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Results	Query	Domains (original links)
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>	-
Unique	<a href="#">anisa@gmail.com Received 22 November 2019</a>	-
Unique	<a href="#">Magnetite sand Fe<sub>3</sub>O<sub>4</sub> was used as Fe- source in LFP/C synthesized</a>	-
Unique	<a href="#">Calcination temperature of the sample performed at 400, 500, and 600°C</a>	-
Unique	<a href="#">The theoretical charging capacity of LFP is 170 mAh/g [2]</a>	-
Unique	<a href="#">Moreover, the LFP is environmentally more benign material than the other cathode materials</a>	-
Unique	<a href="#">For example LiCoO<sub>2</sub>, indicates toxicity issue and also costly in preparation</a>	-
Unique	<a href="#">However, the LFP based material has a low electrical conductivity</a>	-
Unique	<a href="#">However, these materials are still very expensive and even some of them are toxics</a>	-
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>	-
Unique	<a href="#">It was analyzed using x-ray fluorescene (XRF) spectrometer</a>	-
Unique	<a href="#">The Fe<sub>3</sub>O<sub>4</sub> is composed of FeO</a>	-
Unique	<a href="#">However no work have been reported in manufacturing using of the local Indonesian resources</a>	-
Unique	<a href="#">H<sub>2</sub>O, and natural iron sand with 98% Fe element content</a>	-

Unique	<a href="#">Synthesis of Precursor LFP/C compounds were prepared by high energy ball milling method [8]</a>		-
Unique	<a href="#">The resulted precursor was dried at 100°C (LFP-0) in oven</a>		-
Unique	<a href="#">Then, similar procedure was applied and calcination was undergone in different temperature,</a>		-
Unique	<a href="#">400°C (LFP-1), 500°C (LFP-2), and 600°C (LFP-3)</a>		-
Unique	<a href="#">XRD was adopted to identify crystalline phases on the samples</a>		-
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>		-
Unique	<a href="#">This indicates, that within this temperatures, a phases transformation undergoes</a>		-
Unique	<a href="#">It is showed by thermal uprising decline the mass of sample</a>		-
Unique	<a href="#">At temperature 200-300 °C, a weight-mass loss is sharply observed</a>		-
Unique	<a href="#">However, in temperature above 300°C, there is no significant mass reduction observed</a>		-
Unique	<a href="#">In this temperature, the phase of Lithium Ferro Phosphate LFP is formed</a>		-
1 results	<a href="#">The result from DSC-TGA analysis of the sample</a>	<a href="http://jpacr.ub.ac.id">jpacr.ub.ac.id</a>	
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>		-
Unique	<a href="#">The mass loss recorded is 2.99, 2.96, and 0.70 mg/°C, respectively</a>		-
Unique	<a href="#">In overall, similar diffraction pattern is observed for LFP-1, LFP-2, and LFP-3</a>		-
Unique	<a href="#">These similarity can also indicate similar composition and crystallinity of the LFP/C composite synthesized</a>		-
1 results	<a href="#">Calcination process at higher temperature give different LFP/C composite phase</a>	<a href="http://jpacr.ub.ac.id">jpacr.ub.ac.id</a>	
Unique	<a href="#">Lastly, at the highest temperature</a>		-
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>		-
Unique	<a href="#">Furthermore, it is also observed that increasing calcincation temperature indicate crystal agglomeration undergone</a>		-
Unique	<a href="#">The FTIR spectra of LFP/C sample</a>		-
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>		-
Unique	<a href="#">Modes in diphosphate consist of PO 3 and POP, and double bond PO [13]</a>		-
Unique	<a href="#">Their phase and composition are also able to be determined</a>		-

Unique	<a href="#">The diphosphate phase LiFeP<sub>2</sub>O<sub>7</sub> at is obtained at 400°C (LFP-1)</a>	-
