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Production of Ceramic Membrane Based on Acid- and Alkali-Activated Metakaolinite as Cooling Material for Monocrystalline Silicon Solar Cell

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Abstract

Ceramic membrane based on acid- and alkali-activated metakaolinite has been produced. It was tested as a cooling material for monocrystalline silicon solar cells. The membrane was made by several stages, such as calcination of natural kaolinite at 600 °C for 6 hours to obtain metakaolinite, activation of metakaolinite by concentrated HCl and KOH, and preparation of ceramic membrane. Kaolinite, metakaolinite, and activated metakaolinite were characterized by X-ray diffraction (XRD), X-ray fluorescence (XRF), Fourier Transform Infra-red (FTIR), and gas sorption analyzer (GSA). Diffractogram of XRD showed that there was a structural change between activated metakaolinite and natural kaolinite. XRF analysis indicated that the Si/Al of HCl activated metakaolinite was three times higher than natural kaolinite. Activated metakaolinite was made into the membrane by adding a binder, then heated at 800 °C for 6 hours. Photovoltaic (PV) cells with and without cooling material were then analyzed their electrical performances. It was found that the maximum energy conversion yield of PV cells without using cooling material was 2.30%, while the maximum energy conversion yield of PV cells with cooling material of meta-kaolinite activated by HCl and KOH were 2.72% and 2.94%, respectively.

***Keywords**:* Ceramic membrane, cooling material, metakaolinite, photovoltaic.

Abstrak

Membran keramik dari metakaolinit teraktivasi asam dan basa telah berhasil diproduksi. Membran keramik tersebut digunakan sebagai material pendingin pada sel surya berbasis silikon. Membran dibuat melalui beberapa tahapan, di antaranya kalsinasi kaolinit alam pada 600 °C selama 6 jam untuk menghasilkan metakaolinit, aktivasi metakaolinit menggunakan larutan HCl dan KOH pekat, dan pembuatan membran keramik. Kaolinit, metakaolinit, dan metakaolinit teraktivasi terlebih dahulu dikarakterisasi menggunakan difraktometer sinar-x (XRD), fluoresensi sinar-x (XRF), spektrofotometer infra merah (FTIR), dan analisis serapan gas (GSA). Hasil analisis XRD menunjukkan telah terjadi perubahan struktur pada metakaolinit teraktivasi dari kaolinit alam. Hasil analisis XRF mengindikasikan peningkatan nilai rasio Si/Al metakaolinit teraktivasi asam tiga kali lebih besar daripada kaolinit alam. Metakaolinit teraktivasi kemudian dibuat menjadi membran dengan penambahan bahan pengikat, dan dipanaskan pada 800 °C selama 6 jam. Sel fotovoltaik (PV) dengan dan tanpa menggunakan membran kemudian dianalisis kinerjanya. Berdasarkan hasil pengukuran kinerja, rendemen konversi energi maksimum pada intensitas cahaya 100 Watt/m^2 yang dihasilkan oleh sel PV tanpa menggunakan material pendingin adalah 2.30%, sedangkan sel PV yang menggunakan material pendingin dari metakaolinit teraktivasi asam dan basa, masing-masing adalah 2.72% dan 2.94%.

Kata Kunci: fotovoltaik, material pendingin, membran keramik, metakaolinit.

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1. INTRODUCTION

Electricity has become an important part of modern society. Almost all the human activities are running because of electricity. However, at the national level (in Indonesia), the primary energy sources for electricity production are currently fossil fuels (Kholiq, 2015). This has resulted in a growing depletion of fossil fuel reserves, rising the price of electricity per kWh (kilowatt-hour), and also participating in carbon dioxide (CO_2) emissions. In order to overcome these issues, in 2050 the Government targeting for using alternative energy to 31% of national consumption (National Energy Board, 2016).

