Real-time monitoring of room-temperature ionic liquid purity through optical diode-based sensing

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Abstract

Room-temperature ionic liquids (RTILs) are promising for use in many industries due to their unique properties, including wide electrochemical windows, low vapor pressures, high ionic conductivities, and chemical and thermal stability. All of these properties require high RTIL purity, and achieving this high purity is a major driver of RTIL manufacturing costs. Continuous flow processes to synthesize highly pure RTILs at a reduced cost have been developed, but due to exothermic synthesis reactions and temperature dependent reaction rates, these processes require realtime control. An ultraviolet LED based optical sensor has been designed to measure RTIL purity at millisecond sampling rates using a liquid flow cell. The sensor is demonstrated by measuring 1-methylimidazole (MIM) concentration in 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ([EMIM][TFSI]). Sensor results are compared against spectroscopic measurements with good agreement.

Keywords: Room temperature ionic liquid (RTIL), Optical sensor, Impurity detection

1. Introduction

Room temperature ionic liquids (RTILs) are a class of chemicals which are liquids at or below room temperature and are composed entirely of ion pairs. These RTILs have desirable properties as green solvents due to their low vapor pressure [1–4]. RTILs are also stable across a wide range of temperatures and possess a wide electrochemical window, making RTILs a promising class of electrolytes [5–7]. The properties of RTILs are highly dependent on purity. For example, the electrochemical window of [EMIM][TFSI] has been shown to be dependent on the water content of the sample [7, 8]. Residual precursors and byproducts of RTIL synthesis can lead to electrode corrosion and other

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performance degradation mechanisms. For example, synthesis of the [EMIM]-cation typically begins with MIM, whose redox stability limit is within the electrochemical window of [EMIM][TFSI]. Currently, most RTILs are produced through batch processes. After the synthesis reaction is complete, the product must be separated from impurities, adding to the cost of RTILs. Due to the low vapor pressure of ionic liquids, purification through a distillation process is not feasible, and considerable effort is spent in purifying ionic liquids through filtration or recrystallization processes [9, 10].

Continuous flow synthesis processes have recently been developed that have the potential to significantly reduce costs – making more industrial RTIL applications feasible [9, 11]. In a continuous flow process, it is desirable to monitor the purity of the finished product in real time as the product exits the production line to ensure product quality and process control. Synthesis of quaternary amine cations pose particular challenges due to the exothermic reaction and requirement that the precursors’ stoichiometry be precisely controlled. Any deviation from a 1:1 stoichiometry of tertiary amine and quaternizing agent will result in an excess of one reactant that may be difficult to remove from the product RTIL. By monitoring the purity of the finished product, it is possible to determine if the synthesis process is proceeding within normal operating conditions [9, 11]. Currently, methods for determining the purity of RTILs such as ion chromatography tend to be expensive and ex-situ [12, 13]. An in-situ sensor which is capable of determining the purity of the RTIL in real time would allow the production process to be self monitoring.

The ultraviolet absorption spectrum provides a unique identifier for ionic liquids and their chemical precursors. Previously, it was shown that by using UV spectroscopy, it was possible to detect the presence of impurities in ionic liquids by analyzing the absorption spectrum of each constituent [14]. The aim of the work reported here is to apply these findings to the problem of sensing impurities in real time. The time response and non-intrusive nature of optical sensors make them ideal for monitoring chemical processes. For example, optical sensors are commonly used to measure the presence of analytes in samples [15–17]. Although spectroscopic methods have been used before to characterize RTILs [18], to the best of the authors’ knowledge this sensor is the first spectroscopic device designed for process control in the production of RTILs.

