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Crosslinked Poly(1-butyl-3-vinylimidazolium bromide): A Super Efficient Receptor for Removal and Storage of Iodine from Solution and Vapour Phases

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Supplementary Information

Materials:

1-Vinyl imidazole (>99% purity), bromobutane (99% purity), acetonitrile (anhydrous, 99.8%), were obtained from Sigma-Aldrich and used without further purification. The crosslinking agent N,N'-methylenebis(acrylamide) (>98%) was obtained from Merck and used as received. The free radical initiator 4,4′-Azobis(4-cyanovaleric acid) (>98%) was obtained from Sigma-Aldrich and used after re-crystallization from dry methanol. Dry methanol was obtained from S D Fine, India and stored under activated molecular sieves. Potassium iodide (AR, S D Fine, India), Iodine crystals (ExcelAR, Qualigens, India), and potassium iodate (AR, Merck) were used as received.

Laser Raman spectrum of the crosslinked poly(1-butyl-3-vinylimidazolium bromide) (PIL):

The Raman spectrum (Figure SI-1) of the synthesised polymer was recorded using Jobin-Yvon (France) HR-800 laser Raman spectrometer using a 514 nm He-Ne laser,1800 g/mm grating, and CCD detector. The Raman spectrum of the PIL (Figure SI-1) showed the presence of the monomer and the crosslinker units. The peaks at 1306 and 1289 cm^{-1} (in-plane symmetric stretching - imidazole ring), 1505 cm^{-1} (the butyl group (symmetric bend)), 1448 cm^{-1} (CH_2(N) bend), and 632.4 cm^{-1} (ring out of plane bend) show the presence of the IL monomer. While, the peaks at 1658 cm^{-1} (carbonyl) and 1108 cm^{-1} (C-C stretch) show the presence of the N,N'-methylenebis(acrylamide) (crosslinker) units.

