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Effect of Calcination Temperature and Steaming Time on Activation of Freundlich Isotherm Practicum Waste in Analytical Chemistry Laboratory

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Abstract: *This study aims to determine the effect of calcination temperature and steaming time on activation of Freundlich isotherm practicum waste (FIPW) in the Analytical Chemistry Laboratory. The FIPW was taken from the waste of the Physical Chemistry Practicum at the Analytical Chemistry Laboratory, Chemistry Department, Ganesha University of Education. To achieve the research objectives, the FIPW was calcined at 165°C and 195°C, then steamed with 900 mL of distilled water at 400°C for 60 minutes and 45 minutes, respectively. The results of FIPW activation were characterized by absorption, increasing absorption, FTIR spectrum, and SEM morphology. The results of the study showed that there were influences of calcination temperature and steaming time on FIPW in the Analytical Chemistry Laboratory. The best temperature of calcination and steaming time for activation of FIPW was calcination temperature of 195°C and steaming for 45 minutes.*

Keywords*: activation, freundlich isotherm, calcination, steaming, practicum waste*

INTRODUCTION

Learning chemistry was related very closely to practicum. Chemistry practicum is very important because it can strengthen the theory that has been owned by students. Practicum can develop process skills, motor skills, and the formation of scientific attitudes. Scientific attitudes that can be formed through chemical practicum include: conducting research, research, and studying nature in more depth (Hudha, 2002: 2). Besides that, chemical practicum can also increase interaction and between groups so that they can appreciate the work of others. Thus, chemical practicum can train emotional, social, and spiritual intelligence (Mursid, R., 2013: 27). All of these chemical practicum impacts can be achieved if supported by laboratory facilities and infrastructure.

Practicum material is a part of laboratory facilities and infrastructures. Chemical laboratory materials are classified into general materials and special materials. Activated carbon is one of the chemical practices material which is classified as a special material because activated carbon used in chemical practices has a purity level of more than 90%, storage must be waterproof so that activated carbon does not absorb water in the air. The impact of these activated carbon characteristics causes the price of activated carbon to become expensive (Peraturan Bersama Menpan dan Kepala BKN No. 02/V/PB.2010. No.13 Th. 2013 about Petunjuk Pelaksanaan Jabatan Fungsional PLP dan Angka Kreditnya).

The Freundlich isotherm practicum is one of the chemistry practicum titles in the Physics Chemistry Practicum course at the Chemistry Department, Ganesha University of Education using activated carbon. Freundlich isotherm practicum aims to verify adsorption

by Freundlich isotherm. Freundlich isotherm practicum uses activated charcoal as an adsorbent and oxalic acid as an adsorbate. The number of activated charcoal used 60 grams per group or 840 grams per semester. The total cost of using activated carbon is Rp. 6,300,000.00 every semester (I Ketut Lasia and I Ketut Budiada, 2017).

The residue of the Freundlich isotherm practicum is activated carbon waste contaminated with oxalic acid. The amount of waste collected from 2013 until 2018 year reached 4200 grams. This carbon waste is not utilized, so it has the potential become the place problem in the Analytical Chemistry Laboratory, Chemistry Department, Ganesha University of Education. On the other hand, the amount of activated carbon available at the Chemistry Laboratory is decreasing and not in accordance with the needs of the practicum. This was compounded by the reduction in operational aid funds for the laboratory. For this reason, efforts are needed to reactivate the Freundlich isotherm practicum waste (FIPW) to reactivate carbon (I Ketut Lasia and I Ketut Budiada, 2017).

Research about how to activate carbon has been done. Efforts to activate carbon can be done using K_2CO_3. The surface area of carbon using K_2CO_3 activators reaches 2000 m^2/g (P. Ponkarthikeyan and K. Sutha Sree, 2017). Other chemicals can be used for carbon activation are HNO_3, H_2O_2, H_2SO_4, ammonium peroxidisulfate, ammonia, and H_3PO_4 (S.M. Yakout and G. Sharaf El-Deen, 2016; Hyung Won Lee et al., 2018).