1.1. Absorbance in liquid samples

The LED sensor operates on the principle of optical absorption. As described by the Beer-Lambert law, at certain optical wavelengths, light is absorbed as it passes through a sample:

$$-\ln\left(\frac{I_{\lambda}}{I_{0,\lambda}}\right) = \sigma_{\lambda}X_i nL \quad (1)$$

where I_{λ} is the intensity of light at wavelength λ exiting the sample, $I_{0,\lambda}$ is the intensity of light at wavelength λ incident on the sample, σ_{λ} is the molar absorption cross-section or molar absorptivity (m^2/mol), X_i is the mole fraction

49 of species i , n is the total molar concentration of the sample, and L is the path-
 50 length the light travels through the sample. The absorption cross-section, σ_λ , is
 51 a wavelength-dependent material property of the sample that is measured in an
 52 optical cell of known path length and material concentration using a spectrom-
 53 eter. Figure 1 shows the measured UV absorption spectra of [EMIM][TFSI] and
 54 MIM at ambient conditions. From these spectra, it can be seen that MIM has an
 55 absorption peak near 275 nm, while [EMIM][TFSI] has an absorption peak near
 56 265 nm. Between 275 nm and 300 nm, the absorption cross-section of MIM is
 57 greater than that of [EMIM][TFSI]. At wavelengths longer than 340 nm, neither
 58 MIM nor [EMIM][TFSI] absorb light. The sensor described in this work utilizes
 these spectral properties to determine the presence of MIM in [EMIM][TFSI].

Figure 1: Absorbance of [EMIM][TFSI] and MIM. The full-width half-maximum range of the
 295 nm LED channel is shown as the shaded area on the left. The shaded area on the right
 is the full-width half-maximum of the 365 nm channel. Data were previously collected and
 presented as described in [14].

59

60 2. Experimental methods

61 Due to the differences in spectral properties between MIM and [EMIM][TFSI]
 62 seen in Figure 1, the presence of MIM may be detected in a sample by changes in
 63 absorption at wavelengths between 275 nm to 300 nm. In order to measure the
 64 differences in absorption between [EMIM][TFSI] and MIM, a custom LED array
 65 with wavelengths selected to measure the spectral features of [EMIM][TFSI] and

66 MIM was obtained from Sensor Electronic Technologies Inc. A diagram of the
67 array may be seen in Figure 2.

68 *2.1. Sensor construction and testing*

69 The LED array contained 9 separate diodes: 4 diodes which emitted at a
70 peak wavelength of 295 nm (9.7 nm full-width half-maximum), 4 diodes which
71 emitted at a peak wavelength of 320 nm (10.4 nm full-width half-maximum),
72 and as a single 365 nm diode (18.1 nm full-width half-maximum). The LED
73 array was housed in a 12 pin TO-8 package. This package was soldered to a
74 custom printed circuit board which was designed to fit into a standard 1 in.
75 optical mount. The LED was driven by a 0-12 V logic circuit with appropriate
76 current limiting resistors to protect the diodes in the array. The 295 nm diodes
77 were used to detect increased absorbance due to the presence of MIM. While
78 a larger difference in absorbance is seen at about 270 nm, inexpensive diodes
79 at this wavelength are not yet commercially available. The 365 nm diode has
80 been selected to provide a reference for the transmission through the RTIL. The
81 320 nm diodes are used for detecting a second impurity which is specific to the
82 proprietary synthesis process developed by Boulder Ionics.

83 The RTIL flowed through a 2.5 mm path length flow cell (FIA Labs SMA-
84 Z-2.5 #79048) constructed from PEEK and fitted with a pair of fused silica
85 windows to allow transmission of ultraviolet light. Light was focused through
86 the cell's windows by means of a series of free space optics mounted on ThorLab's
87 30 mm cage mount components, as shown in Figure 2. The light transmitted
88 through the RTIL was measured by a ThorLabs PDA10A amplified photode-
89 tector (measurement detector). An uncoated UV fused silica window directed
90 a portion of the LED light to a second PDA10A to monitor the output of the
91 LED (reference detector). The purpose of the reference detector was to measure
92 and correct for variation in the output intensity of the LED. Detector output
93 signals were filtered with 50 Hz RC low pass filters ($R=330\ \Omega$, $C=10\ \mu\text{F}$) and
94 read by a National Instruments PCI-MIO-16E-1 data acquisition card.