Unique	<a href="#">The FTIR analysis also confirms the presence of their functional groups vibration modes</a>	-
Unique	<a href="#">Technol., 2016, 19(1), 178-188</a>	-
Unique	<a href="#">Acta, 2009, 54 (20), 4595-4599</a>	-
Unique	<a href="#">W., Powder Technol., 2010, 197(3), 309-313</a>	-
Unique	<a href="#">Power Sources, 2009, 189 (1), 155-162</a>	-
Unique	<a href="#">Colloid Interface Sci., 2018, 512, 398-403</a>	-
Unique	<a href="#">and Grout, H., Inorganics, 2014, 2(1), 132-154</a>	-
Unique	<a href="#">Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www</a>	-
Unique	<a href="#">Matter, 2011, 23(15), 3495-3508</a>	-
Unique	<a href="#">S., Kalska, B., Eyob, P., Aernout, D., Häggström,</a>	-
Unique	<a href="#">O., Solid State Ion, 2001, 140(1-2), 63-70</a>	-
33 results	<a href="#">A, Jozwiak, P., Zaghib, K., Garbarczyk, J., Gendron, F., Mauger,</a>	<a href="#">jpacr.ub.ac.id ui.adsabs.harvard.edu</a> <a href="#">pubmed.ncbi.nlm.nih.gov mdpi.com</a> <a href="#">iopscience.iop.org researchgate.net</a>
Unique	<a href="#">permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly</a>	-
Unique	<a href="#">Science and Engineering, University of Bojonegoro 2 Physics Department, Faculty of Mathematic and Sciences, Institute</a>	-
Unique	<a href="#">Accepted 15 April 2020 ABSTRACT Lithium Ferro Phosphate Carbon Composite (LFP/C) had been synthesized</a>	-
Unique	<a href="#">The phase and composition of samples determined by Rietveld analysis of X-ray diffraction (XRD)</a>	-
Unique	<a href="#">the others phases were nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> and hematite</a>	-
Unique	<a href="#">As the temperature getting higher the diphosphate phase LiFeP<sub>2</sub>O<sub>7</sub> transform to</a>	-
Unique	<a href="#">The chemical bonds, lattice vibration and other structural features of the sample were investigated using</a>	-
Unique	<a href="#">Specific vibration modes in LFP-1 to LFP-3 for each bonding were shown by the</a>	-
Unique	<a href="#">Key word: LFP/C, Magnetite, Nasicon, Diphosphate, Phase INTRODUCTION The demand for rechargeable batteries, lithium-ion</a>	-

Unique	<a href="#">Unfortunately several lithium batteries such as LiCoO<sub>2</sub> , LiNiO<sub>2</sub> are toxic and not</a>	-
Unique	<a href="#">Another efforts has paid attention by using Fe-based material as an alternative replacement for</a>	-
Unique	<a href="#">Recently, the LFP has also attracted manufacturer interest because it has a lot of</a>	-
Unique	<a href="#">And also, it has a high value of capacity than the other cathode materials</a>	-
Unique	<a href="#">Conversely, the LFP based materials also has a good cycle stability, and excellent thermal</a>	-
Unique	<a href="#">These advantages, coupled with abundantly source of iron for the LFP synthesis become</a>	-
Unique	<a href="#">This paper applies citric acid as a carbon source was to be added to</a>	-
Unique	<a href="#">Research using natural iron sand composed of Fe<sub>3</sub>O<sub>4</sub> , can be</a>	-
Unique	<a href="#">Fe(CH<sub>3</sub>COO)<sub>2</sub> , FeSO<sub>4</sub> , and Fe(NO<sub>3</sub>)<sub>3</sub> had been</a>	-
Unique	<a href="#">the preliminary study indicated that the natural iron sand from Tanah Laut Kalimantan, Indonesia, contained</a>	-
Unique	<a href="#">Further analysis using x-ray diffraction (XRD) spectrometer showed the natural iron sand phase was</a>	-
Unique	<a href="#">Fe<sub>2</sub>O<sub>3</sub> which have trivalent Fe<sup>3+</sup> in Fe<sub>2</sub>O<sub>3</sub> and</a>	-
Unique	<a href="#">This paper reports the work result of applying of this natural iron sand from</a>	-
Unique	<a href="#">Several papers have reported similar concerns, due to the electrochemical performance of Fe</a>	-