Carbon activation using chemicals has weaknesses. The disadvantage, it requires chemicals and a large amount of time and energy to remove chemical activators from carbon. If carbon contaminated with chemicals, that it reduces the quality of activated carbon produced (Byeong Ho Min and Kyeong Youl Jung, 2017, Hyung Won Lee et al., 2018). To overcome this problem, carbon activation is carried out by physical means. One of the physical ways to activate carbon is calcination and steaming. The effects of calcination temperature and steaming time on FIPW activation found in the Laboratory of Analytical Chemistry, Chemistry Department, Ganesha University of Education are examined in this paper.

RESEARCH METHODS

Materials and Tools

The material used in this study was FIPW obtained from the Analytical Chemistry Laboratory, Chemistry Department, Ganesha University of Education. Whereas other ingredients with pro analysis quality from Merck are aquabides, activated carbon, I_2, $Na_2S_2O_3$, and starch.

The equipment was used glassware, furnace Naberthem, ZHWY-304 shaker, pH meter PCSTestrtm 35 Oakton, UF160 oven, Shimadzu's IR Affinity-1 FT, and SEM type JEOL JSM 6510-LA.

Procedures

FIPW activation

FIPW was separated into a control group and an experimental group. The experimental group was calcined at 195^oC and 165^oC for 1 hour, respectively. The calcination results are steamed with 4 repetitions. Steaming is carried out with 900 mL of distilled water at a heater temperature of 400^oC. Steaming carried out for 60 minutes and 45 minutes. Every steaming, the pH of the remaining of water steaming is tested. The steamed sample is dried in 110^oC oven until its mass is constant.

Characterization of FIPW Activation Results

The results of FIPW activation are characterized by absorption, increased absorption, spectrum with FTIR, and morphology with SEM. The absorption characterization of the FIPW activation results was carried out by inserting 1 gram of the activation results into an Erlenmeyer containing 50 mL I_2 0.1N; then beaten 150 rpm for 60 minutes using a shaker and then filtered. The filtrate was titrated with sodium thiosulfate ($Na_2S_2O_3$) 0.1 N to light yellow color, then titration of 3 drops of 1% starch. Titration is stopped when the blue color has disappeared. Carbon absorption is determined by the ASTM method using iodine with the equation

$$\text{DSI} = \frac{(mL sample - \frac{TxC1}{C2})xWx5}{g sample}$$

In this case: DSI = iodine absorption (mg/g), mL sample = titrated filtrate (10 mL), T = titration volume $Na_2S_2O_3$ (mL), C1 = $Na_2S_2O_3$ concentration (N), C2 = concentration of Iodine (N), W = iodine weight (12,693 mg / mL), 5 = dilution factor.

Increased absorptive capacity uses equation: PDs = (DsFIPW-DsK) / DsK, in this case PDs: absorptive capacity, DsFIPW: absorptive capacity FIPW, DsK: absorptive control. Characterization FIPW by FTIR was performed at wavenumbers 500-4000 cm^{-1}. The spectrum produced from FTIR is compared with standard active carbon spectrum and inter-sample spectrum. While morphological of characterization of FIPW activation results were used SEM JEOL JSM 6510-LA. The morphology between samples is compared with each other.

RESULTS AND DISCUSSION

Activation of FIPW

The results of FIPW waste activation were seen visibly showing difference color, namely calcination of the temperature of 195°C was darker and cleaner than calcination of 165°C. The results of FIPW activation are presented in Figure 1.