95 [EMIM][TFSI] (trade name Iolyte P1) was obtained from Boulder Ionics
96 Corp. Total halide content was less than 100 ppm and water content was less
97 than 20 ppm. The [EMIM][TFSI] was used without further purification. MIM
98 was also obtained from Boulder Ionics. All samples were stored and prepared
99 in a dry nitrogen environment. Samples of [EMIM][TFSI] with known MIM
100 concentrations were created in a controlled atmosphere glovebox with low oxy-
101 gen and low humidity. Quantities of MIM were weighed using a digital balance
102 and added to samples of pure [EMIM][TFSI]. Separate 2 mL samples were pre-
103 pared in syringes and injected through the flow cell while sensor readings were
104 recorded. Each sample was discarded after passing through the sensor to pre-
105 vent contamination. Only steady-state readings were retained to avoid erroneous
106 readings due to mixing of samples in the flow cell.

107 *2.2. Determination of MIM concentration*

Accurate MIM concentration measurements require correcting for background
noise, fluctuation in LED output, and for changes in optical transmission through

Figure 2: The optical layout of the sensor. Two detectors are used to measure both the output of the LED (bottom) and the light transmitted through the RTIL (right). The beam is split using an uncoated UV fused silica window to allow the reference detector (bottom) to measure LED output. The RTIL flows through an optical flow cell manufactured by Flow Injection Labs. Free space optics are used to focus the light through the cell and onto the detector. Not to scale.

the cell. Background subtraction removes the small DC offset due to ambient light striking the detectors. This is accomplished by subtracting the voltage reading from each detector when all LED wavelengths are off. Fluctuations in

the intensity of the LED are removed by taking the ratio of the measurement detector signal, I_M , over the reference detector signal, I_R : I_M/I_R . Taking this ratio in real-time corrects for transient fluctuations in LED output as well as intensity drift over time. These corrections can be represented in the Beer-Lambert law (Equation 1) as:

$$-\ln \left(\frac{I_M^{295} I_{0,R}^{295}}{I_{0,M}^{295} I_R^{295}} \right) = \sigma^{295} X_{MIM} n L \quad (2)$$

108 where I_0 denotes the measurement in a pure [EMIM][TFSI] sample, and the
 109 superscript denotes the wavelength of the LED that was active while the voltage
 110 reading was taken. Each value of I was calculated by averaging the voltage
 111 reading while that wavelength was on (as shown by the shading in Figure 3).

Figure 3: Raw voltage readings from the measurement detector. Each wavelength of the LED array is seen as a discrete pulse in the voltage reading. I values are taken by averaging the ‘top hat’ of each voltage pulse. The standard deviation of points in the ‘top hat’ is also recorded to allow uncertainty in the measured concentration of MIM to be calculated.

Monitoring the light transmitted through the RTIL at 365 nm corrects for beam steering, optical scattering from small bubbles in the sample, and variability in the overall absorbance of the RTIL (as has been reported even for high purity samples [10, 19, 20]). The light lost due to these factors is modeled with a modified version of the Beer-Lambert law:

$$-\ln \left(\frac{I_M^{295} I_{0,R}^{295}}{I_{0,M}^{295} I_R^{295}} \right) = \sigma^{295} X_{MIM} n L + \tau \quad (3)$$

112 where τ is attenuation not due to the presence of MIM. Taking this ratio for

the 365 nm signal results in a direct measurement of signal attenuation not due to absorption. This is possible because the absorption cross-section of MIM at 365 nm (σ^{365}) is zero.

$$-\ln \left(\frac{I_M^{365} I_{0,R}^{365}}{I_{0,M}^{365} I_R^{365}} \right) = \sigma^{365} X_{\text{MIM}} n L + \tau = \tau \quad (4)$$

In order to correct for this extra attenuation, the signal ratio for the 365 nm channel is subtracted from the 295 nm signal ratio. Using the properties of logarithms, the 365 nm channel may be subtracted from the 295 nm channel to eliminate the τ term and produce a final corrected ‘absorbance ratio’:

$$-\ln \left(\frac{I_M^{295} I_R^{365} I_{0,M}^{365} I_{0,R}^{295}}{I_M^{365} I_R^{295} I_{0,M}^{295} I_{0,R}^{365}} \right) = (\sigma^{295} X_{\text{MIM}} n L + \tau) - \tau \quad (5)$$

$$= \sigma^{295} X_{\text{MIM}} n L \quad (6)$$

Over a small range of MIM concentrations, n may be assumed to be constant. Thus the corrected absorbance ratio provides a direct measure of the MIM mole fraction in the RTIL if the flow cell path length, L , and the MIM absorption cross-section, σ^{295} , are known.