Unique	<a href="#">Many methods have been used to make LFP/C such as coprecipitation, solvothermal, hydrothermal, solid-liquid,</a>	-
1 results	<a href="#">In this study, LFP/C was synthesized by a simple methodology, and implementing of the</a>	<a href="http://jpacr.ub.ac.id">jpacr.ub.ac.id</a>
Unique	<a href="#">CO<sub>3</sub> (Sigma-Aldrich), di- ammonium hydrogen phosphate (NH<sub>4</sub>)<sub>2</sub> HPO<sub>4</sub> (Sigma-Aldrich) with</a>	-
Unique	<a href="#">15 o to 65 o ) to identify the phase of LFP/C, and the FTIR</a>	-
Unique	<a href="#">The materials being used are Li<sub>2</sub>CO<sub>3</sub> , (NH<sub>4</sub>)</a>	-
Unique	<a href="#">A 5.0% weight citric acid as carbon sources was added to these materials to</a>	-
Unique	<a href="#">Then, 5.0 mL of alcohol was poured into a milling jar and milled together</a>	-
Unique	<a href="#">Characterization The thermal properties of the sample was investigated using DSC-TGA for determine the</a>	-
Unique	<a href="#">The calcination temperatures were varied in the range of (400-700 °C) to observe the</a>	-
Unique	<a href="#">The sample before getting heat treatment named LFP-0, while the samples had been calcinated</a>	-

Unique	<a href="#">Chemical bonds, structural properties, and lattice dynamics was examined by Fourier Transformed Infrared Spectroscopy</a>	-
Unique	<a href="#">The crystalline phases and its composition in the XRD pattern were analyzed using Search</a>	-
Unique	<a href="#">DISCUSSION DSC-TGA Analysis The thermal properties of LFP/C were analyzed using DSC-TGA at room temperature</a>	-
Unique	<a href="#">A large negative gradient at 100 to 300°C range temperature indicates a significant mass</a>	-
Unique	<a href="#">The heat flow diagram in figure 1 shows that there is a thermal phenomenon</a>	-
Unique	<a href="#">Within this temperature range, the implemented heat can not reduce the the mass sample</a>	-
Unique	<a href="#">However, it is predicted that, the heat radiated the sample is absorbed for</a>	-
1 results	<a href="#">such as diphosphate <math>\text{LiFeP}_2\text{O}_7</math>, nasicon <math>\text{Li}_3\text{Fe}_2(\text{PO}_4)_3</math></a>	<a href="http://jpacr.ub.ac.id">jpacr.ub.ac.id</a>
Unique	<a href="#">id p-ISSN : 2302 – 4690   e-ISSN : 2541 – 0733 19 The detailed</a>	-
Unique	<a href="#">The stepwise loss occur in peak of 149.81 °C and 196.83 °C, and 276.69</a>	-
Unique	<a href="#">It is predicted the decomposed of volatile compounds, such as water, alcoholic matter and</a>	-
Unique	<a href="#">Furthermore, the rest of the temperature shows a steady line and is oredicted that</a>	-
Unique	<a href="#">XRD Analysis Figure 2 is showed the XRD diffractogram of the samples lithium iron</a>	-
Unique	<a href="#">Calcination at 100 °C (LFP-0), 400 °C (LFP-1), 500 °C (LFP-2), and 600 °C</a>	-
Unique	<a href="#">75.4: 16.9: 7.7 % weight comparison for <math>(\text{NH}_4)_2\text{HPO}_4</math> : Fe</a>	-
Unique	<a href="#">, monoclinic <math>\text{LiFeP}_2\text{O}_7</math> and <math>\text{Li}_3\text{Fe}_2(\text{PO}_4)_3</math></a>	-
Unique	<a href="#">The magnetite <math>\text{Fe}_3\text{O}_4</math> is transformed become hematite <math>\text{Fe}_2\text{O}_3</math></a>	-
Unique	<a href="#">The major phase was formed at 400°C (LFP-1) calcination is diphosphate <math>\text{LiFeP}_2\text{O}_7</math></a>	-
Unique	<a href="#">is converted gradually, and turn it into nasicon <math>\text{Li}_3\text{Fe}_2(\text{PO}_4)_3</math></a>	-
Unique	<a href="#">(LFP-3), the dominant phases formed are nasicon <math>\text{Li}_3\text{Fe}_2(\text{PO}_4)_3</math></a>	-
1 results	<a href="#">The disphosphate form, <math>\text{LiFeP}_2\text{O}_7</math> is predicted become triphosphate, <math>\text{LiFe}(\text{PO}_4)_3</math></a>	<a href="http://jpacr.ub.ac.id">jpacr.ub.ac.id</a>