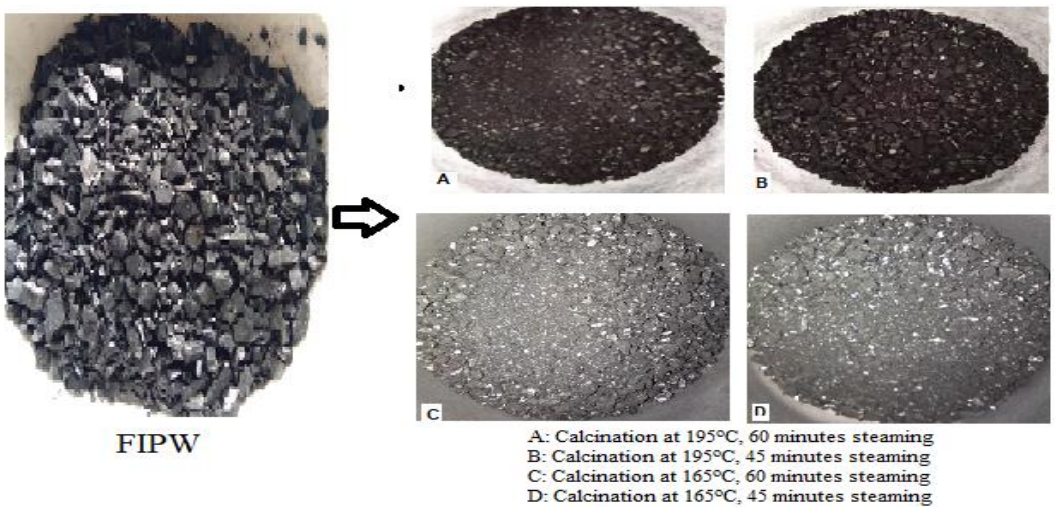

Figure 1. FIPW activation results

The whiteness of 165°C calcination shows that impurities are still present in FIPW. While in calcination 195°C shows oxalic impurities have been reduced.

The mass difference resulting from FIPW activation by calcination at 165°C and 195°C for 1 hour is presented in Figure 2. Figure 2 shows the oxalic acid found in carbon decomposed. Badawari (1989) reported the decomposition temperature of oxalic acid was 189.5°C. Oxalate ions also decompose into CO_3^+ ions at that temperature (Yu. O. Lagunova et al., 2012) and oxalic acid experiences the following reactions: $H_2C_2O_4$ -----> H_2CO_3 + H_2O, or CO_2 + H_2O. The biggest difference in carbon mass occurs in calcination of 195°C because at that temperature, oxalic acid contained in carbon decomposes more.

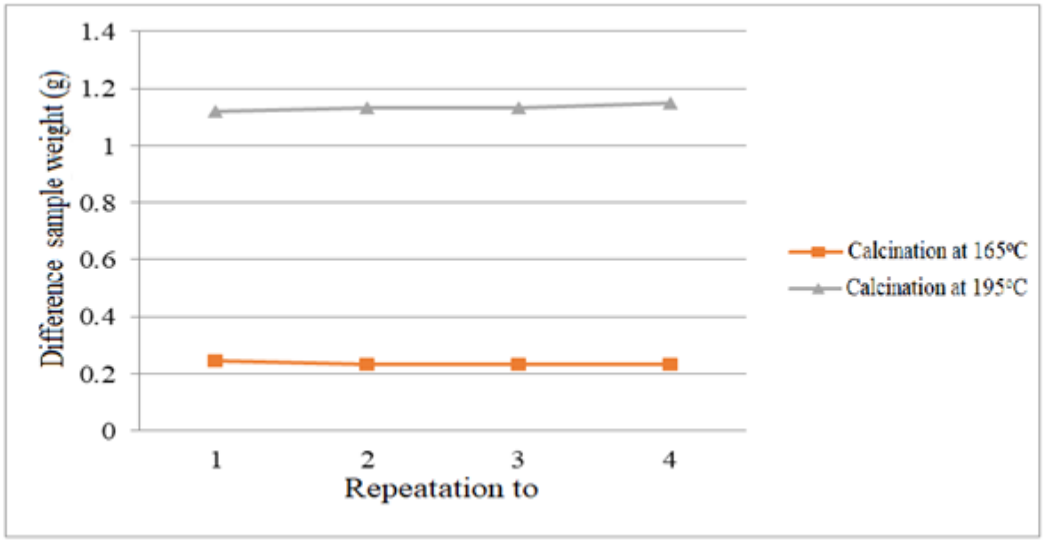

Figure 2. Difference in weight of sample at calcination for 1 hour

The release of decomposed oxalic acid which is still present in carbon when calcination is continued by steaming. Steaming aims to remove the adsorbate left in carbon. The acidity (pH) of the FIPW carbon steaming after calcination is presented in Figure 3.