Indonesia, particularly Pontianak-Kalimantan Barat (West Borneo) is located at 0 degrees of latitude or crossed by Equator line that receives an average of 1800-2000 kWh/m^2 of sunlight per year. Based on that data, solar energy is one of clean alternative energy sources that could potentially develop especially in Indonesia and generally in the world (Ministry of Energy and Mineral Resources of the Republic of Indonesia, 2016).

One of the electrical devices that converts the solar (light) energy into electricity without CO_2 emissions is photovoltaic (PV) cell. Crystalline silicon is the most dominant material used in PV cells, in the form of a thin layer of polycrystalline silicon (poly-Si) or monocrystalline silicon (mono-Si). However, monocrystalline PV cells can convert solar energy into electrical energy with higher efficiency (up to 15% at 25 ^oC) than polycrystalline PV cells (around 13% at 25 ^oC) (Akbarzadeh and Wadowski, 1996; De Mey *et al.*, 2013).

Moreover, Akbarzadeh and Wadowski (1996) and De Mey *et al.*, (2013) have been reported that an increase in the temperature of PV cells will lead to a decrease in electrical efficiency. The efficiency decreases to 12-15% at a temperature of 50 ^oC. Therefore, the cooling techniques for PV cells are needed.

One of the cooling techniques is passive cooling that has been reported by Alami (2015) using the cooling material made from synthetic clays, a well-known porous material. Those pores can help the process of evaporating heat transfer from the cell through the pore network that leads to an increase in electrical energy of PV cells around 34.6% compared to PV cells which is not given by cooling material.

In this study, modified clays material was used as a cooling material for PV cells. Natural clays modified into metakaolinite by acid and alkali activation separately. The selection of kaolinite is based on its abundance in Indonesia, especially in West Borneo. In 2016, kaolinite deposits were reported in Sintang district with 7 million tonnes, in Kapuas Hulu district with 32 million tonnes, and Bengkayang district with 7 million tonnes (Ministry of Energy and Mineral Resources of the Republic of Indonesia, 2016).

Furthermore, Law Number 4 of 2009 about Mineral and Coal Mining mandated that mining commodities are compulsory to be processed domestically before being exported. This law also supported by Minister of Energy and Mineral Resources Regulation Number 7 of 2012 about an increase in activity for adding the minerals value which leads to a reduction in export trading of raw material of kaolinite. This resulted in making more processed things from this mineral in the country because its utilization has not been optimal yet (Ministry of Trade, 2013).

Kaolinite ($Al_2Si_2O_5(OH)_4$) is the main component of kaolin or clay which has been widely studied. It is a 1:1 clay mineral which builds up by octahedral layers of $Al(O, OH)_6$ and tetrahedral layers of SiO_4 with diameter of 0.2 to 10 μm, thickness 0.7 nm, a range of interlayer 7.16 Å, and density 2.6 g/cm^3. Kaolinite also reported build-up from pseudohexagonal triclinic crystals. Kaolin from West Borneo was reported to contain quartz, kaolinite, and illite with whiteness value of 87.74 and cation exchange rate of 38.74 meq/100 g (Varga, 2007; Lusiana and Cahyanto, 2014).

The metastable phase of kaolinite is metakaolinite which formed using a high temperature around 650-900 ^oC. During the heat treatment, or calcination, the structure of kaolinite is broken down. Thus it is much more reactive to acid and alkali activation than kaolinite (Justice *et al.*, 2005). Belver et al., (2002) reported the solids obtained by acid activation are promising adsorbents and catalyst supports, while in alkali activation may be a good method for preparing K-F zeolite. Besides, both activation reported an increase in specific surface area. The formation reaction of metakaolinite presented in Figure 1 below.

$$\mathbf{Al_2O_3(SiO_2)_2(H_2O)_2\ (kaolinite) \rightarrow Al_2O_3(SiO_2)_2(H_2O)_x + (2-x)H_2O\uparrow}$$

Figure 1. Formation reaction of metakaolinite

Referring to the research of Belver et al. (2002), in this study, metakaolinite was activated using hydrochloric acid (HCl) for acid activation, while the alkali activation was using potassium hydroxide (KOH). The cooling performance was conducted by comparing the electrical efficiency of PV cells with and without cooling the material in order to know the best cooling material using modified clays.