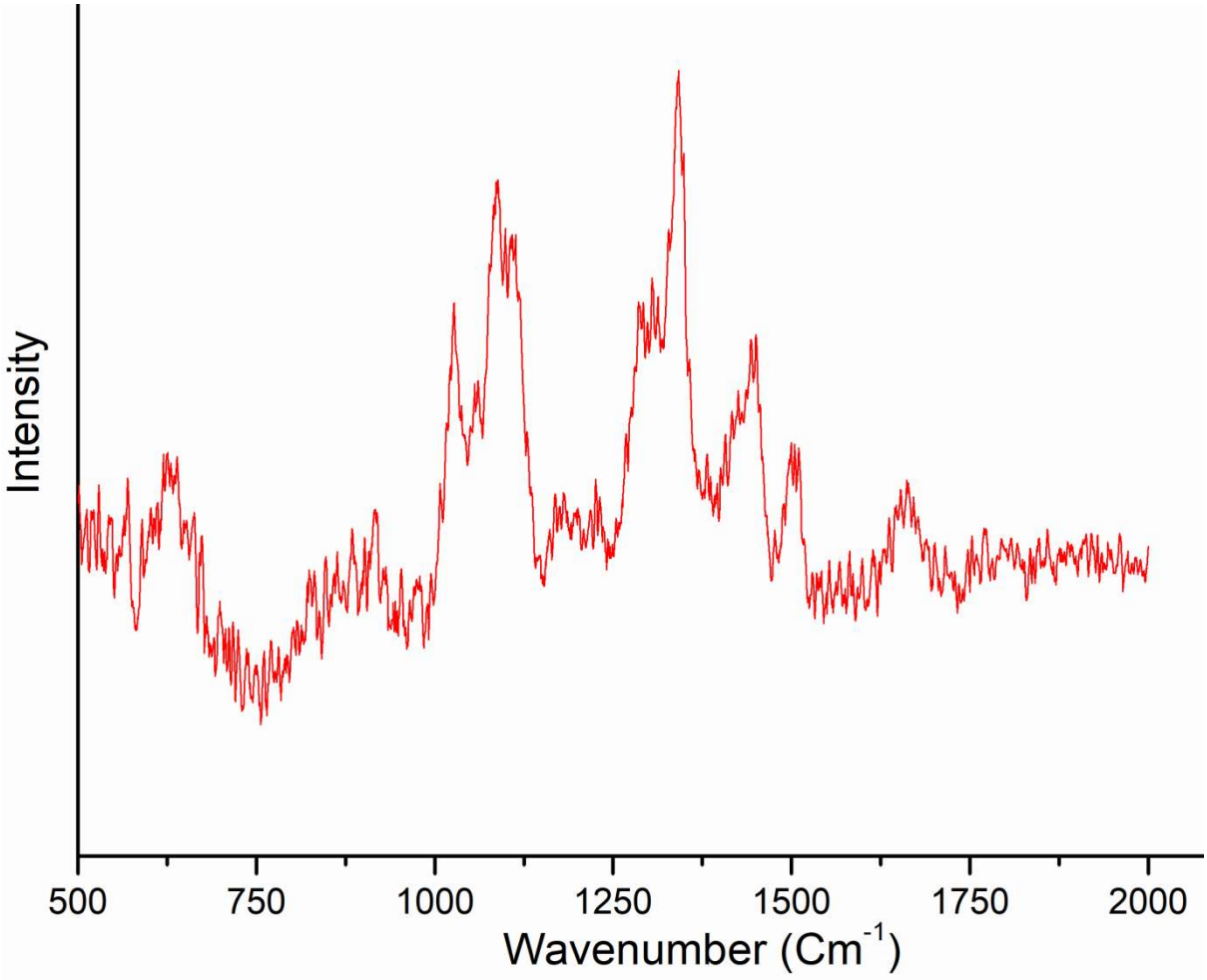

Figure SI-1. Raman spectrum of the crosslinked Poly(1-butyl-3-vinylimidazolium bromide) (PIL)

TG-DTA analysis of the polymer crosslinked poly(1-butyl-3-vinylimidazolium bromide) (PIL):

The synthesised polymer (PIL) was subjected to TG-DTA analysis (Figure SI-2). The analysis was carried out in a horizontal thermal analysis system (Model Seiko 320) in presence of argon with a set heating rate of 10 ^{0}C/min. The first weight loss seen in the curves between 100 to 150 ^{o}C is due to the moisture loss, and the decomposition occurs only above 300 ^{0}C. While the decomposition temperature was same when recorded in presence of static air, the decomposition was seen to be exothermic due to the decomposition reaction with the oxygen (not shown).