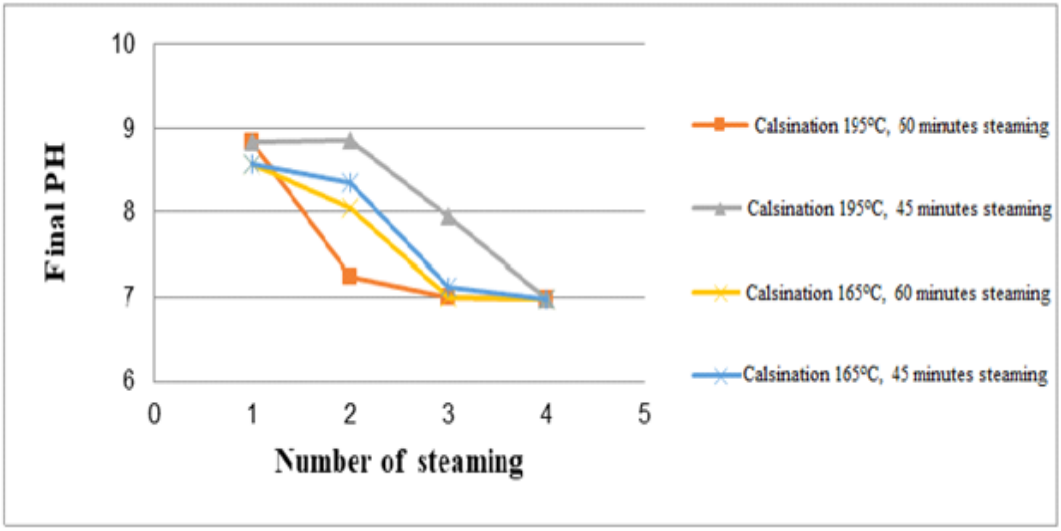

Figure 3. Effect of calcination and steaming on pH

Steaming with variations of time 45 minutes and 60 minutes repeatedly, showing an increase in pH. The increase in pH occurs because the amount of oxalic acid trapped in the FIPW carbon decreases along with repeated steaming. The steaming system carried out on the FIPW calcination is adopting an extraction system. According to the extraction theory applied by Andreas O. Wagner et al. (2015) which states the more repetition of extraction, the more separated of substances. Figure 3 shows the solubility of oxalic acid getting higher with increasing temperature. Oxalic acid when it has an ionic reaction: $H_2C_2O_4 \rightleftharpoons 2H^+ + C_2O_4^=$ (William Blum, 2017). The separation of oxalic acid from charcoal was shown from the decrease in the pH of the steaming water to the pH of distilled water, which was 6.98.

Characteristics of FIPW

The characteristics of FIPW in terms of absorption, increase in absorption, FTIR spectrum, and morphology. Absorption and increasing absorption of FIPW which has been activated shows that calcination at a temperature of 195°C and steaming for 45 minutes produces the best absorption, which is 164.37 mg/g or 49.31% against I_2 and the highest increase in absorption from 93.37%. This shows the calcination temperature and steaming time have an impact on the absorption and efficiency of carbon absorption. When calcination at 195°C and steaming for 45 minutes, the carbon porosity of the FIPW becomes more open because the amount of oxalic acid covering the carbon shaft decreases. This research is in line with what was revealed by Taza Gul et al. (2018), that the increase in thermal energy causes the reduction of clogging material in the porous material. This can also be seen from the carbon morphology such as Figure 6 and the carbon spectrum of Figure 5e which is the closest to the standard carbon spectrum (Figure 5f). Thus, FIPW absorption at calcination of 195°C and steaming for 45 minutes produced the highest absorption. The Comparison of absorption and increase in absorption of activated FIPW is shown in Figure 4.