2. MATERIALS AND METHODS

The instruments used in this study were hotplate, magnetic stirrer, stirrer bar, X-Ray Diffractometer (PANalytical XPERT POWDERT PW60/40), X-Ray Fluorescence Spectrophotometer (Analytical), Fourier Transform Infra-Red Spectrophotometer (Perkin Elmer Frontier), Gas Sorption Analyzer (Quantachrome Instruments version 10.01), and standard laboratory glassware. Then, for the materials used in this study were distilled water (H_2O), hydrochloric acid (HCl MERCK), potassium hydroxide (KOH MERCK), kaolin, and starch (STARCH SOLUBLE MERCK).

Preparation of Kaolinite

The natural kaolin sample was procured commercially from an online marketplace in Indonesia (Tokopedia). The kaolin (200 g) was crushed into a fine powder, then screened using a 200-mesh sieve, and finally characterized using XRD to determine its mineralogy, using XRF to determine its chemical composition, and using FTIR to identify its chemical functional groups.

Preparation and Activation Metakaolinite

The preparation and activation of metakaolinite were adopted from Belver et al. (2002). The prepared natural kaolin was calcinated at a temperature of 600 ^{o}C for 6 hours. After thermal treatment, the metakaolin which contains metakaolinite was left to cool down until a room temperature was reached. and weighed as much as 50 g, then activated with 500 ml 6 M HCl solution and stirred. The mixture was then refluxed at 90 ^{o}C for 6 hours. Next, the suspension was allowed to stand, and the solids were washed with distilled water until its pH became neutral. Titration was used to determine the pH of Ag^+ solution, if there is any sediment still formed in suspension, then the neutralization would be retaken. After the suspension became neutral, the solids were dried at 103 ^{o}C for 4 hours. The same procedures were performed for alkali activation using 5 M KOH (except the testing with Ag^+). Finally, acid- and alkali-activated metakaolinite were characterized by XRD, XRF, and FTIR analysis to determine their properties. Activated metakaolinite using HCl then called MKA-HCl, while activated metakaolinite using KOH called MKA-KOH.

Production of Ceramic Membrane Based on Activated Metakaolinite (Ma'ruf *et al.*, 2015; Bouzerara *et al.*, 2009)

The Production of the ceramic membrane was made by modifying the method of Ma'ruf *et al.*, (2015) and Bouzerara *et al.*, (2009). A total of 20 g for each MKA-HCl and MKA-KOH were separately inserted into a beaker glass. Then, 2 g of starch as a binder and 20 ml of distilled water were added. Next, the mixture was stirred until homogenous and printed above the mold to form a rectangle with the size 12 × 7 cm and with a thickness of 3 mm. After that, the mixture was heated at a temperature of 800 ^{o}C for 6 hours. The resulting ceramic membranes were then analyzed using the GSA.

Electrical Measurements

PV cells used in this study were monocrystalline silicon PV cells with size 11 × 6 cm, which connected by a jumper cable on the positive and negative pole with the help of soldering iron. The resulting ceramic membrane was placed under the PV cells, arranged as shown in Figure 2 with the PV test criteria listed in Table 1. The light source used in this study was a 1000 watt halogen lamp as a substitute for sunlight whose intensity was set using the dimer from 0 to 500 watts/m^2. The electrical properties that measured were electric current (I), voltage (V), and Power (P) produced by PV cells when irradiating regularly. The measurements were using a 34461 Å scientific 6.5 digit Agilent multimeter.

