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## Preface

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## **Preface**

The International Conference on Theoretical and Applied Physics (The ICTAP 2019) is a conference organized by Physical Society of Indonesia (PSI), University of Lampung (Dept. of Physics, Dept. of Physics Education, and Dept. of Geophysics Engineering); Dept. of Physics Education - Raden Intan State Islamic University of Lampung; Dept. of Physics - Technology Institut of Sumatera; Dept. of Physics Education- Univ. of Muhammadiyah Metro; Research Unit for Mineral Technology Lampung- Indonesian Institute of Sciences; Research & Standardization Industry Lampung, and Physics Teacher Forum of Bandar Lampung. It is an annual conference of the Society and moves from one major city to another, started seven years back in Bandung (Capital city of West Java Province), then consecutively moved to Palangkaraya (Capital city of Central Kalimantan Province, 2012), Malang (East Java, 2013), Denpasar (Capital city of Bali Province, 2014), Kendari (Capital city of South-East Sulawesi Province, 2015), Makasar (Capital city of South Sulawesi Province, 2016). Yogyakarta (Capital city of Special Region of Yogyakarta Province, 2017), and Medan (Capital city of South Sumatera Province, 2018).

The ICTAP 2019 was held in Bandar Lampung, Lampung, on 26<sup>th</sup>-28<sup>th</sup> September 2019, with the main theme "Physics and Smart Society." It was aimed for promoting, developing, and disseminating interdisciplinary research from many different fields of physics. Researchers from universities, institutes, and related industries, working on various fields of physics were invited to participate to present papers or just as a participant.

We would like to acknowledge all of those who have supported ICTAP 2019. Each individual and institutional help were very important for the success of this conference. Especially we would like to thank the organizing committee for their valuable advices in the organization and helpful peer review of the papers.

We hope that ICTAP 2019 will be a forum for excellent discussions that will put forward new ideas and promote collaborative researches. We are sure that the proceedings will serve as an important research source of references and the knowledge, which will lead to not only scientific and engineering progress but also other new products and processes.

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## Table of contents

## Volume 1572

## 2020

◆ Previous issue Next issue ▶

The 9th International Conference on Theoretical and Applied Physics (ICTAP) 26-28 September 2019, Bandar Lampung, Indonesia

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Preface	
OPEN ACCESS	011001
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<b>+</b> Open abstract	
OPEN ACCESS	011002
Peer review statement	
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Papers	
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Y Dwihapsari, M Evalisa and F Nazir	
<b>+</b> Open abstract	
OPEN ACCESS  A comparation of different strategies in Principle Component for clustering human tooth surface using Laser-Induced Break	
N L P Trisnawati, A Krisandi, I G A Widagda, I E Suprihatin and H Su	yanto
<b>+</b> Open abstract	
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Z Zulmaidah, A Suyatna and U Rosidin

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OPEN ACCESS 012006

Analysis of gravity anomaly for groundwater basin in Bandar Lampung city based on 2D gravity modeling

A Zaenudin, R Risman, I G B Darmawan and I B S Yogi

+ Open abstract





OPEN ACCESS 012007

Analysis of mineral sediment characteristics of Bantimurung Bulusaraung National Park in the Karst Maros Region

M Arsyad, N Ihsan and V A Tiwow

+ Open abstract





OPEN ACCESS 012008

Analysis of physical properties and mechanics of rocks in the karst region of Pangkep Regency

M Arsyad, V A Tiwow, Sulistiawaty and I A Sahdian

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Application of learning models conceptual understanding procedures by using experimental methods on understanding physics concepts of students

N Nurlina, R A Lestari and R Riskawati

+ Open abstract





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Assessing the temperature of plasma produced in carbon dioxide (CO<sub>2</sub>) Laser-Induced Breakdown Spectroscopy (LIBS) from soil sample prepared by two different methods of metal mesh This site uses cookies. By continuing to use this site you agree to our use of cookies. To find out more, and silicone grease cookies policy.

