# SYNTHESIS AND CHARACTERIZATION NANOPARTICLES OF $TiO_2$/ACTIVATED CARBON COMPOSITES USING DEEP COATING METHOD.

*Rahmawati*
Departement of Physic, Faculty of Engineering
University of Samudra, Langsa, Indonesia
watiusman@gmail.com

*Puji Wahyuningsih*
Departement of Chemistry, Faculty of Engineering
University of Samudra, Langsa, Indonesia
puji.chemistry@gmail.com

*Nitra Yulius*
Departement of Physic, Faculty of Engineering,
University of Samudra, Langsa, Indonesia
nitra.physics@gmail.com

***Abstract —*** A research to synthesis and characterizes nanosized $TiO_2$ particles on activated carbon (AC) were prepared by deep coating. Nanotechnologies are a set of methods and techniques for the treatment of matter and aimed at obtaining materials with novel functionalities and improved characteristics. In this research, the preparation $TiO_2$/AC composites was carried out in several stages, the first stage was prepared active carbon from empty palm oil bunches, then synthesized $TiO_2$ nanoparticles and last step was synthesis nanosized $TiO_2$ particles on activated carbon (AC) using deep coating method. The $TiO_2$/AC composites were characterized by various methods such as Fourier Transmission Infrared spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscope (SEM). The XRD pattern of synthesized of the $TiO_2$ nanoparticles shown one strogest peak, that indicative the prepared sample is single crystal structure. The crystallite size of the Titanium dioxide nanoparticles is 79,6 nanometer. SEM photo analysis of the morphology of TiO2 particles with uneven white clumps on the surface of activated carbon in the form of porous bars of non-uniform size. Based on this study has reported the properties of $TiO_2$/AC has been considered used in photocatalysis of the degradation of substances in an aqueous solution and the reduction of inorganic ions.

***Keywords*** : Titanium dioxide ($TiO_2$), Activated Carbon, Deep Coating.

## I. INTRODUCTION

Recently, several works were carried out on the preparation and application of $TiO_2$/AC composite as photocatalyst based on properties of that two particels. The photo catalytic activity of $TiO_2$ is dependent on its crystal structure, crystal size distribution, surface roughness, surface hydroxyl group density, etc. Beside of doping and structural modification of titania nanoparticles, supporting titania has been reported to exhibit different photocatalytic performance from that titania itself. The contribution of support material has been explained to give better mechanism regarding the interaction between titanium oxide and the support [1][5].

Activated carbon is one of the most effective, versatile and useful adsorbents for the removal of pollutants from polluted gas and liquid streams because of their large adsorption capacities, extremely high surface areas, well developed porous structures, fast adsorption kinetics and good mechanical properties. By this scheme, $TiO_2$/AC is the well-known and intensively studied. Some studies revealed the relationship among surface titanium oxide structure, preparation method and loading amount, etc. with its photocatalytic activity. They proposed a combination of photocatalytic degradation with granular AC treatment. The effluent from the photocatalytic process was filtered through a granular AC adsorber. The total cost was reduced considerably by using AC adsorption as the last step of treatment [3][4].

The deposition of $TiO_2$ nanophotocatalyst on commercial AC can be categorized into chemical and physical methods. Deep coating is the easiest and fast method to prepare thatcomposit with the highest degree of control. In specific high technology cases, it is used to deposit coatings on large surfaces. The principle is as simple as dipping the substrate into the initial solution before withdrawing it at a constant speed. During which the solution naturally and homogeneously spreads out on the surface of the substrate by the combined effects of viscous drag and capillary rise. Evaporation then takes over and leads to solidification of the final coating.

## II. EXPERIMENTAL METHOD

### *2.1. Material*

The activated carbon from oil palm empty fruit bunch (EFB) was prepared by chemical activation. The EFB was supplied by the Oil Palm Mill PKS Aramiah, Bireum Bayeun, East Aceh, Langsa. The materials were cleaned with distilled water several times to remove dust and impurities. Then, EFB samples were dried in oven at $100^0$C for 24h to remove any surface moisture.

The synthesis of titanium dioxide nanoparticles was done by sol-gel method. The first $Ti^{4+}$ solution is prepared by mixing titanium isopropoxide (TIPO:$Ti[OCH(CH_3)_2]_4$) and triethanolamine (TEOA: $N(CH_2CH_2OH)_3$) with a molarity ratio of 1: 2, then added aquadest to make a 0.5 M $Ti^{4+}$ . Then pH solution was increased by adding a NaOH solution to get pH of 9.6. The obtained solution was heated at a temperature of $100^0$C for 24 hours. After 24 hours the temperature is raised to $140^0$C and hold for 72 hours to form the nucleus and grow the $TiO_2$ crystals.

