

# Sintesis dan karakterisasi material katoda $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$ dengan $0 < x < 0.2$ dalam aplikasi baterai Li-Ion = Synthesis and characterization of cathode material $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$ with $0 < x < 0.2$ in application of Li-Ion battery

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## Abstrak

Struktur Olivine  $\text{LiMnPO}_4$  sebagai material katoda baterai Li-ion memiliki daya tarik tersendiri dikarenakan nilai potensial oksidasi-reduksi yang tinggi yaitu 4.2 volt terhadap  $\text{Li}/\text{Li}^+$ , stabil secara termal, dan relatif ramah lingkungan (nontoxic).

Namun nilai konduktifitas ionik dan elektronik yang rendah sekitar ( $10^{-9}$  S/cm), nilai specific capacity yang rendah akibat distorsi kisi (Jahn-Teller effect),

menjadi tantangan tersendiri. Proses pelapisan karbon pada bahan aktif  $\text{LiMnPO}_4$  dengan menggunakan starch atau pati singkong, substitusi kation dengan penambahan Fe dan Ni (covalent-doping) dimana formulasi  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}$

$\text{Ni}_x\text{PO}_4/\text{C}$  dengan  $0 < x < 0.2$  digunakan untuk meningkatkan konduktifitas elektronik-ionik, nilai specific capacity dan working voltage (Voksidasi/reduksi).

Pengujian XRD menunjukkan pola difraksi struktur kristal  $\text{LiMnPO}_4$  telah berhasil terbentuk melalui proses milling (330 rpm, 48 jam) dan sintering disuhu  $800^\circ\text{C}$  (solid state reaction). Proses reduksi ukuran dan coating karbon dengan Ball Milling mampu menghasilkan partikel bahan aktif  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  dengan  $0 < x < 0.2$  berukuran hingga 290 nanometer dengan ukuran kristalit hingga 60 nanometer.

Pertumbuhan pelapisan karbon kearah horizontal pada bahan aktif  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  dengan  $0 < x < 0.2$  menjadi bukti bahwa starch atau pati singkong berperan sebagai fasilitator pengintian pelapisan karbon dan terlihat pada pengujian SEM (perbesaran 50000 x) dan pengujian EDX dengan kadar Mn

yang tinggi menjadi bukti penguat. Frame network polianion terbentuk pada bahan aktif  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}$

$\text{Ni}_x\text{PO}_4/\text{C}$  dengan  $0 < x < 0.2$  ditandai dengan nilai vibrasi  $\nu_1$ -  $\nu_4$  ( $1138$  dan  $1098$   $\text{cm}^{-1}$ ) yang dominan

muncul pada hasil pengujian FTIR. Penambahan karbon sebagai pelapis bahan aktif memberikan nilai

konduktifitas elektronik (pasif) dan ionik (aktif) yang cukup tinggi sekitar  $1 \times 10^{-3}$  S/cm dan  $7.2$  S/cm,

dimana penambahan Ni (doping kation) berkontribusi dalam peningkatan nilai konduktifitas elektronik

(pasif). Komposisi bahan aktif

$\text{LiMn}_{0.7}\text{Fe}_{0.25}\text{Ni}_{0.05}\text{PO}_4/\text{C}$  menunjukkan nilai specific capacity oksidasi hingga  $60.92$  mAh/gr dan nilai

Voksidasi-reduksi sekitar 4.13 volt dan mampu digunakan

sebagai bahan aktif katoda baterai Li-ion secara praktikal dari hasil pengujian cyclic voltammetry. Puncak

Voksidasi/reduksi ganda yang merupakan kontribusi

Voksidasi  $\text{Fe}^{2+}/\text{Fe}^{3+}$  dan  $\text{Mn}^{2+}/\text{Mn}^{3+}$  sering terlihat pada hasil pengujian cyclic voltammetry.

.....Olivine  $\text{LiMnPO}_4$  structure as cathode material in Li-ion battery have very

attractive because its high potential oxidation/reduction around 4.2 volts vs.  $\text{Li}/\text{Li}^+$ ,

thermally stable, and nontoxic. Its low electronic and ionic conductivity around

( $10^{-9}$  S/cm), low specific capacity by lattice distortion (Jahn-Teller effect),

become its challenges. Carbon-coating process with starch of cassava in cathode

material  $\text{LiMnPO}_4$ , co-substitution by adding Fe and Ni where  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  with  $0 \leq x \leq 0.2$  formulation have been used to enhanced ionic/electronic conductivity, specific capacity, and working voltage of cathode material. Pattern diffraction of XRD shown  $\text{LiMnPO}_4$  structure have been formed via milling process (330 rpm, 48 hours) and sintering process at  $800^\circ\text{C}$  (solid state reaction). Size reduction process and carbon coating have been carried and produced cathode material  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  with  $0 \leq x \leq 0.2$  with the particle size up to 290 nanometers and crystallite size up to 60 nanometers. Carbon-coating process have been grown in horizontal direction in cathode material  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  with  $0 \leq x \leq 0.2$  and become approval that the starch of cassava have been facilitates nuklea of carbon-coating to grown in cathode material and can be seen by SEM with magnification 50000 times, and also the high content of Mn that have founded by EDX evaluation agreed. Frame network of polyanion have formed in cathode material  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  with  $0 \leq x \leq 0.2$  indicated by vibration value of  $\nu_1$ -  $\nu_4$  (1138 and 1098  $\text{cm}^{-1}$ ) that appeared dominantly during FTIR evaluation. Electronic conductivity (passive) of cathode material  $\text{LiMn}_{0.7}\text{Fe}_{0.3-x}\text{Ni}_x\text{PO}_4/\text{C}$  with  $0 \leq x \leq 0.2$  increased significantly up to  $1 \times 10^{-3} \text{ S/cm}$  by carbon-adding process as carbon-coating in cathode material, where the process of Ni-added as cation-doping also contribute in increasing the value of electronic conductivity. Based of cyclic voltammetry evaluation the formulation  $\text{LiMn}_{0.7}\text{Fe}_{0.25}\text{Ni}_{0.05}\text{PO}_4/\text{C}$  of cathode material shown the highest specific capacity oxidation near 60.92 mAh/gr and  $V_{\text{oxidation/reduction}}$  around 4.13 volts and practically can be used as Li-ion battery. Doble  $V_{\text{oxidation/reduction}}$  peak appeared several times as the contribution of  $V_{\text{oxidation/reduction}}$   $\text{Fe}^{2+}/\text{Fe}^{3+}$  and  $\text{Mn}^{2+}/\text{Mn}^{3+}$  in cyclic voltammetry evaluation.