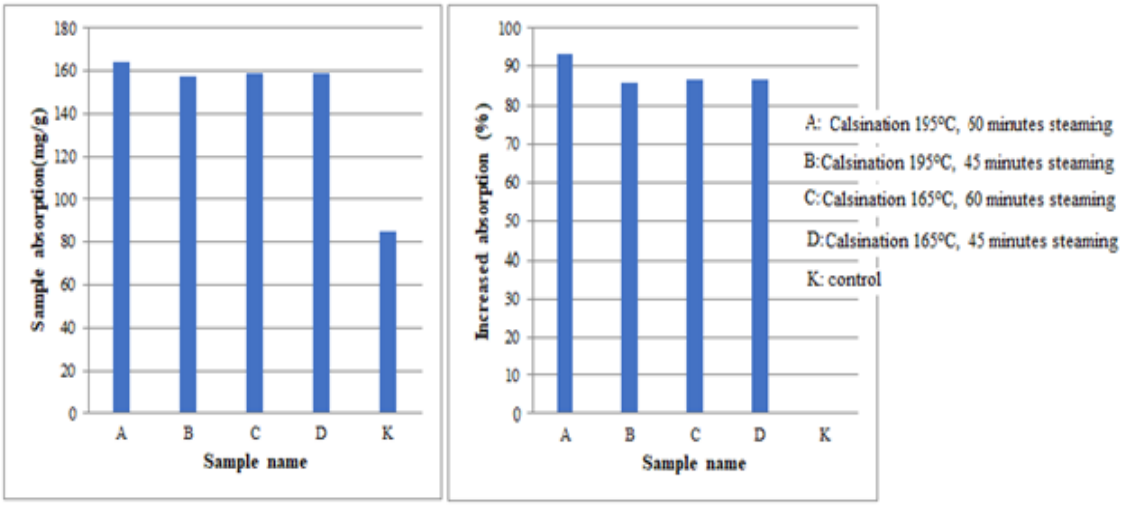

Figure 4. Absorption and increasing absorption after FIPW is activated

FIPW activation has a maximum absorption of 49.31% of iodine. Carbon absorption by steaming-calcination technique is higher than carbon absorption at 550°C heating for 2

hours, which is 21.88% (Sani, 2011). In addition, calcination of temperature of 195°C and steaming for 45 minutes showed the most effective way to activate the Freundlich isotherm practicum waste, because of the low heating temperature and short time, the less electrical energy used and the cost of producing activated carbon from the Freundlich isotherm practicum waste decreased.

The quality of activated FIPW was tested by FTIR also. Testing with FTIR to determine the impurities by the oxalic acid remaining in FIPW. The test results with FTIR are presented in Figure 5. Based on Figure 5, there is a difference in the FIPW spectrum due to calcination and steaming. The FIPW spectrum before calcination and steamed (Figure 4.a) and FIPW calcined at 165°C and steamed 45 minutes (Figure 5.c) shows a wavenumber of 1680 cm^{-1}. Whereas in Figure 5.b, Figure 5.d, and Figure 5.e wave number 1680 cm^{-1} does not appear in the spectrum. Wavenumber 1680 cm^{-1} shows the carboxyl group (C = O) experiencing stretching and vibration (J. H. You et al., 1994). The carboxyl group shows the presence of oxalic acid impurities found in FIPW (Ni Made Wiratini and Nyoman Retug, 2014).

Morphological testing of FIPW to determine the physical changes that occur due to calcination and steamed. Based on the results of measurements with SEM, it can be seen the number of impurities and porosity of a sample (Byeong Ho Min and Kyeong Youl Jung, 2017). The number of impurities resulting from calcination and steaming variation morphologically is indicated by a white dot on the red circle. The number of impurities is very different in each of the calcination and steaming variations. The number of FIPW impurities is greater before calcination and steaming than after calcination and steaming. Impurities of FIPW is least when calcination at 195°C and steamed for 45 minutes (Figure 6.c). FIPW calcination at 195°C temperature, steamed for 45 minutes showed more open pores than FIPW at 165°C calcination and steamed for 60 minutes. Therefore, the absorption of FIPW at calcination at 195°C and 45 minutes steaming (Figure 6.c) is higher than FIPW at calcination at 165°C and 60 minutes steaming, namely 49.31% and 48.91%, respectively.