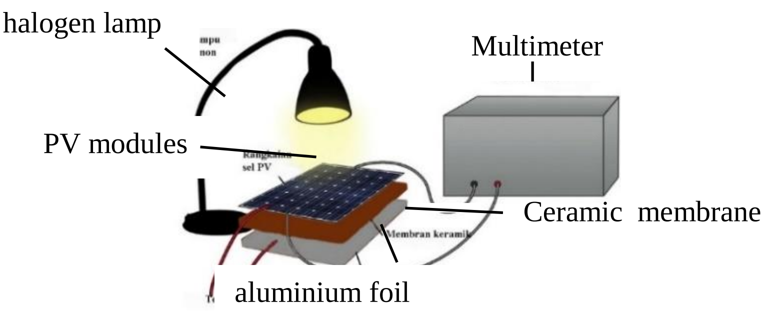

Figure 2. PV cells experimental test series (Alami, 2015)

Table 1. PV Cells Criteria

PV Cells	Cooling Material (Base)
PV Cells A	Ceramic membrane made of MKA-HCl
PV Cells B	Ceramic membrane made of MKA-KOH
PV Cells C	Aluminium Foil

3. RESULTS AND DISCUSSION

Characterization of Kaolinite, Metakaolinite, MKA-HCl, and MKA-KOH

Kaolinite is a relatively inactive clay mineral due to their high passivity and crystallinity. The improvement of the properties of kaolinite by chemical methods such as using strong acid and alkali have not been able to give chemical structure changes significantly (Rahmalia *et al.*, 2018). Therefore, in this study, before activation using HCl and KOH, kaolinite was firstly transformed into metakaolinite by calcination. Next, the resulting metakaolinite was activated by HCl and KOH, then analyzed with XRD, XRF, and FTIR to determine its properties.

Diffractogram analysis (Figure 3.1) showed mineral clays in this study was largely drafted by kaolinite. It was showed by the value of the reflection on the 2θ = 12.33; 19.82; 20.31; 21.18; 24.85 dan 35.89^o. After the calcination, the crystal structure of kaolinite becomes amorphous, it was indicated by widening the summit of reflection of kaolinite (Figure 3.2) which means the kaolinite became metakaolinit. This showed the kaolin has the form layers due to the bonding interactions between layers that are not too strong. Then the ties between layers of kaolinit were going to be broken with a heating process , and the peaks of kaolin before the calcination will be lost as a result of defective structure of kaolinite and formed metakaolinit with amorphous phase (Sunardi *et al.*, 2011). Figure 3 shows that there is no significant difference between the diffractogram of metakaolinit before and after activation, neither with HCl nor KOH. However, quartz was still shown by reflection 2θ = 26.58^o due to the solid quartz has high durability of acid or alkali treatment (Sunardi *et al.*, 2011).

Then, FTIR analysis was performed to see the changes that occured in the functional groups of kaolinite, metakaolinite, MK-KOH, and MKA-HCl. The results are presented in Figure 4.

Based on FTIR analysis, kaolinite shows the characteristic absorption bands at 3677 and 3635 cm^{-1} that are related to the vibrational strain of the surface -OH group; 1001 cm^{-1} associated with strain Si-O group relaxation; 917 cm^{-1} related to Al-OH deformation; 768 and 680 cm^{-1} related to the perpendicular vibrations of Si-OH. After calcination, the peak of spectra at 3635 cm^{-1} is not observed, indicating that there had been a reduction in water content due to calcination of kaolinite to metakaolinite. The heating process at high temperatures can change the chemical and physical properties of the kaolinite solid structure. The MKA-HCl and MKA-KOH FTIR spectra showed differences with the FTIR metakaolinite spectra, especially with the appearance of the widening spectra peaks from between 3400-3000 cm^{-1}, indicating the existence of -OH group vibration from water molecules after activation. The FTIR spectra of MKA-HCl clearly shows a significant decrease in peak intensity at 917 cm^{-1}, which indicates a reduction in the amount of Al-OH in the kaolinite and metacolinolinite structures. Whereas in the FTIR spectra of MK-KOH, there is a shift in the peak at 966 cm^{-1}, which corresponded to the -OH group on the surface. This shows that activation with KOH increase the presence of the -OH group in metakaolinite.