Unique	<a href="#">And also, the hematite <math>\text{Fe}_2\text{O}_3</math> composition is gradually decreased and turn</a>	-
Unique	<a href="#">Previously, the presence of this kind impurity deteriorated the battery performance, such as cyclability,</a>	-
Unique	<a href="#">Calcination at 600 o C give the highest content of LFP and a very</a>	-

Unique	<a href="#">FTIR Analysis Lattice dynamics of lithium iron phosphate-based materials composite in carbon (LFP- 1, LFP-2,</a>	-
Unique	<a href="#">The presence of functional group of these materials in the surface are recorded as</a>	-
Unique	<a href="#">Absorbance values (in absorption unit) is correlated to the energy absorbed by the specific</a>	-
Unique	<a href="#">2 (PO 4 ) 3 in which lithiums situated in the interstitial void in this</a>	-
Unique	<a href="#">The basic crystallography unit in diphosphate LiFeP 2 O 7 is P 2</a>	-
Unique	<a href="#">The mode vibration located at 1227 cm -1 assigned to the terminal P</a>	-
Unique	<a href="#">The spectra band appears at 764 cm -1 which is attributed to stretching modes</a>	-
Unique	<a href="#">The width and intense modes observed in 949 cm -1 are due to the</a>	-
Unique	<a href="#">Moreover, the asymmetric stretching vibration of PO 3 appears in 1126 cm -1</a>	-
Unique	<a href="#">The nasicon framework, Fe 2 (PO 4 ) 3 , consists of the octahedral</a>	-
Unique	<a href="#">vibration of the valence bond from the PO 4 3- group is observed in between</a>	-
Unique	<a href="#">to the coupling vibration of PO 4 in the nasicon compound, and that is not</a>	-
Unique	<a href="#">Conversely, these band peak are clearly observed in LFP-3 which is dominated by nasicon</a>	-
Unique	<a href="#">The more broadening bands observed in LFP-1 and LFP-2 show more variation of vibration</a>	-
Unique	<a href="#">In LFP-3 there is two phase, so the peak intensity can be differ clearly,</a>	-
Unique	<a href="#">CONCLUSION In short, lithium iron phosphate/carbon composite-based material cane synthesized in three different temperature</a>	-
Unique	<a href="#">The higher calcination temperature to at 600°C (LFP-3), decreased it gradually and transformed into</a>	-
Unique	<a href="#">The highest composition of nasicon is produced in 85.72% after calcination at 600</a>	-
Unique	<a href="#">It is also provide a low amount of impurity and give a stable LFP</a>	-
Unique	<a href="#">A., Synthesis of Li-ion battery cathode materials via freeze granulation, Master's thesis, Chalmers University of</a>	-
Unique	<a href="#">id p-ISSN : 2302 – 4690   e-ISSN : 2541 – 0733 22 [11] Hautier,</a>	-

DOI: 10.21776/ub.jpacr.2020.009.01.517 J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 This is an open access article distributed under the terms of the Creative Commons Attribution-NonCommercial 4.0 International which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. (<http://creativecommons.org/licenses/by-nc/4.0/>) Synthesis and Characterization of Lithium Iron Phosphate Carbon Composite (LFP/C) using Magnetite Sand Fe<sub>3</sub>O<sub>4</sub> Zuffa Anisa, \*1 Mochammad Zainuri 2 1 Chemistry Department, Faculty of Science and Engineering, University of Bojonegoro 2 Physics Department, Faculty of Mathematic and Sciences, Institute Technology of Sepuluh Nopember \* Corresponding email : zuffa.anisa@gmail.com Received 22 November 2019; Accepted 15 April 2020 ABSTRACT Lithium Ferro Phosphate Carbon Composite (LFP/C) had been synthesized using solid-state reaction method. Magnetite sand Fe<sub>3</sub>O<sub>4</sub> was used as Fe-source in LFP/C synthesized. Calcination temperature of the sample performed at 400, 500, and 600°C. The phase and composition of samples determined by Rietveld analysis of X-ray diffraction (XRD) pattern. The