*2.2. Formulation $TiO_2$/AC composites*

In this research, the preparation $TiO_2$/AC composites was carried out in several stages, the first stage was prepared active carbon from empty palm oil bunches, then synthesized $TiO_2$ nanoparticles and last step was synthesis nanosized $TiO_2$ particles on activated carbon (AC) using deep coating method.

*2.3. Synthesis of $TiO_2$/AC composites*

The $TiO_2$/AC composites were characterized by various methods such as Fourier Transmission Infrared spectroscopy (FTIR), X-ray Diffraction (XRD), and Scanning Electron Microscope (SEM).

## III. RESULTS AND DISCUSSION

*3.1 Fourier Transmission Infrared spectroscopy (FTIR)*

The FT–IR spectra of the raw material EFB and activated carbon EFB were given in Fig. 1 and 2 respectively.

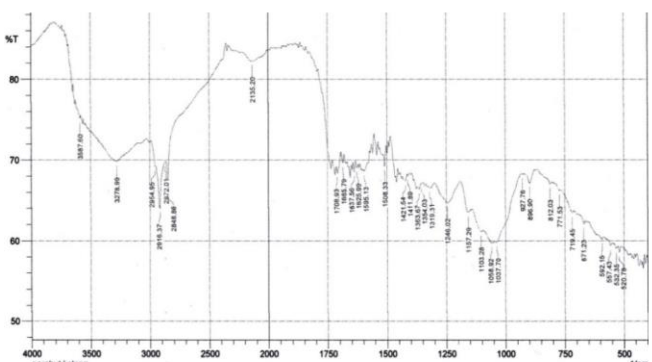



Figure 1. The FT–IR spectra of the raw material EFB.

The EFB sample shows a broad adsorption peak at 3302 $cm^{-1}$ which attributed to O–H stretching functional group. This indicates the presence of bonded hydroxide in the raw EFB.

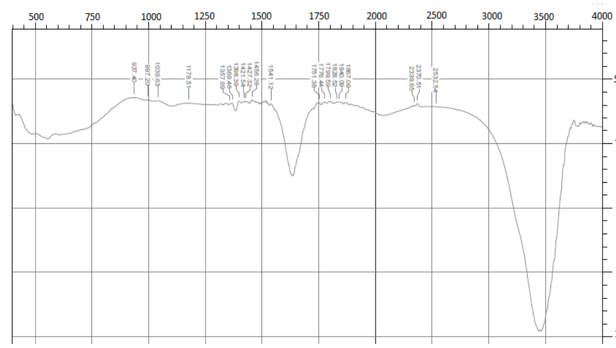


Figure 2. The FT–IR spectra of the activated carbon EFB

The FT–IR spectra of activated carbon EFB was represented in Figure 2. The adsorption peak in Fig. 2 indicate that carbonization and activation process caused some of the adsorption peak of functional groups in activated carbon were dissapeared. This is occured because the heating at high temperature cause some of functional groups in raw material EFB were vaporized as volatile materials. This proved that the activation process was successfully converted raw material EFB into carbon. FTIR spectra show similiarties of some peaks; adsorption peak at 1751,36 $cm^{-1}$ which attributed to C=O functional group and adsorption peak at 1043,49 $cm^{-1}$ which attributed to C–O stretching functional group but adsorption peak at 1265,3 $cm^{-1}$ was vaporized while activation process.

*3.2 X-Ray Diffraction Analysis*

The crystallite size of the particles was calculated from XRD peak of TiO2 by applying Scherrer's formula .

$$D = \frac{0.9\,\lambda}{\beta Cos\ \theta} \qquad (1)$$

$$2dsin\theta\ = n\lambda \qquad (2)$$

where λ is the X-ray wavelength β is the full width at half maximum(FWHM) in radians, and is the angular position of the peaks. The patterns reveal the $TiO_2$ were shown in Table 1.

Tabel 1. The patterns reveal the $TiO_2$

| 2θ | cos θ | λ (Å) | k | FW HM | FWHM (rad) | D (Å) | D (nm) |
|---|---|---|---|---|---|---|---|
| 25,3 | 0,98 | 1,5 | 0,9 | 0,20 | 0,0018 | 795,6 | 79,6 |

The crystalline phase of synthesized nanoparticles $TiO_2$ was analyzed by XRD and their XRD patterns are shown in Figure 3.