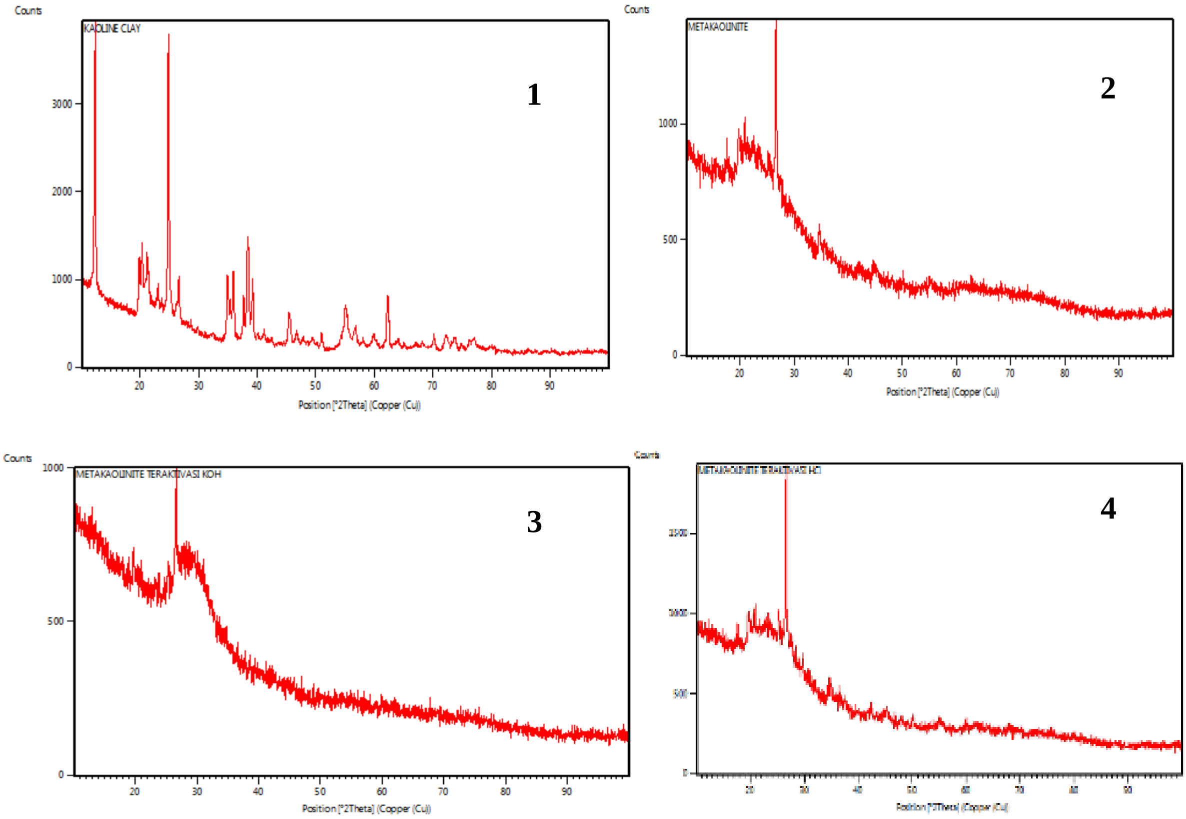

Figure 3. X-Ray diffracyograms of natural kaolinite (KA) (1), metakaolinite (MKA) (2), activated metakaolinite using KOH (MKA-KOH) (3), activated metakaolinite using HCl (MKA-HCl) (4)