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Journal of Physics: Conference Series, Volume 1572, 2020 - IOPscience **OPEN ACCESS** 012018 Development of physics practicum module based on collaborative teamwork learning model M Mustari, P Marwoto, R S Iswari, F A Ginanjar and Y Anjelinar + Open abstract **■** View article 🄼 PDF **OPEN ACCESS** 012019 Digital oscillation rails: developing physics learning media to determine the acceleration value of earth's gravity R Diani, R B Satiarti, N Lestari, N B Haka, D Reftyawati, A Padilah and H Komikesari **■** View article PDF + Open abstract **OPEN ACCESS** 012020 ECIRR (Elicit, Confront, Identify, Resolve, Reinforce) learning model with the pictorial riddle method: is it effective in reducing physics misconceptions? R Diani, Y Yuberti, S Anggereni, G N Utami, A Iqbal and I Kurniawati View article **PDF** + Open abstract **OPEN ACCESS** 012021 Effect of ball-milling time on chemical property of coconut shell powder I Ismail, Arliyani, S Fathmiyah, Mursal, Z Jalil and H P S A Khalil + Open abstract **■** View article 🄼 PDF **OPEN ACCESS** 012022 Effect of free parametrized TOV on properties of neutron stars D Purnamasari and A Sulaksono View article 🔁 PDF + Open abstract **OPEN ACCESS** 012023 Effect size test of 7e learning cycle model: conceptual understanding and science process skills on senior high school students H Komikesari, W Anggraini, N Asiah, P S Dewi, R Diani and M N Yulianto View article 🔁 PDF + Open abstract **OPEN ACCESS** 012024 Effectiveness of physics electronic modules based on Self Directed Learning Model (SDL) towards the understanding of dynamic fluid concept

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Effects of magnetic fields on the Current-Voltage (I-V) characteristics of the chitosan membrane

N N Rupiasih, M Sumadiyasa, I K Putra, R Wulandari and I W Sari

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OPEN ACCESS 012027

Electrodepolymerisation of polyaniline film for use as an electrode in FeNi alloy electrodeposition

S Budi, A S Putri, A Mahmud, Afrizal, M Paristiowati, S Muhab, U Cahyana and A Purwanto

+ Open abstract

View article



OPEN ACCESS 012028

Elemental analysis of agriculture soil by a pulsed carbon dioxide (CO<sub>2</sub>) Laser Induced Breakdown Spectroscopy (LIBS)

N Idris, D Susilayani, A M Sari, K Lahna, M Ramli, K Kurihara and Marwan

+ Open abstract

View article



OPEN ACCESS 012029

Environment management in prospect coal mining area at Handil Kutai Kartanegara, East Kalimantan using resistivity tomography, wenner configuration

D Parwatiningtyas and B W A Putro

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Escape hill development as a strategy to improve urban safety after earthquake and tsunami Aceh 2004 based on regional planning and geotechnical aspect

M Munirwansyah, H Munirwan, M Irsyam and R P Munirwan

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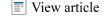


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D Kartini and A Sulaksono

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A Suyatna

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O Dewanto, Maulina and T B Nainggolan

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## A preliminary study of phases, elemental mapping, and electrical properties on Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica

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# A preliminary study of phases, elemental mapping, and electrical properties on Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica

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**Abstract.** This work reports a preliminary investigation about the phase, element mapping, and electrical properties of Na<sub>2</sub>FeSiO<sub>4</sub> prepared from rice husk silica, (FeNO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O, NaOH, and C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>.H<sub>2</sub>O using the sol-gel method. A sample sintered at 800 °C with a holding time of 10 hours at peak temperature. The phases identification shows that the main phase of Na<sub>2</sub>FeSiO<sub>4</sub> had been formed in the sample accompanied by two impurity phases, i.e., Na<sub>2</sub>SiO<sub>3</sub> and SiO<sub>2</sub>. Elemental mapping shows that Na, Fe, Si, and O elements are evenly distributed over the entire surface of the sample. The band gap energy value of the sample is relatively small, around 2.58 eV - 2.87 eV. Its electrical conductivity varies depending on frequency, i.e., 6.13x10<sup>-5</sup> S/m at 1 Hz and decreases gradually up to 4.27x10<sup>-5</sup> S/m at 1000 Hz.