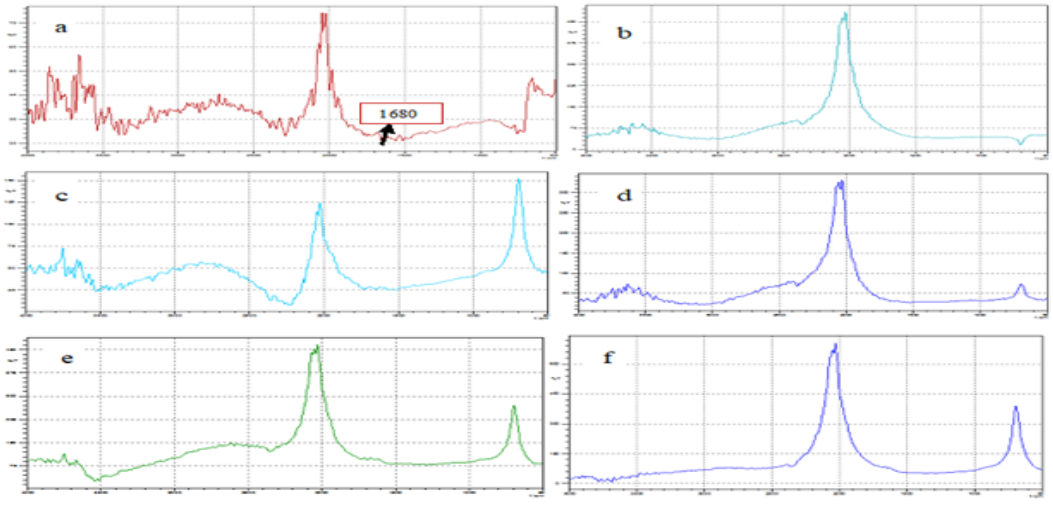

Figure 5. FIPW FTIR spectrum (a); FIPW calcination at 165°C, 60 minutes steaming (b); FIPW calcination at165°C, 45 minutes steaming (c); FIPW calcination at 195°C, 60 minutes steaming (d); FIPW calcination at 195°C, 45 minutes steaming (e); Activated Carbon Pro analysis Standards from Merck (F)

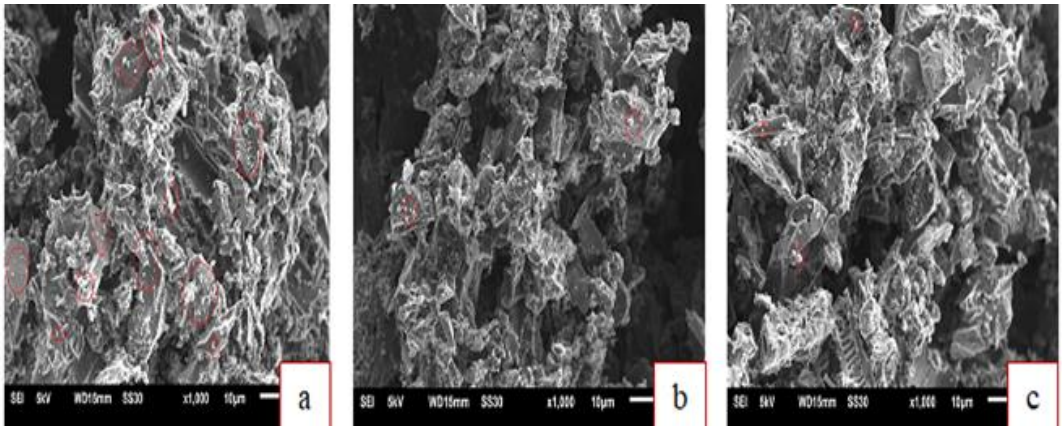

Figure 6. FIPW (a), FIPW calcination at 165ºC, 60 minutes steaming (b), FIPW calcination at 195ºC, 45 minutes steaming (c)

CONCLUSIONS

Calcination temperature and steaming duration have an influence on FIPW activation in the Analytical Chemistry Laboratory. The results showed to activate FIPW calcination at a temperature of 195ºC and 45 minutes steaming. The activated FIPW has the highest absorption and efficiency of absorption, FTIR spectrum closest to standard carbon, and the cleanest morphology with SEM from oxalic acid impurities.