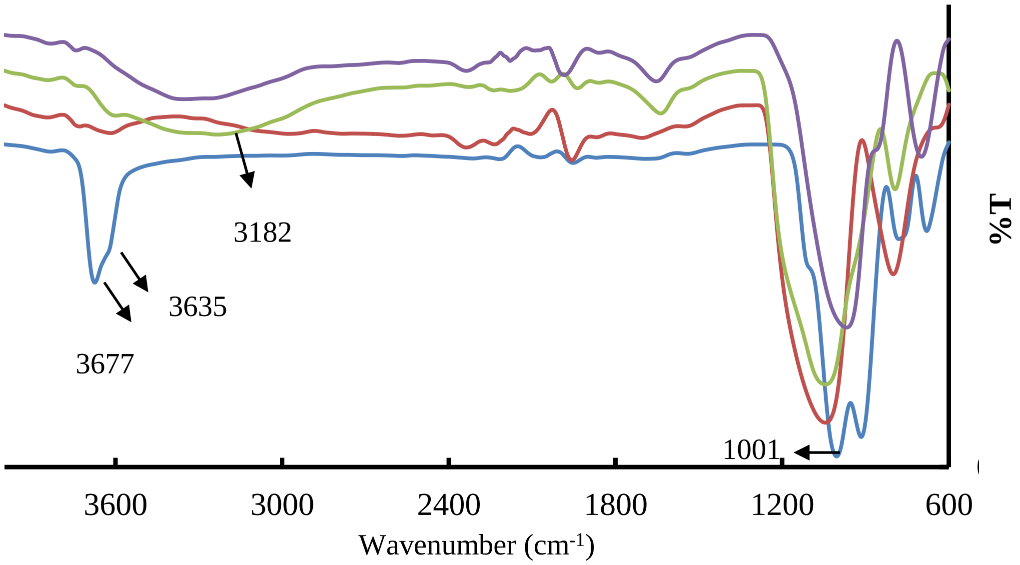

Figure 4. FTIR spectra of Kaolinite (blue), metakaolinite (orange), MKA-KOH (yellow), and MKA-HCl (gray)

Information about the main components of kaolinite, metakaolinite, MKA-HCl and MKA-KOH materials was confirmed by the XRF analysis results presented in Table 2. Table 2 shows that kaolin has layers composed of Al^{3+} and Si^{4+} cations, and contains several other cations such as Mg^{2+}, K^{+}, Ca^{2+}, Ti^{4+}, and Fe^{3+}.

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The main components of the mineral kaolinite are silica (SiO_2) and aluminum oxide (Al_2O_3), with a Si/Al ratio of 1.165. The value of Si/Al ratio > 1 is most likely due to the presence of quartz minerals originating from initial kaolinite samples. As defined by Jorge, *et al.* (2003) that kaolinite consisted of one layer of alumina which formed an octahedral structure and one layer of silica which formed a tetrahedral structure. This layer binds with oxygen atoms and the repetitive layer of minerals binds with hydrogen.

The calcination process caused a decrease in the Si/Al ratio due to damage to the kaolinite structure. However, the process of activation of acids and alkalines in metakaolinite caused an increase in the Si/Al ratio. A significant increase in the Si/Al ratio even occurred when metakaolinite was activated using HCl, being 3 times higher than the Si/Al ratio in kaolinite. This was predicted because HCl is a strong acid which can reduce the Al^{3+} content in Al_2O_3 through the dealumination process.

Table 2. Chemical compositions of kaolinite, metakaolinite, MKA-HCl, and MKA-KOH

Chemical Composition	Kaolinite (%)	Metakaolinite (%)	MKA-HCI (%)	MKA-KOH (%)
MgO	0.374	0.000	1.170	0.032
Al_2O_3	44.92	47.33	23.17	39.94
SiO_2	52.34	50.80	73.50	47.69
P_2O_5	0.507	0.406	0.156	0.376
K_2O	0.812	0.661	0.903	10.98
CaO	0.150	0.131	0.087	0.142
TiO_2	0.283	0.232	0.444	0.229
V_2O_5	0.005	0.005	0.007	0.002
Cr_2O_3	0.003	0.002	0.003	0.002
MnO	0.003	0.002	0.003	0.003
Fe_2O_3	0.504	0.387	0.512	0.354
ZrO2	0.011	0.009	0.014	0.010
Si/Al	1.165	1.073	3.171	1.194

The octahedral layer Al_2O_3 in kaolinite has lower stability than the tetrahedral SiO_2 layer, so that the octahedral cation can be removed through the hydrolysis process with acids and alkalines. The release or reduction of the octahedral cation will open the kaolin structure, so that the surface area and pore size will be greater (Wahyuni *et al.*, 2008). The greater surface area and pore size of the clay material was predicted to increase its performance as a cooling material in PV cells.