3. Results

Figure 4 shows the two color sensor’s response to MIM in [EMIM][TSFI]. As predicted by the results shown in Figure 1, the absorbance ratio (Equation 6) increases with increasing MIM content due to increased absorption of the 295 nm light. The absorbance ratio has also been predicted from the spectra shown in Figure 1 by calculating an average absorbance across the spectral range of the 295 nm and 365 nm channels of the LED array. The average absorbance was calculated by performing a numerical integration of the molar absorptivity of both MIM and [EMIM][TFPI] over the spectral range of the 295 nm and 365 nm channels of the LED. This integrated average absorption cross-section was then multiplied by the MIM mole fraction and cell path length for comparison with the sensor absorbance ratio (Equation 1). This comparison is complicated by the fact that the intensity of the LEDs was not a constant with respect to wavelength. The integration of the molar absorptivity was therefore weighted according to the output of the LED. For the integration, the output of the LED was assumed to be spectrally Gaussian and each peak was integrated from two standard deviations below the peak output wavelength to two standard deviations above peak output. Thus the spectral average of the absorption cross-section becomes:

$$\bar{\sigma} = \left(\frac{C}{\lambda_f - \lambda_0} \right) \int_{\lambda_0}^{\lambda_f} \Omega(\lambda) \sigma(\lambda) d\lambda \quad (7)$$

Figure 4: Sensor results for MIM in [EMIM][TFSI]. Error bars are calculated through a propagation of uncertainties based on the standard deviation of voltage readings for each wavelength. Additionally, the absorbance ratio has been calculated from the spectra in Figure 1 (solid line). The absorbance ratio is defined in Equation 6.

where $\bar{\sigma}$ is the average molar absorptivity over the wavelength range of the LED, C is a constant which takes into account the difference in the LED intensity and detector responsivity for each LED channel (For the 295 nm channel $C = 0.25$, for 365 nm $C = 8.5$), Ω is the Gaussian weighting function to estimate the spectral dependence of the LED's output, λ_0 is the wavelength two standard deviations shorter than the peak output wavelength of the LED, and λ_f is the wavelength two standard deviations longer than the peak output wavelength.

The limit of detection (LOD) of the sensor was calculated from the standard deviation of 82 sensor readings of pure and low MIM concentration (5 w%) samples, as described by Looock and Wentzell [21]. The 99% confidence LOD was determined to be 4 molar percent MIM. The sensor described in this article is less sensitive than methods such as gas chromatography-mass spectrometry which is used to measure ppm levels of MIM in [EMIM][TFSI]. However, the 4 molar percent LOD is sufficient to determine if the continuous flow synthesis process is operating outside of normal parameters.

The uncertainty of each measurement in Figure 4 is calculated through a

141 propagation of uncertainties based on the standard deviation of all the voltage
142 readings used in the measurement. By reducing the noise further through the
143 use of a lock-in amplifier or another filtering scheme, the LOD of the sensor
144 could be reduced.

145 4. Conclusions

146 A novel LED-based optical sensor for monitoring the purity of RTIL in real
147 time has been developed. The sensor was constructed using commercially avail-
148 able LEDs and photodiode detectors. These relatively inexpensive components
149 allow the sensor to be constructed at a cost far less than even a simple laboratory
150 spectrometer and light source. This sensor allows a continuous flow production
151 process to be monitored automatically, which represents an important step to-
152 wards providing affordable RTILs for a variety of applications. This sensor has
153 been shown to detect MIM in [EMIM][TFSI] with a detection limit of 4 molar
154 percent.

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