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Name: NOORAZMI BIN HASSAN

Title : PREPARATION AND CHARACTERIZATION OF NaI-Na₃PO₄ SODIUM ION

CONDUCTING SOLID ELECTROLYTE WITH PLLTMEDA AS AN ADDITIVE

FOR SODIUM BATTERIES

Supervisor: PROF. DR. HJH. AZIZAH HANOM AHMAD (MS)

DR. AZLIN SANUSI (CS)

New binary inorganic salt sodium iodide (NaI)-sodium phosphate (Na₃PO₄) prepared by mechanical milling for 3 hours and low sintering temperature method at 50 °C exhibits maximum ionic conductivity of (1.02±0.19) × 10₋₄ S cm₋₁ at room temperature for the composition 0.50 NaI: 0.50 Na₃PO₄. The increase in conductivity is due to the increase in number of mobile Na+ charge carriers through the conducting pathway provided by tetrahedral structures of Na₃PO₄. The presence of P-O at wavenumber 580 cm₋₁ and PO_{4 3-} bands at wavenumber 1012 cm₋₁ respectively were detected by the infrared technique. Fourier transform infrared spectroscopy had been shifted indicating changes in polyhedral structure which in turn led to the formation of conducting channel by corner sharing or through edges. The spectra also implies that chelation of iodide anion gave rise to high mobility and elevations of the charge carriers to traverse along the conducting pathway created from tetrahedral phosphate thus giving rise to the conductivity of the sample. However the ionic conductivity value is still not high enough for application in electrochemical devices. Improve conductivity can be achieved by incorporation of an additive to the binary system. Poly(L-Leucine)1,3-diamino propane (PLLTMEDA) has been chosen as an additive due to its unique properties that able to further increase the ionic conductivity. The electrical conductivity of NaI-Na₃PO₄ and NaI-Na₃PO₄-PLLTMEDA were obtained by employing impedance spectroscopy (IS) technique. It was found the addition of PLLTMEDA resulted in an increase of electrical conductivity. The maximum conductivity of the new system NaI-Na₃PO₄ with 4 wt. % of PLLTMEDA shows maximum conductivity of $(1.12\pm0.68) \times 10_{-3}$ S cm₋₁. The temperature dependence conductivity studies



show that both systems are Arrhenius in nature and the transport properties can be descript by the hopping mechanism. The activation energy obtained for NaI-Na₃PO₄ is 0.34 eV and NaI- Na₃PO₄ with PLLTMEDA is 0.26 eV. The collected data from IS studies were analyzed in various complex planes such as impedance, admittance and permittivity for dielectric studies. Ac conductivity is analyzed using the Johscher's universal power law and the hopping mechanism of the charge carriers for both systems follow quantum mechanical tunneling (QMT) model. Ionic transference number was found to be 0.92 and 0.96 for the optimum composition of binary system and binary with additives respectively. This implies that the samples are ionic in nature. The FTIR spectra of NaI-Na₃PO₄-PLLTMEDA also verify the chelation of I- resulted in the immobilization of anion to give rise to high mobility and elevations of the charge carriers to traverse along the conducting pathway created from tetrahedral phosphate thus giving rise to the conductivity of the sample. Result obtained from NMR revealed the narrowing of the line width 23Na spectra in the optimum composition of the binary NaI-Na₃PO₄-PLLTMEDA system can be assigned to Na population with higher ion mobility. The X-ray diffractogram of the binary with PLLTMEDA shows that the system has become semi-crystalline in nature. Field emission scanning electron microscopy micrographs revealed finer microstructure of the milling samples with grains growth formation and densification upon sintering. The fabricated cell using 50 wt. % of NaI and 4 wt. % of PLLTMEDA showed better performance with discharge time of 173 hours at 1.0 µA current and the value of open circuit voltage is 3.0 V at room temperature.