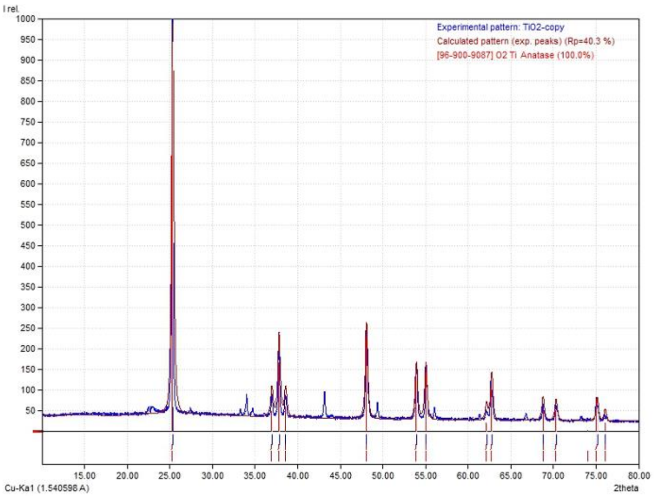
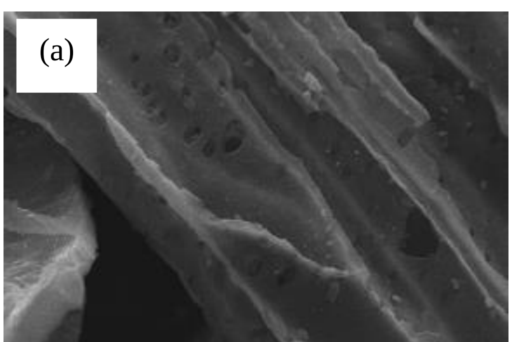


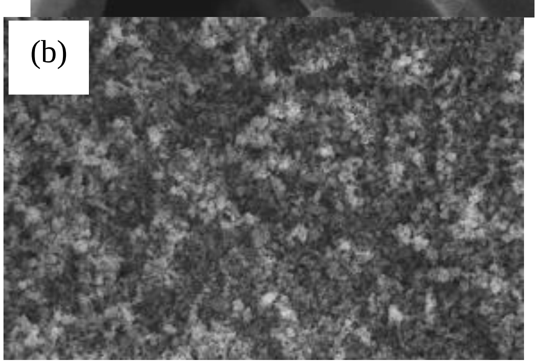

**Figure 3.** X-ray diffraction of nanoparticles $TiO_2$

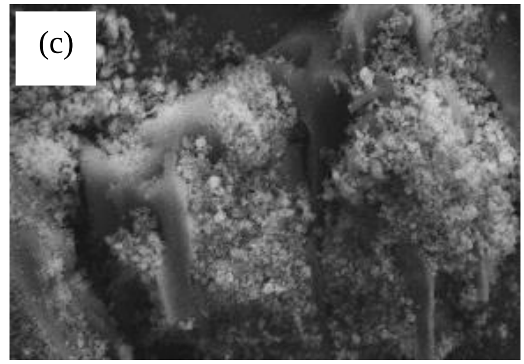
*3.3 SEM of $TiO_2$/Activedcarbon composite*

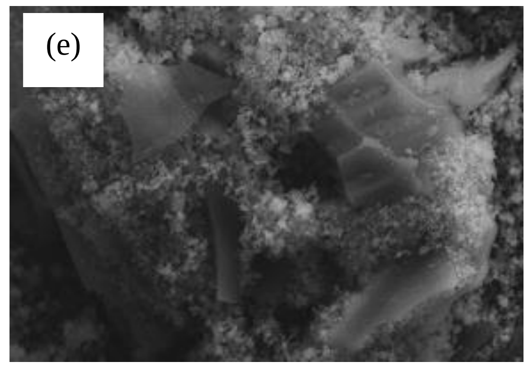
SEM analyzes are used to determine $TiO_2$ on the surface of the activated carbon. The results of SEM analysis of $TiO_2$/AC (Sample A, Sample B, Sample C, Sample D and sample E) are shown in Fig.4 (a, b, c, d, e, f, and g) below.

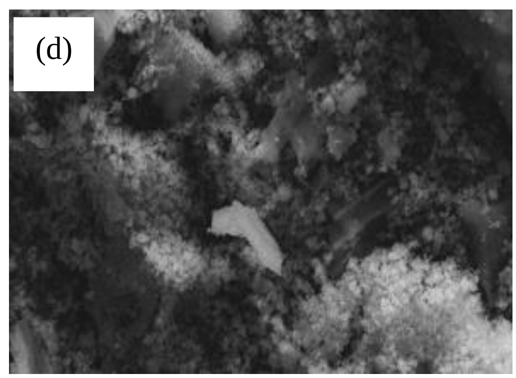
Figure (4.a) shown a pure SEM $TiO_2$ analysis image with magnitude of 5000 times. From the results, it is seen that $TiO_2$ particles are accumulate together, it is looks like a white blob with a size that is not uniform. Figure (5.6.b) was a SEM image of activated carbon with 5000 times of magnitude. From Figure SEM has seen that activated carbon in the form of bars are white and have pores.