### 1. Introduction

The search for new cathode materials has become a severe concern for researchers to create batteries with high specific capacities and low production costs. Li<sub>2</sub>FeSiO<sub>4</sub> orthosilicate with polyanion structure attracts the interest of researchers because the structure of the material allows the presence of two lithium ions per formula. Because of that, it can produce high specific capacities; theoretically, it can reach 330 mAhg<sup>-1</sup> [1-2]. The material also has lattice stability compared to other polyanion materials but shows poor electrical conductivity [3]. Besides, the limited availability of lithium causes the cost of making Li<sub>2</sub>FeSiO<sub>4</sub> to be expensive [4]. Therefore, other materials with similar characteristics but abundant raw materials available in nature need to be developed. Na<sub>2</sub>FeSiO<sub>4</sub> is a new type of polyanion material that can be used as an alternative to Li<sub>2</sub>FeSiO<sub>4</sub> because it has a similar ionic radius and potential redox characteristics [5-8]. Na<sub>2</sub>FeSiO<sub>4</sub> has excellent structural stability similar to Li<sub>2</sub>FeSiO<sub>4</sub>, and high theoretical specific capacity reaches 278 mAhg<sup>-1</sup> [9–11]. Although, in theory, Na<sub>2</sub>FeSiO<sub>4</sub> has a slightly lower specific capacity than Li<sub>2</sub>FeSiO<sub>4</sub>, Na<sub>2</sub>FeSiO<sub>4</sub> has much higher electrical conductivity and cheaper and also abundant raw materials compared to Li<sub>2</sub>FeSiO<sub>4</sub>. Electrical conductivity is one crucial factor in determining the performance of a cathode [12]. Therefore, Na<sub>2</sub>FeSiO<sub>4</sub> is very potential to be developed as a cathode material.

Na<sub>2</sub>FeSiO<sub>4</sub> can be produced using various types of methods, a kind of simple technique that is widely applied is the sol-gel method [13-14]. The source of silica used in this production is generally derived from tetraethyl orthosilicate (TEOS) [14-17]. The use of TEOS as a source of silica may be replaced with silica from rice husk to reduce production costs [18-19]. Rice husk has abundant availability in nature, and from that, rice husk can be extracted with silica reaching 99% purity through simple methods [20]. Besides, silica from rice husk has an amorphous structure and reactive so that it

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can be used as a raw material in the manufacture of various materials [21-22]. In our previous studies, rice husk silica was successfully used as a raw material for forsterite [23-24] and cordierite [25-26]. By utilizing rice husk silica in the process of making various materials, especially Na<sub>2</sub>FeSiO<sub>4</sub>, it can reduce production costs.

In this work, we prepare Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica. Preparation was carried out using the sol-gel method and followed by thermal treatment at 800 °C. The work aims to investigate the potential of rice husk silica as a raw material in the manufacture of Na<sub>2</sub>FeSiO<sub>4</sub> and investigate the possibility of Na<sub>2</sub>FeSiO<sub>4</sub> as a cathode in batteries system considered from its electrical characteristics. This work clearly describes the structure and electrical properties of the Na<sub>2</sub>FeSiO<sub>4</sub> sample, which included functional groups, phases, morphology, elemental distribution, bandgap energy, and electrical conductivity.

#### 2. Materials and methods

#### 2.1. Silica extraction from rice husk

Silica was extracted from rice husk refers to our previous study [27]. As much as 50 grams of rice husk was boiled in 500 ml of 5% KOH solution. The mixture obtained from this process was then left in room temperature for 24 hours and then filtered to obtain a silica sol. Silica sol was added 10% HNO<sub>3</sub> solution by dropwise to form a gel with a pH of 7. The gel was cleaned using deionized water and then dried at 110 °C to obtain a solid. The solid was ground and then sieve to get silica powder with particle size 200 meshes.

## 2.2. Na<sub>2</sub>FeSiO<sub>4</sub> preparation

Na<sub>2</sub>FeSiO<sub>4</sub> preparation using the sol-gel method refers to previously reported studies [28-31]. Na<sub>2</sub>FeSiO<sub>4</sub> was prepared from (FeNO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O, NaOH, rice husk silica, and C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>.H<sub>2</sub>O with a mole ratio of 1:2:1:1. Silica powder was dissolved in NaOH at 60 °C for 30 minutes, then added (FeNO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O and C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>.H<sub>2</sub>O solution by dropwise until it reached pH 1. The mixture was refluxed at 80 °C for 5 hours and then poured to the beaker glass. It was evaporated at 75 °C under magnetic stirring until a gel was obtained. The gel was dried at 130 °C for 3 hours and then ground to get a fine powder. After that, the fine powder was sintered at 800 °C with a temperature of 3 °C/minute and a holding time of 10 hours at peak temperature.