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REFERENCES

Andreas O. Wagner, Nadine Praeg, Christoph Reitschuler, Paul Illmer. (2015). Effect of DNA extraction procedure, repeated extraction and ethidium monoazide (EMA)/propidium monoazide (PMA) treatment on overall DNA yield and impact on microbial fingerprints for bacteria, fungi and archaea ina reference soil, *Appl Soil Ecol*, 93, 56–64.

Byeong Ho Min, Kyeong Youl Jung. (2017). Improved porosity and ionic sorption capacity of carbon particles prepared by spray pyrolysis from an aqueous sucrose/NaHCO3/ TEOS solution, *RSC Adv*, 7, 21314–21322.

Hudha, A. M. 2002. Penyelenggaraan praktikum pada mahasiswa Jurusan Pendidikan Biologi FKIP UMM, Laporan Penelitian, Malang: Lemlit UMM.

Hyung Won Lee, Young-Min Kim, Seungdo Kim, Changkook Ryu, Sung Hoon Park, Young Kwon Park. (2018). Review of the use of activated biochar for energy and environmental applications, *Carbon Letters*, 26, 1-10.

I Ketut Lasia, I Ketut Budiada. (2017). Efisiensi pengolahan karbon limbah praktikum di laboratorium kimia analitik, Proseding Seminar Nasional Riset Inovatif, 67-72.

Lagunova Yu. O., Seliverstov F., Ershov G., Basiev. (2012). Oxidative decomposition of oxalate ions in water solutions of concentrated g ozone, *Atomic Energy*, 113(2), 93–95.

Mursid, R. 2013. Pengembangan model pembelajaran praktik berbasis kompetensi berorientasi produksi, *Cakrawala Pendidikan*, XXXII (1), 27-40.

Ni Made Wiratini, Retug Nyoman. (2014). Penuntun Praktikum Kimia Fisika, Singaraja: Jurusan Pendidikan Kimia FMIPA Universitas Pendidikan Ganesha.

Peraturan Bersama Menpan dan Kepala BKN No. 02/V/PB.2010. No.13 Th. 2013 tentang Petunjuk Pelaksanaan Jabatan Fungsional PLP dan Angka Kreditnya.

Ponkarthikeyan P., Sutha Sree K. (2017). A review of activated carbon by chemical activation, *ICRTCETM*, Special issue, 524-531.

Sani. 2011. Pembuatan karbon aktif dari tanah gambut, *Jurnal Teknik Kimia*, 5(2), 400-4006.

Taza Gul, Shakeela Afridi, Farhad Ali, Ilyas Khan, Ali Saleh Alshomrani. (2018). Heat transmission in the liquid film flow of micropolar fluid in a poros medium over a stretching sheet with thermal radiation, *Journal of Nanofluids*, 7, 1–9.

Werner Mäntele, Erhan Deniz. (2017). UV–vis absorption spectroscopy: lambert-beer reloaded, *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 173(15), 965-968

William Blum. 2017. The hydrolysis of sodium oxalate and its influence upon the test for neutrality (Classic Reprint), Forgotten Books, 521.

Yakout S.M., Sharaf El Deen G. (2016). Characterization of activated carbon prepared by phosphoric acid activation of olive stones, *Arabian Journal of Chemistry*, 9, S1155–S1162.

You J. H., Chiang H. L., Chiang P. C. (1994). Comparison of adsorption characteristics for VOCs on activated carbon and oxidized activated carbon, *Environmental Progress*, 13(1), 31-36.