dominant identified phase at 400°C was diphosphate LiFeP<sub>2</sub>O<sub>7</sub>, and the others phases were nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> and hematite Fe<sub>2</sub>O<sub>3</sub>. As the temperature getting higher the diphosphate phase LiFeP<sub>2</sub>O<sub>7</sub> transform to nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>. The chemical bonds, lattice vibration and other structural features of the sample were investigated using FTIR spectroscopy in range of 1400 – 400 cm<sup>-1</sup>. Specific vibration modes in LFP-1 to LFP-3 for each bonding were shown by the high intense in certain wavenumber. Key word: LFP/C, Magnetite, Nasicon, Diphosphate, Phase INTRODUCTION The demand for rechargeable batteries, lithium-ion batteries has increased significantly. Unfortunately several lithium batteries such as LiCoO<sub>2</sub>, LiNiO<sub>2</sub> are toxic and not environmentally benign [1]. Another efforts has paid attention by using Fe-based material as an alternative replacement for the active materials of Lithium Ferro Phosphate (LFP) in lithium ions batteries. Recently, the LFP has also attracted manufacturer interest because it has a lot of advantages like charging capacity and many others. The theoretical charging capacity of LFP is 170 mAh/g [2]. And also, it has a high value of capacity than the other cathode materials such as LiMn<sub>2</sub>O<sub>4</sub> with 117 mAh/g charging capacity. Moreover, the LFP is environmentally more benign material than the other cathode materials. For example LiCoO<sub>2</sub>, indicates toxicity issue and also costly in preparation. Conversely, the LFP based materials also has a good cycle stability, and excellent thermal stability. These advantages, coupled with abundantly source of iron for the LFP synthesis become a promising and reasonable concern for production. However, the LFP based material has a low electrical conductivity. This paper applies citric acid as a carbon source was to be added to make a Lithium Iron Phosphate Carbon composite (LFP/C) for overcoming of the drawback [3]. Research using natural iron sand composed of Fe<sub>3</sub>O<sub>4</sub>, can be used as iron sources to form LFP/C precursor are still a wide apart. Some divalent and trivalent Fe-sources such as Fe<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O, Fe(CH<sub>3</sub>COO)<sub>2</sub>, FeSO<sub>4</sub>, and Fe(NO<sub>3</sub>)<sub>3</sub> had been practiced as well [4]. However, these materials are still very expensive and even some of them are toxic. J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 17 Based on the preliminary study indicated that the natural iron sand from Tanah Laut Kalimantan, Indonesia, contained 98% of Fe. It was analyzed using x-ray fluorescence (XRF) spectrometer. Further analysis using x-ray diffraction (XRD) spectrometer showed the natural iron sand phase was identified as magnetite Fe<sub>3</sub>O<sub>4</sub>. The Fe<sub>3</sub>O<sub>4</sub> is composed of FeO.Fe<sub>2</sub>O<sub>3</sub> which have trivalent Fe<sup>3+</sup> in Fe<sub>2</sub>O<sub>3</sub> and divalent Fe<sup>2+</sup> ion in FeO. This paper reports the work result of applying of this natural iron sand from this specific location for LFP/C precursor. Several papers have reported similar concerns, due to the electrochemical performance of Fe<sub>3</sub>O<sub>4</sub> and also the low-cost production potency. However no work have been reported in manufacturing using of the local Indonesian resources. Many methods have been used to make LFP/C such as coprecipitation, solvothermal, hydrothermal, solid-liquid, and freeze granulation [5], but some of them are rather complicated. **In this study, LFP/C was synthesized by a simple