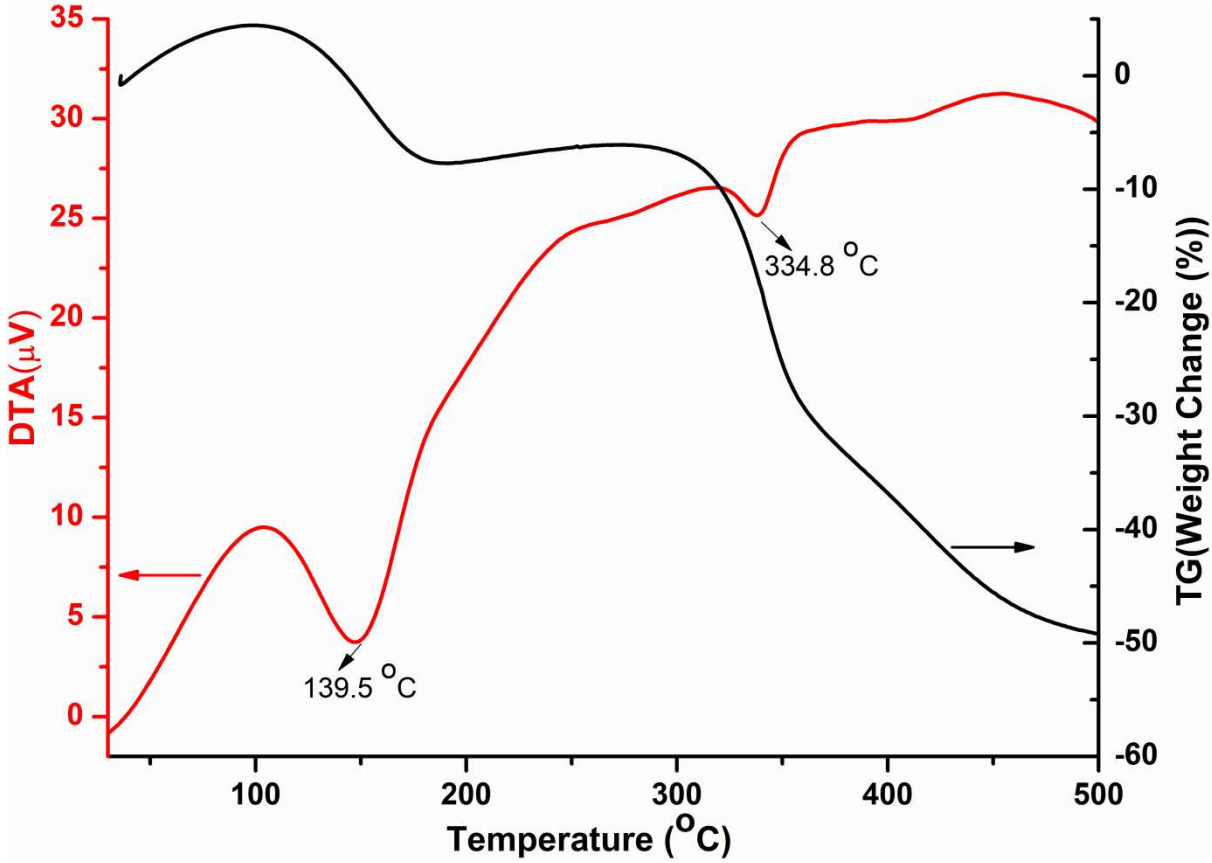

Figure SI-2: TG-DTA curves of the crosslinked poly(1-butyl-3-vinylimidazolium bromide) (PIL)

Spectrophotometric estimation of iodine in aqueous solution:

Iodine present in the aqueous solution was spectrophotometrically estimated by converting the iodine to triiodide. The conversion was done by adding 1.5 g of potassium iodide (as solid) to 25 ml of aqueous iodine solution. The absorbance of the formed triiodide at 352 nm was measured using a spectrophotometer (Shimadzu, UV-2600; 1 cm quartz cell). The concentration was calculated from the calibration curve (Figure SI-3) prepared using iodine solutions of known concentrations.

