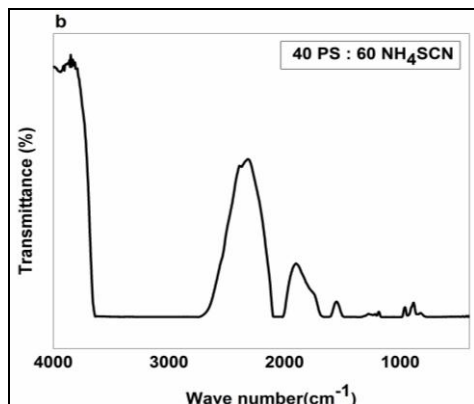


Fig 1: FTIR spectra of (a) Pure PS and (b) 40 PS: 60 NH<sub>4</sub>SCN Polymer electrolyte

Table 1: Absorption bands for PS & 40 PS: 60 NH<sub>4</sub>SCN polymer electrolytes

Vibrational peaks of the biopolymer electrolytes (cm <sup>-1</sup> )		Assignments
Pure PS (mol %)	40 PS:60NH <sub>4</sub> SCN (mol %)	
3579	3576	O-H (s)
2931	2958	C-H (s)
1635	1658	C = O (s)
1411	1427	O-H (b) & C-H (b) of CH <sub>2</sub>
1056	1068	C – O(s) of C – O – C

The hydroxyl band in the spectrum of pure Potato starch electrolyte is located at 3579 cm<sup>-1</sup>. The strong and wide absorption in hydroxyl band region of Pure Potato starch film suggests that there are plenty of hydroxyl groups in starch. This absorption peak has got shifted to lower wave numbers 3576 cm<sup>-1</sup> in 60 mol% NH<sub>4</sub>SCN doped polymer electrolyte. It suggests that there is interaction between NH<sub>4</sub>SCN and Potato starch. The Buraidah et al reported that in Polymer Chitosan – ammonium salt complexes, the conducting charge species is

Table 2: Ionic conductivity values of Potato starch - NH<sub>4</sub>SCN polymer electrolytes at different temperatures

Composition PS: NH <sub>4</sub> SCN (mol %)	Ionic conductivity(σ <sub>dc</sub> ) for different compositions of Potato starch:NH <sub>4</sub> SCN (S cm <sup>-1</sup> )		
	303K	313K	323K
100:0	5.98×10 <sup>-10</sup>	6.10×10 <sup>-10</sup>	1.46×10 <sup>-9</sup>
50:50	7.47×10 <sup>-7</sup>	8.89×10 <sup>-6</sup>	1.34×10 <sup>-6</sup>
40:60	3.93×10 <sup>-4</sup>	4.24×10 <sup>-4</sup>	5.23×10 <sup>-4</sup>
30:70	1.57×10 <sup>-4</sup>	2.31×10 <sup>-4</sup>	2.96×10 <sup>-4</sup>

Conductance spectra of Pure Potato starch has frequency independent plateau region corresponding to dc conductivity and the high frequency dispersion region corresponding to bulk relaxation phenomenon. At the same time the conductance spectra of complexes has low frequency region due to the space charge polarization at the blocking electrodes and plateau region corresponding to dc conductivity [6]. From the Table 2 it has been observed that the dc conductivity raises with increase of temperature suggesting that free volume around the polymer chain causes the mobility of ions and polymer segments.

Proton ion from ammonium ion (NH<sub>4</sub><sup>+</sup>) [5]. The absorption peak of pure Potato starch at 2931 cm<sup>-1</sup> assigned to C-H stretching mode of starch is shifted to wave number at 2958 cm<sup>-1</sup> in the salt – doped system [4]. The absorption peak of pure Potato starch at 1635 cm<sup>-1</sup> and 1056 cm<sup>-1</sup> are assigned to C = O stretching and C – O (s) are shifted to wave number at 1658 cm<sup>-1</sup> and 1068 cm<sup>-1</sup> in the salt – doped system. The vibrational peak at 1411 cm<sup>-1</sup> assigned to O–H (b) and C–H (b) of CH<sub>2</sub> of pure Potato starch are shifted to 1427 cm<sup>-1</sup> in 40:60 compositions of Potato starch: NH<sub>4</sub>SCN polymer electrolyte respectively.