methodology, and implementing of the** solid state synthesis strategy using high energy ball milling process [6,7]. EXPERIMENT Chemicals and Instrumentation Materials used for this research were lithium carbonate Li<sub>2</sub>CO<sub>3</sub> (Sigma-Aldrich), di-ammonium hydrogen phosphate (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (Sigma-Aldrich) with purity >98%, citric acid C<sub>8</sub>H<sub>8</sub>O<sub>7</sub>.H<sub>2</sub>O, and natural iron sand with 98% Fe element content. The instruments used for this study are oven, furnace, differential scanning calorimetry-thermal gravimetric analysis (DSC-TGA) (Mettler Toledo Star SW 10.0, thermal analysis from room temperature to 1400 °C and sample weight 15.2 mg), x-ray diffraction (XRD) spectrometer (Siemens D-501, Ni filter and graphite monochromator, x-ray source from Cu Kα radiation lambda 1.5406 Å, with scanning angle from 15 ° to 65 °) to identify the phase of LFP/C, and the FTIR spectrophotometer (recorded in Shimadzu 8400S). Synthesis of Precursor LFP/C compounds were prepared by high energy ball milling method [8]. The materials being used are Li<sub>2</sub>CO<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, and Fe<sub>3</sub>O<sub>4</sub> with 3:6:2 of mole ratio. A 5.0% weight citric acid as carbon sources was added to these materials to enhance the electron conductivity and to avoid oxidation of Fe ion. Then, 5.0 mL of alcohol was poured into a milling jar and milled together in 300 rpm rotation speed, for 3 hours. The resulted precursor was dried at 100°C (LFP-0) in oven. Then, similar procedure was applied and calcination was undergone in different temperature, i.e. 400°C (LFP-1), 500°C (LFP-2), and 600°C (LFP-3). Characterization The thermal properties of the sample was investigated using DSC-TGA for determine the variation temperature will be given to the sample. The calcination temperatures were varied in the range of (400-700 °C) to observe the formed phases of Lithium Ferro Phosphate Carbon composite (LFP/C) [9]. The sample before getting heat treatment named LFP-0, while the samples had been calcinated in 400, 500, and 600 °C named LFP-1, LFP-2, and LFP-3 respectively. XRD was adopted to identify crystalline phases on the samples. Chemical bonds, structural properties, and lattice dynamics was examined by Fourier Transformed Infrared Spectroscopy (FTIR). The crystalline phases and its composition in the XRD pattern were analyzed using Search Match software qualitatively and Rietica quantitatively. J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 18 RESULT AND DISCUSSION DSC-TGA Analysis The thermal properties of LFP/C were analyzed using DSC-TGA at room temperature until 1300°C. A large negative gradient at 100 to 300°C range temperature indicates a significant mass reduction in sample weight. The heat flow diagram in figure 1 shows that there is a thermal phenomenon at 400°C until 700°C. This indicates, that within this temperatures, a phases transformation undergoes. It is showed by thermal uprising decline the mass of sample. At temperature 200-300 °C, a weight-mass loss is sharply observed. However, in temperature above 300°C, there is no significant mass reduction observed. Within this temperature range, the implemented heat can not reduce the the mass sample farther. However, it is predicted that, the heat radiated the sample is absorbed for a phase transformation. In this temperature, the phase of Lithium Ferro Phosphate LFP is formed. Phases transformation from the initial composition for Li<sub>2</sub>CO<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub>, and magnetite Fe<sub>3</sub>O<sub>4</sub>, to other phases, **such as diphosphate LiFeP<sub>2</sub>O<sub>7</sub>, nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> and hematite.