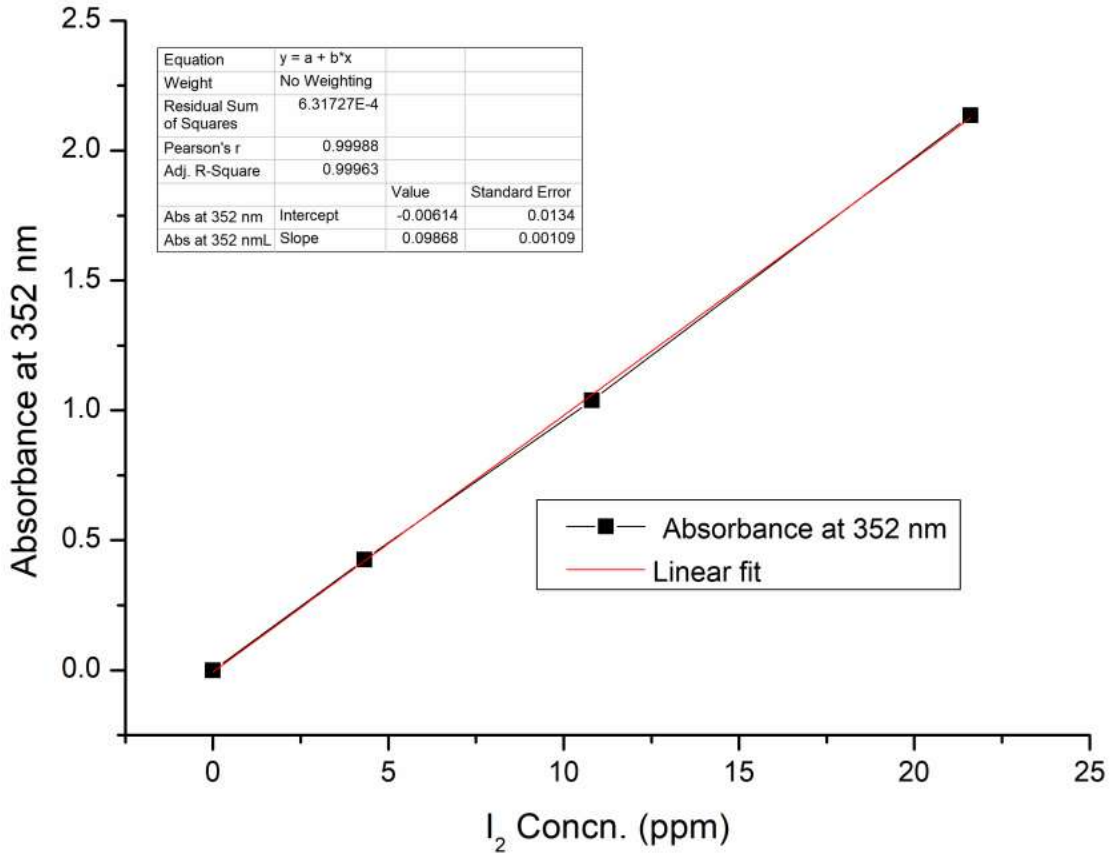

Fig. SI-3. Calibration curve obtained for iodine standards in the triiodide method of iodine estimation

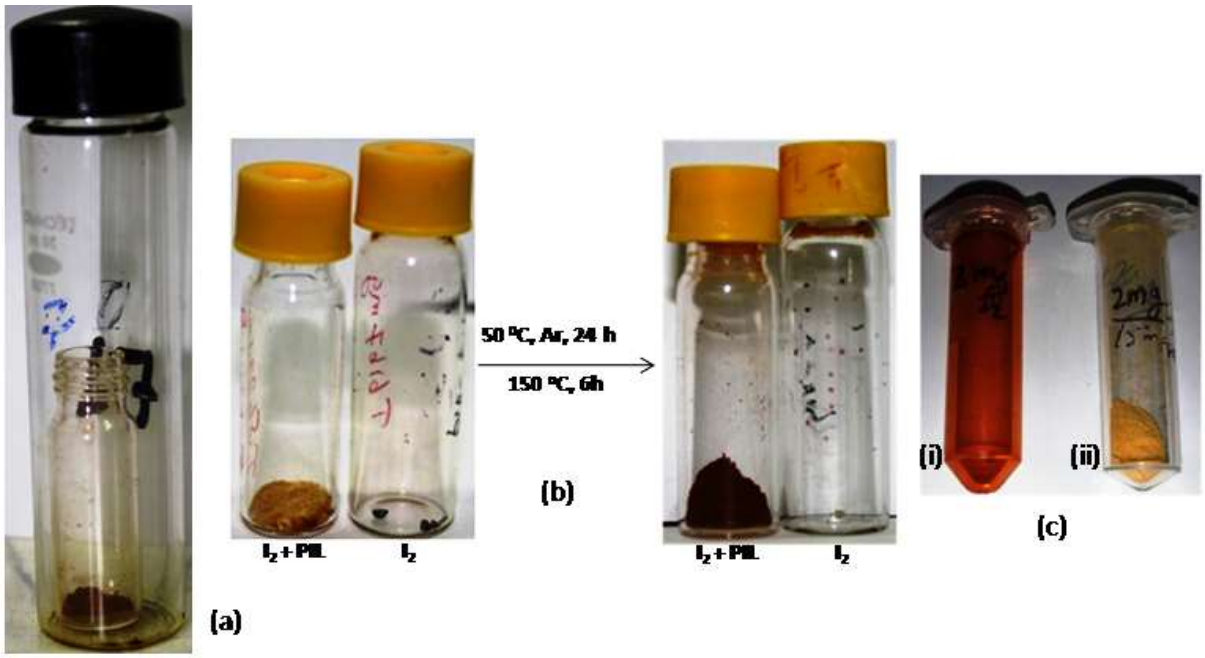

Figure SI-4. (a) Iodine binding by PIL from solid iodine crystals; (b) Binding and retention of iodine vapours by the PIL under heating and argon flow (c) Iodine crystals in (i) polypropylene (PP) vial, (ii) in PP vial containing PIL, both kept at room temperature

Figure SI-4 shows the strong binding of iodine vapours by the PIL under heating and continuous argon flow (Fig. 3a, 3b). Figure 3c shows a polypropylene vial with iodine crystals (left) and a vial containing PIL and iodine crystals (right), which were closed and kept at room temperature. The pictures clearly show that the PIL had bound and retained the iodine effectively.