## 2.3. Functional groups analysis

Na<sub>2</sub>FeSiO<sub>4</sub> sample and KBr powder were ground and then pressed into a pellet. The functional groups in the prepared pellet were analyzed using the Nicolet iS10 FTIR Spectrometer by scanning in the wavelength range of 1250-400 cm<sup>-1</sup>. The analysis was conducted by comparing the FTIR spectrum with references published in previous studies.

## 2.4. Phase analysis

The phases were characterized by X'Pert Powder PW 30/40 XRD with Cu-Kα radiation. Samples were scanned at 2θ 10°-100°. Phase analysis was carried out using the search match method using Qual X software version 2.24 with Crystallography Open Database (COD).

## 2.5. Surface morphology and elemental distribution analysis

The surface morphology and distribution of the elements in the samples were analyzed using SEM/EDS Tescan Vega3.

## 2.6. Band gap energy estimation

Shimadzu UV-2450 UV-Vis spectrophotometer was used to measure the diffuse reflection of Na<sub>2</sub>FeSiO<sub>4</sub> sample powder in the wavelength range of 200-800 nm. The band gap energy was estimated using the Kubelka-Munk theorem shown by Equation (1)

**1572** (2020) 012003 doi:10.1088/1742-6596/1572/1/012003

$$F(R) = K/S = (1 - R)^2/2R \tag{1}$$

where F(R) is the function of Kubelka-Munk, S and K are the scattering and absorption coefficients respectively, and R is the diffuse reflection. The band gap  $(E_g)$  and the absorption coefficient is related through the Tauc relation. Tauc relation to the direct band gap is given in Equation (2)

$$(\alpha h \nu) = A \left( h \nu - E_g \right)^{1/2} \tag{2}$$

where  $\alpha$  is the linear absorption coefficient,  $h\nu$  is photon energy, A is the proportional constant, and  $E_g$  is band gap. When incident radiation scatters are perfectly diffuse manner, the absorption coefficient K becomes equal to  $2\alpha$ . In this case, considering the scattering coefficient S as constant concerning wavelength, the Kubelka-Munk is proportional to the absorption coefficient  $\alpha$ , applying Equation 1 can be obtained from the relation such as Equation (3)

$$[F(R)h\nu]^2 = A(h\nu - E_g)$$
(3)

## 2.7. Electrical conductivity measurement

LCR meter was used to measure the conductance of  $Na_2FeSiO_4$  pellet with a diameter of  $1x10^{-2}$  m and a thickness of  $3x10^{-3}$  m. The conductance was measured in the frequency range of 1-1000 Hz. The value of the conductance was converted to electrical conductivity using Equation (4)

$$\sigma = G l/A \tag{4}$$

where  $\sigma$  is the electrical conductivity (S/m), G is the conductance (S), l and A are thickness (m) and cross-section of the sample (m<sup>2</sup>), respectively.

#### 3. Result and discussion

The FTIR spectrum of the Na<sub>2</sub>FeSiO<sub>4</sub> sample shown in Figure 1. It shows the presence of several absorption peaks associated with a typical functional group contained in the Na<sub>2</sub>FeSiO<sub>4</sub> compound. The absorption peaks at wave numbers 972.12 cm<sup>-1</sup> and 879.54 cm<sup>-1</sup> are related to the vibration stretching of the Si-O group from SiO<sub>4</sub> tetrahedra [32]. Absorption peaks at wave numbers 640.37 cm<sup>-1</sup> and 509.21 cm<sup>-1</sup> are related to the vibration stretching of the Fe-O group of [FeO<sub>4</sub>] tetrahedra [33]. Meanwhile, the absorption peak at wave number 432.05 cm<sup>-1</sup> indicates the presence of vibration of the Na-O group of [NaO<sub>4</sub>] tetrahedra [34]. The appearance of the absorption peaks of the Si-O, Fe-O, and Na-O groups in the FTIR spectrum shows a strong indication that the Na<sub>2</sub>FeSiO<sub>4</sub> phase has formed in the sample.