PV Cell Performance

Determination of the performance of PV cells with and without the addition of cooling material is done by measuring the current and voltage produced by the PV cell at the variation of the given light intensity. The measurement results are presented in Figure 5.

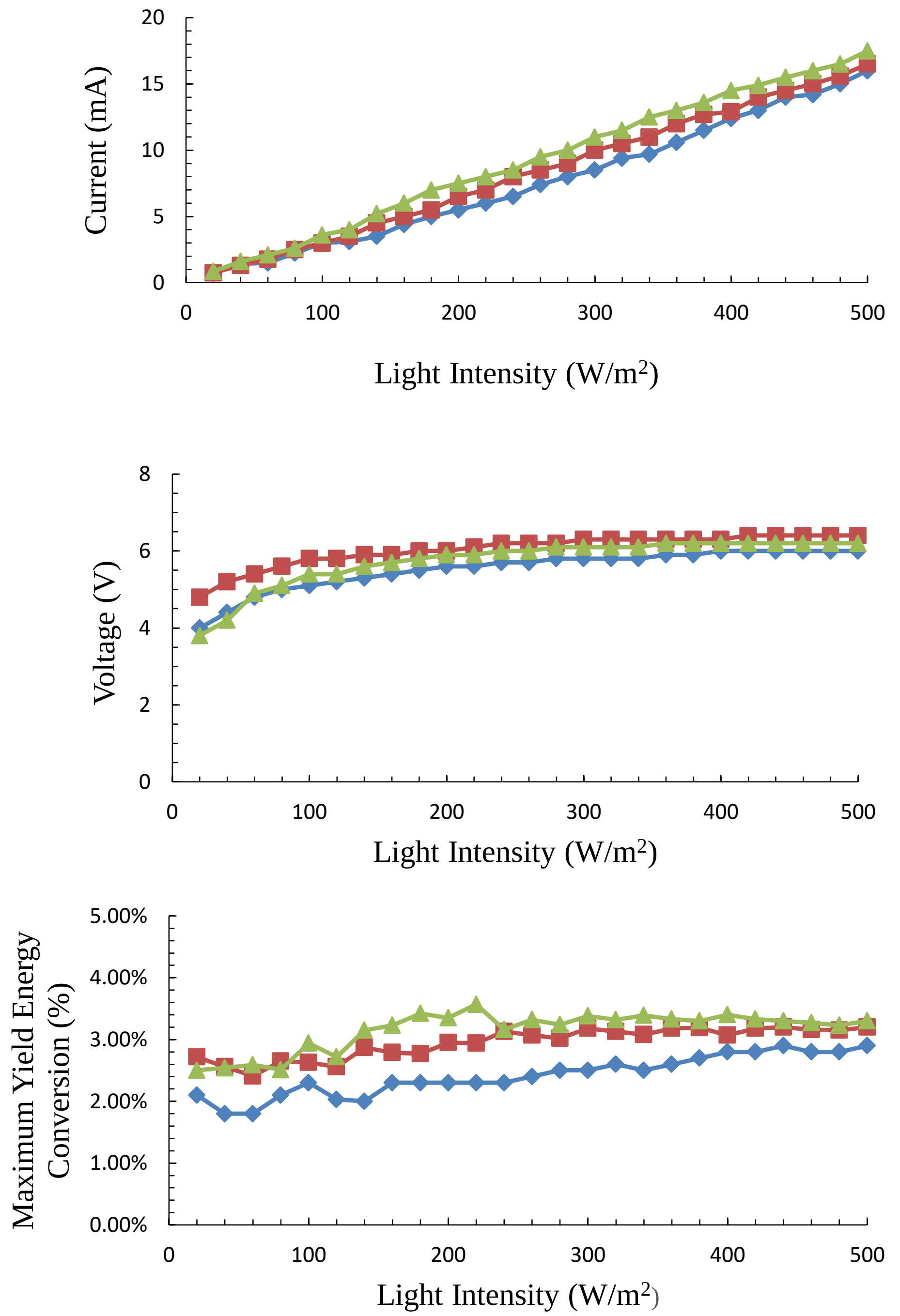

Figure 5. Performance measurment results of PV cells A (orange), PV cells B (gray), and PV cells C (blue)