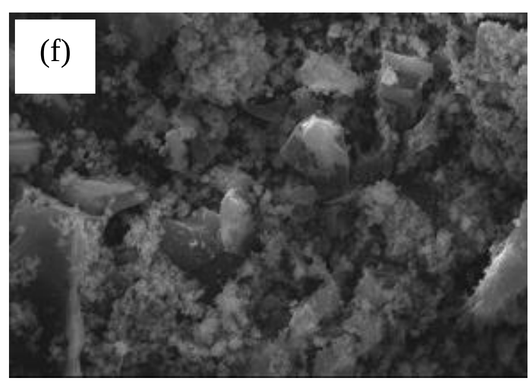



(b)

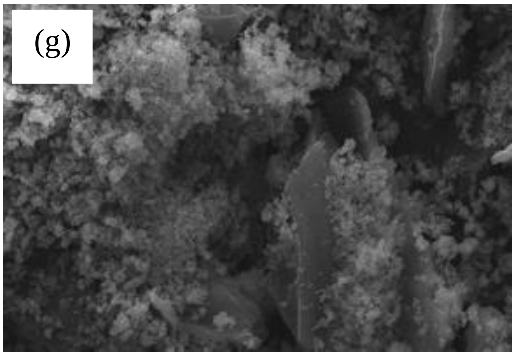



**Figure 4.** SEM analyzed (a) Active Carbon, (b.) $TiO_2$, (c.) Sample A, (d.) Sample B, (e.) Sample C, (f.) Sample D, (g) Sample E on magnitude 5000 times.

Figure 4 (c, d, e, f, and g) shown images of SEM analysis of $TiO_2$/AC with different variation (Sample A; Sample B; Sample C; Sample D and sample E). Overall SEM images of $TiO_2$/AC with various variation indicate that the $TiO_2$ particles are attached and spread unevenly on the surface of activated carbon. With the increase of activated carbon particles in the sample of $TiO_2$/AC causes the coagulation between the other $TiO_2$ particles is blocked. Activated carbon may block the buildup that occurs in the fellow $TiO_2$ particles. SEM photo analysis of the morphology of $TiO_2$ particles with uneven white clumps on the surface of activated carbon in the form of porous bars of non-uniform size.

## IV. Conclusions

$TiO_2$/AC composites were successfully prepared by deep coating. . The crystallite size of the Titanium dioxide nanoparticles is 79,6 nanometer. SEM photo analysis of the morphology of TiO2 particles with uneven white clumps on the surface of activated carbon in the form of porous bars of non-uniform size. Mobilizing TiO2 on AC can result in a synergistic combination of both adsorption and photocatalysis. Regeneration on the TiO2/AC composite is achieved by photocatalytic oxidation of pollutants.

## References


[1] Fujishima A, Hashimoto K, Watanabe T. 1999. *TiO2 photocatalysis – fundamentals and applications*. BKC, Tokyo.

[2] Hidayu,A.R., Mohammad, N.F., Matali, S., Sharifah, A.S.A.K., 2013, characterizationof Activated Carbon Prepared From Oil Palm Empty Fruit Bunch Using BETand FT-IR Techniques. *Procedia Engineering*. 379-384.

[3] Yong Tao, 2006. *Development of $TiO_2$ Carbon Composite Photocatalyst for The Removal of Methanol And Hydrogen Sulfide Paper Mills.* University of Florida

[4] Ameena, Y.K. 2003. *Titanium Dioxide Coated Activated Carbon: A Regenerative Technology For Water Recovery*, University Of Florida.

[5] Hashimoto, Kazuhito. 2007. $TiO_2$ Photocatalyst Toward Novel Building Material. *International RILEM Symposium on Photocatalyst, Environment Constraction Materials.* Florence-Italy.

[6] Ao, C.H., Lee, S.C., 2003. *Enhancement effect of TiO2 immoblized on activated carbon filter for the photodegradation of pollutants at typical indoor air level.* Appl. Catal. B: Env. 44, 191-205.

[7] Ming,C.L, Joug.N.C, Kuo.T.C., 1999. Effect ofAdsorbents Coated With Titanium Dioxide On The Photocatalytic Degradation Of Propoxur. *Elsevier Science.* Vol. 38 : 617-627.


## Authors' background


| Your Name | Title* | Research Field | Personal website |
|---|---|---|---|
| Rahmawati | S.Si, M.S. | Material Sciences | *watiusman@gmail.com* |
| Puji Wahyuningsih | S.Si. M.Sc. | Chemical Organic | *puji.chemistry@gmail.com* |
| Nitra Yulius | S.Pd. M.Si. | Applied Physic | *nitra.physic@gmail.com* |
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