**1572** (2020) 012003

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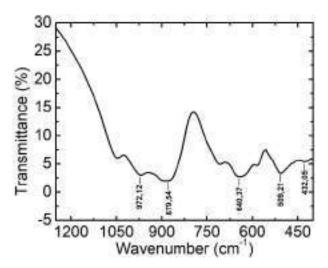


Figure 1. FTIR spectrum of a sample of Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica.

The diffractogram of the Na<sub>2</sub>FeSiO<sub>4</sub> sample shown in Figure 2. The phase analysis conducted by comparing the diffraction lines with the COD database using the search-match method. Referring to previous studies [17], because Na<sub>2</sub>FeSiO<sub>4</sub> is not yet available in the database, the analysis may use crystallographic databases of similar polyanion compounds, for example, Na<sub>2</sub>CaSiO<sub>4</sub>. The compounds have the same crystal structure. The characterization showed that the diffraction line from the Na<sub>2</sub>FeSiO<sub>4</sub> sample was in agreement with the database of Na<sub>2</sub>CaSiO<sub>4</sub> (COD 00-101-0111) which was marked by the presence of diffraction peaks at 20 of 16.82°; 20.47°; 29.36°; 33.72°; 48.47°; 60.38°; and 81.09° which correspond to the Miller index (110), (111), (211), (220, (412), (400), (422) and (602) respectively. According to studies conducted by Kee et al., (2016), the formation of the Na<sub>2</sub>FeSiO<sub>4</sub> phase was characterized by the presence of diffraction peaks with the Miller index [17]. The presence of a diffraction peak at  $2\theta$  of 33.72°, which is the peak with the highest intensity, confirms that the phase is the main phase in the sample. The establishment of this phase is in agreement with the results of FTIR analysis, which shows the presence typically functional groups of the Na<sub>2</sub>FeSiO<sub>4</sub> structure, such as Na-O, Fe-O, and Si-O [35]. Besides the crystalline phase Na<sub>2</sub>FeSiO<sub>4</sub>, the diffractogram also shows diffraction peaks, which indicate the presence of impurity phases such as Na<sub>2</sub>SiO<sub>3</sub> (COD 00-231-08580) and SiO<sub>2</sub> (COD 00-900-0520) as shown in Figure 2. The presence of the Na<sub>2</sub>SiO<sub>3</sub> phase is characterized by the appearance of the diffraction peak at 2θ of 29.65°, which is the main peak of the phase as well as several other diffraction peaks, as shown in Figure 2. Whereas, the presence of the SiO<sub>2</sub> phase is characterized by the appearance of the peak diffraction peak at 2θ of 20.50°. The formation of this impurity phase is predicted as a result of thermal energy given to the sample in the thermal treatment that does not sufficiently encourage the entire crystallization process to form the Na<sub>2</sub>FeSiO<sub>4</sub> phase.

**1572** (2020) 012003

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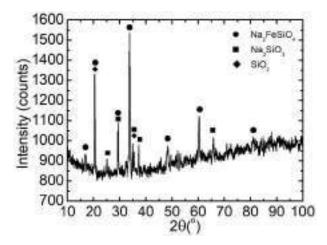


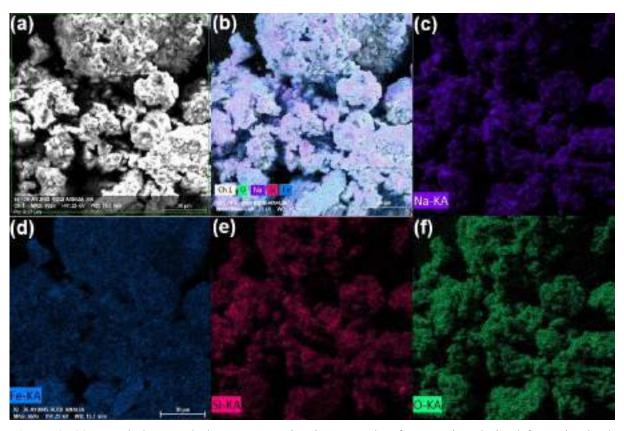
Figure 2. Diffractogram of a sample of Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica.