Table 3. Performance measurement of PV cells A, PV cells B, and PV cells C at 100 Watt/m^2

Material	Maximum Voltage (V)	Maximum Current (mA)	Maximum Yield Energy Conversion (%)
PV cell A	3.00	5.80	2.72
PV cell B	3.60	5.40	2.94
PV cell C	3.00	5.10	2.30

Table 4. Analysis of isoterm adsorption at 77 K

Material	Surface Area (m^2/g)	Pores Volume (cc/g)	Pores Radius (Å)
Membrane-MKA HCl	25.461	0.121	33.676
Membrane-MKA KOH	11.492	0.066	33.667

In general, for all three cells, the greater the intensity of the light given, the higher the current generated by the PV cell. This is because more and more photons are absorbed by PV cells as light intensity increases. However, PV B cells and PV C cells show relatively higher currents compared to PV A cells at an increase in light intensity above 80 Watt/m^2. The increase in light intensity given to the cell is accompanied by an increase in lamp temperature, thus proving that the addition of cooling material in the form of ceramic membranes from MKA-HCl and MKA-KOH can inhibit performance degradation due to increased temperatures. In this case the membrane functions as a cooling cell, so that the temperature of PV A cells and PV B cells are not higher than PV C cells.

The greater the intensity of the light given, the greater the value of the voltage produced by each cell. This phenomenon occurs up to a light intensity of 100 Watt/m^2. Beyond this intensity, the voltage generated by each cell is relatively unchanged. The voltage produced by PV A cells is relatively higher than the voltage generated by PV B cells and PV C cells. The optimum voltage generated by each cell when given a light intensity of 100 Watt/m^2, which is equal to 5.80 V; 5.40 V; 5.10 V each for PV A cells, PV B cells, and PV C cells. The intensity of the light used is 100 Watt/m^2, this is because monocrystalline and polycrystalline solar cells have a power capability of 100 Watt (Tascioglu *et al.*, 2016).

The higher the current and voltage values, the higher the power generated. As power (watts) = I (A) x V (V). The variation of light intensity given has a great influence on the maximum energy conversion yield produced by PV cells, where PV cells A produce a maximum energy conversion yield of 3.20% at 440 Watt/m^2 light intensity, PV cells B of 3.57% at 220 Watt/m^2, and PV cells C at 2.90% at 440 Watt/m^2. Whereas at the intensity of 100 Watt/m^2, the maximum energy conversion rate by PV cells A, PV cells B and PV cells C was 2.72 respectively; 2.94; and 2.30%. In order to study the cooling mechanism by which the ceramic membrane made can function as a cooling material by utilizing pores, a BET analysis is performed to see the pore size and volume of the ceramic membrane. BET analysis results are presented in Table 4. Although based on BET analysis, the membrane of MKA-HCl has a greater surface area and pore volume than the membrane of MKA-KOH, but the PV cells B showed the highest maximum energy conversion rate. This is probably due to the activation of KOH adding to the -OH group on the metakaolinite surface, in accordance with the results of the FTIR analysis, thereby increasing the adsorption properties of MK-KOH.

4. CONCLUSION

Acid- and alkali-activated metakaolinite have been synthesized for membrane building which can be applied as a cooling material in PV cells. In this study, the pore does not really affect its function as a cooling material, but rather the addition of the functional group –OH which increases the adsorption ability of the membrane. PV cells with cooling material have an increased percentage of maximum energy conversion of 18.8% compared to PV cells without cooling material.

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