** Figure 1. **The result from DSC-TGA analysis of the sample** J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 19 The detailed of mass loss of LFP/C from DTA-TGA analysis indicate the physical properties of this material. The stepwise loss occur in peak of 149.81 °C and 196.83 °C, and 276.69 °C. The mass loss recorded is 2.99, 2.96, and 0.70 mg/g, respectively. It is predicted the decomposed of volatile compounds, such as water, alcoholic matter and ammonia. Furthermore, the rest of the temperature shows a steady line and is predicted that the compounds with a stable composition is formed. XRD Analysis Figure 2 is showed the XRD diffractogram of the samples lithium iron phosphat/carbon composite (LFP/C) before and after calcination process. Calcination at 100 °C (LFP-0), 400 °C (LFP-1), 500 °C (LFP-2), and 600 °C (LFP-3). In overall, similar diffraction pattern is observed for LFP-1, LFP-2, and LFP-3. These similarity can also indicate similar composition and crystallinity of the LFP/C composite synthesized. Figure 2. The XRD diffractogram of lithium iron phosphate/carbon (LFP/C) composite Phase compositions of LFP-0 are 75.4: 16.9: 7.7 % weight comparison for (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> : Fe<sub>3</sub>O<sub>4</sub> : Li<sub>2</sub>CO<sub>3</sub> respectively. The formed phases at 400, 500, 600°C temperature are trigonal Fe<sub>2</sub>O<sub>3</sub>, monoclinic LiFeP<sub>2</sub>O<sub>7</sub> and Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> [10], with the composition are summarized in Table 1. **Calcination process at higher temperature give different LFP/C composite phase.** The magnetite Fe<sub>3</sub>O<sub>4</sub> is transformed become hematite Fe<sub>2</sub>O<sub>3</sub>. The major phase was formed at 400°C (LFP-1) calcination is diphosphate LiFeP<sub>2</sub>O<sub>7</sub>. Meanwhile, at higher temperature calcination, 500°C, the diphosphate phase of LiFeP<sub>2</sub>O<sub>7</sub> is converted gradually, and turn it into nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>. Lastly, at the highest temperature J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 20 calcination, 600°C (LFP-3), the dominant phases formed are nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> in 85.72%. in the absence of diphosphate LiFeP<sub>2</sub>O<sub>7</sub> is observed. Furthermore, it is also observed that increasing calcination temperature indicate crystal agglomeration undergone. **The diphosphate form, LiFeP<sub>2</sub>O<sub>7</sub> is predicted become triphosphate, LiFe(PO<sub>4</sub>)<sub>3</sub> form.** And also, the hematite Fe<sub>2</sub>O<sub>3</sub> composition is gradually decreased and turn in trace. Previously, the presence of this kind impurity deteriorated the battery performance, such as cyclability, charge-discharge capacity, and conductivity [11]. Calcination at 600 °C give the highest content of LFP and a very low of impurities. Table 1. %-Weight phase composition of LFP/C after calcination Sample Diphosphate (LiFeP<sub>2</sub>O<sub>7</sub>) (%) Nasicon (Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>) (%) Hematite (Fe<sub>2</sub>O<sub>3</sub>) (%) LFP-1 58.28 12.06 29.66 LFP-2 32.57 47.89 19.54 LFP-3 85.72 14.28 FTIR Analysis Lattice dynamics of lithium iron phosphate-based materials composite in carbon (LFP-1, LFP-2, and LFP-3) are studied by using FTIR spectrophotometric. The presence of functional group of these materials in the surface are recorded as the vibrational of each group due to irradiated by photon in infra red wavelength. Absorbance values (in absorption unit) is correlated to the energy absorbed by the specific functional group composed in LFP/C composite. Figure 