### 3.2 Conductivity Analysis

#### Conductance spectra

The conductance spectra describe the frequency dependence of the conductivity. The typical log σ versus log ω relation for 100 PS, 50 PS: 50 NH<sub>4</sub>SCN, 40 PS: 60 NH<sub>4</sub>SCN, and 30 PS: 70NH<sub>4</sub>SCN at 303 K have been shown in the Figure 2 (a - d) respectively.

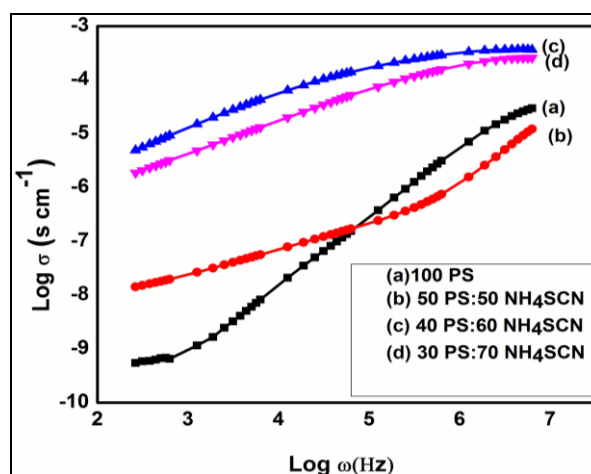


Fig 2: Conductance spectra

### 3.3 Moisture Analysis

An accurate measurement of moisture contents in the polymer electrolytes has been done using MOC63U, Shimadzu moisture analyzer. The percentage of evaporated moisture mass versus the mass before drying process is given by

$$\text{Moisture content (\%)} = \frac{M_{\text{wet}} - M_{\text{dry}}}{M_{\text{wet}}} \times 100$$

Where M<sub>w</sub> is the mass of undried material when measurement starts and M<sub>d</sub> is the mass of dried material when measurements ends.

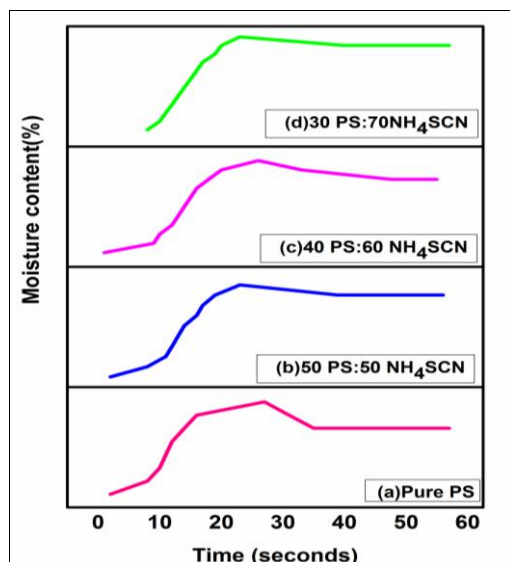


Fig 3: Moisture Spectra

The plot shows percentage of amount of change in moisture content as a function of time. The change in moisture content increases and reaches stable state. Weight loss (%) and Time of evaporation for Potato starch:  $\text{NH}_4\text{SCN}$  Polymer electrolytes are given in Table 3

**Table 3:** Weight Loss (%) and Time of evaporation for Potato starch:  $\text{NH}_4\text{SCN}$  Polymer Electrolytes

Composition of PS: $\text{NH}_4\text{SCN}$ (mol %)	Weight Loss (%)	Time (s)
100:0	0.37	22.17
50:50	2.42	17.17
40:60	5.66	8.81
30:70	2.65	18.59

The weight loss percentage of the prepared samples increases with increase of salt concentration. 40 Potato starch: 60  $\text{NH}_4\text{SCN}$  electrolyte has maximum weight loss with minimum time interval of evaporation among the prepared electrolytes.

#### 4. Conclusion

Potato starch-based polymer electrolytes with different concentrations of ammonium thiocyanate have been prepared using the solution casting technique.

- FTIR studies reveal the complex formation between the salt  $\text{NH}_4\text{SCN}$  and polymer matrix Potato starch.
- The highest ionic conductivity has been found to be  $3.93 \times 10^{-4} \text{ Scm}^{-1}$  for 40Potato starch: 60 $\text{NH}_4\text{SCN}$  polymer electrolyte from the conductance spectra.
- 40 Potato starch: 60  $\text{NH}_4\text{SCN}$  electrolyte has maximum weight loss with minimum time interval of evaporation among the prepared electrolytes.

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