SEM analysis shows that the Na<sub>2</sub>FeSiO<sub>4</sub> sample is consists of micro-scale particles, as shown in Figure 3(a). The constituent elements of Na<sub>2</sub>FeSiO<sub>4</sub>, i.e., sodium (Na), iron (Fe), silicon (Si), and oxygen (O), appear to be evenly distributed on the sample surface as shown in Figure 3(b). The distribution of the elements sodium (Na), silicon (Si) and oxygen (O) on the surface are clearly shown in Figure 3(c)-(f). This mapping element reinforces the results of the phase analysis, which identifies the formation of the Na<sub>2</sub>FeSiO<sub>4</sub> phase, as well as the impurity phase. The results also confirm that silica from the husk is very potential to be used as a raw material in the synthesis of Na<sub>2</sub>FeSiO<sub>4</sub>.

The band gap energy value of the Na<sub>2</sub>FeSiO<sub>4</sub> sample is estimated using the Tauc plot, as shown in Figure 4(a). The Tauc plot has two dominant slopes that intersect the x-axis at 2.58 eV and 2.87 eV. It means that the sample has two different band gaps, i.e., 2.58 eV and 2.87 eV. The presence of two band gap values in the sample is due to the presence of the impurity phase in the sample [36]. This situation is consistent with the results of the phase analysis, which shows the presence of the impurity phases. The band gap energy value estimated from the Tauc plot is low. A high electrical conductivity value follows the low band gap energy value. The electrical conductivity value in frequencies 1-1000 Hz and shown in Figure 4(b). The electrical conductivity of the sample varies with frequency, at a frequency of 1 Hz is 6.13x10<sup>-5</sup> S/m, and then decreases gradually to 4.27x10<sup>-5</sup> S/m at a frequency of 1000 Hz. In general, the electrical conductivity of the Na<sub>2</sub>FeSiO<sub>4</sub> sample along the measurement frequency has a much higher value than Na<sub>2</sub>FeSiO<sub>4</sub>, which is only in the order of ~10<sup>-12</sup> S/m [37]. By considering the characteristics of the electrical properties obtained, it shows that the Na<sub>2</sub>FeSiO<sub>4</sub> samples prepared from rice husk silica have a high potential for use as a cathode.

**1572** (2020) 012003

doi:10.1088/1742-6596/1572/1/012003



**Figure 3.** (a) Morphology and elements mapping in a sample of Na<sub>2</sub>FeSiO<sub>4</sub> derived from rice husk silica, (b) element distribution, (c) Na, (d) Fe, (e) Si, and (f) O distribution.

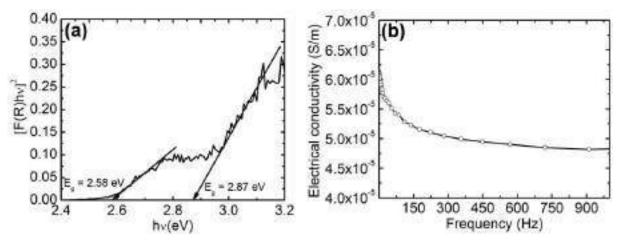


Figure 4. (a) Tauc plot of band gap energy, and (b) Electrical conductivity of a sample of Na<sub>2</sub>FeSiO<sub>4</sub>.

#### 4. Conclusions

Silica from rice husk is very potential to be used as a raw material in the production of Na<sub>2</sub>FeSiO<sub>4</sub>, although phase analysis shows the presence of impurities that accompany the primary phase. The formation of the Na<sub>2</sub>FeSiO<sub>4</sub> phase is supported by the FTIR analysis, which indicates the presence of functional groups that are typical of Na<sub>2</sub>FeSiO<sub>4</sub>, i.e., Na-O, Fe-O, and Si-O groups from the tetrahedra side. The mapping of the elements also strengthens the formation of this phase. The mapping elements

**1572** (2020) 012003

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show that Na, Fe, Si, and O homogeneously distributed on the surface of the sample. Tauc plot indicates that the Na<sub>2</sub>FeSiO<sub>4</sub> has a band gap value of around 2.58-2.87 eV and electrical conductivity 6.13x10<sup>-5</sup> S/m at 1 Hz and decreases gradually up to 4.27x10<sup>-5</sup> S/m at 1000 Hz. This value is far higher than the electrical conductivity of Li<sub>2</sub>FeSiO<sub>4</sub>. From the electrical conductivity, Na<sub>2</sub>FeSiO<sub>4</sub> prepared from rice husk silica has excellent potential for use as a cathode material.

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