3. The FTIR spectra of LFP/C sample J. Pure App. Chem. Res., 2020, 9 (1), 16-22 15 April 2020 X The journal homepage www.jpacr.ub.ac.id p-ISSN : 2302 – 4690 | e-ISSN : 2541 – 0733 21 The nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> is built from anion framework of Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub> in which lithiums situated in the interstitial void in this framework [12]. The basic crystallography unit in diphosphate LiFeP<sub>2</sub>O<sub>7</sub> is P<sub>2</sub>O<sub>7</sub> anion with 2 main PO<sub>4</sub> tetrahedral bridging θ POP angle. Modes in diphosphate consist of PO<sub>3</sub> and POP, and double bond PO [13]. The mode vibration located at 1227 cm<sup>-1</sup> assigned to the terminal P<sub>2</sub>O<sub>7</sub> ions stretching mode [10] as presented in Figure 3. The spectra band appears at 764 cm<sup>-1</sup> which is attributed to stretching modes of P-O-P bridges. The width and intense modes observed in 949 cm<sup>-1</sup> are due to the normal vibration of the bridging oxygen atom with alkali ion [14]. Moreover, the asymmetric stretching vibration of PO<sub>3</sub> appears in 1126 cm<sup>-1</sup>, with the highest intensity detected in LFP-1 and also found in LFP-2. The nasicon framework, Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>, consists of the octahedral of FeO<sub>6</sub> and tetrahedral of PO<sub>4</sub> ion. Meanwhile, the vibration octahedral of FeO<sub>6</sub> occurs below 450 cm<sup>-1</sup>, and vibration of the valence bond from the PO<sub>4</sub> group is observed in between 400 and 700 cm<sup>-1</sup>. The splitting of band peak at 1026, 1038, and 1072 cm<sup>-1</sup> are attributed to the coupling vibration of PO<sub>4</sub> in the nasicon compound, and that is not observed in LFP-1. Conversely, these band peak are clearly observed in LFP-3 which is dominated by nasicon phase. The more broadening bands observed in LFP-1 and LFP-2 show more variation of vibration occurring, and because of more phases is formed. In LFP-3 there is two phase, so the peak intensity can be differ clearly, even in the XRD pattern of LFP-3 show less peak with the high crystallinity. CONCLUSION In short, lithium iron phosphate/carbon composite-based material cane synthesized in three different temperature of calcination. Their phase and composition are also able to be determined. The diphosphate phase LiFeP<sub>2</sub>O<sub>7</sub> at is obtained at 400°C (LFP-1). The higher calcination temperature to at 600°C (LFP-3), decreased it gradually and transformed into nasicon Li<sub>3</sub>Fe<sub>2</sub>(PO<sub>4</sub>)<sub>3</sub>. The FTIR analysis also confirms the presence of their functional groups vibration modes. The highest composition of nasicon is produced in 85.72% after calcination at 600 °C (LFP-3). It is also provide a low amount of impurity and give a stable LFP phase. REFERENCES [1] Kasvayee, K.A., Synthesis of Li-ion battery cathode materials via freeze granulation, Master's thesis, Chalmers University of Technology, Sweden, 2011. [2] Satyavathi, T.V.S.L., Kumar, A.S., and Rao, P.S., Int. J. Eng. Sci. Technol., 2016, 19(1), 178-188. [3] Chang, Z.R., Lv, H.J., Tang, H.W., Li, H.J., Yuan, X.Z., and Wang, H., Electrochim. Acta, 2009, 54 (20), 4595-4599. [4] Zhang, B., Ou, X., Zheng, J.C., Ming, L., Han, Y.D., Wang, J.L. and Qin, S.E., Electrochim. Acta, 2014, 133, 1-7. [5] Liu, X.H., and Zhao, Z.W., Powder Technol., 2010, 197(3), 309-313. [6] Wang, Y., Feng, Z.S., Chen, J.J., and Zhang, C., Mater. Lett., 2012, 71, 54-56. [7] Wang, J., Shao, Z. and Ru, H., Ceram. Int., 2014, 40 (5), 6979-6985. [8] Lu, C.Z., Fey, G.T.K., and Kao, H.M., J. Power Sources, 2009, 189 (1), 155-162. 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