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Eva Marlina Ginting

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Medan, 8 th October 2016

Prof. Dr. Syawal Gultom, M.Pd	Dr. Asrin Lubis, M.Pd	Prof. Dr. Herbert Sipahutar, M.Sc
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Following the successful of the First (2014) and the Second (2015) Annual International Seminar on Trends in Science and Science Education, this year Faculty of Mathematics and Natural Science of State University of Medan presents The Third Annual International Seminar on Trends in Science and Science Education 2016. Emphasizing on the latest trend in science and science education research, we invite researchers from inside and outside Indonesia to present their latest discoveries and gather in this scientific atmosphere.

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MORPHOLOGY AND CONDUCTIVITY FILM IN POLYANILINE DOPED ZnO

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Abstract This study aims to determine the conductivity and the morphology of the film Polyaniline doped with ZnO. The method of making the film of polyaniline / HCl / ZnO done using Galvanostat. Aniline monomer solution of 25 ml, 75 ml of HCl mixed with 1 g ZnO glass.dan beaker and 10 ml H_2O_2 disetirer, the sample results is polymerized with nickel as a partner electrode (anode / CE) and carbon as working electrode (cathode / WE). The mixture will be polymerized with nickel electrodes shaped thin plates and thin beam-shaped carbon electrode. Flow synthesis given by 4 mA with synthesis time for 30 minutes. PANI/ HCl formed will be attached to the carbon electrode in the form of a layer having a thickness of tens of micrometers formed layer is a thin film PANI / HCl / ZnO.The test results obtained by the morphology of the fibers intersect to form a porous structure looks homogeneous ZnO particles, whereas to determine the value of conductivity using the LCR-Meter, the value of orders $10^{-4}\Omega$ / m, which meets the conductivity value as a semiconductor material.

Keywords. Polyaniline, HCl, ZnO, morphology, conductivity

I. INTRODUCTION

Polymers in general is a material with the ability to conduct electricity is low and does not have a response to the magnetic field from the outside. But through several studies conducted, some polymer material turns electrical conductivity can be improved by adding an acid causing a second phase that is conductive. One polymeric material electrical conductivity can be improved is polyaniline (PANi).
Conductive polymer materials of concern because it is conductive can be applied as an electronic material such as capacitors, pH sensors and transistors [1]

Electroactive conductive polymers and new materials that are highly in demand by researchers due to electrical and magnetic properties of the unique and easy way to synthesis. One type of conductive polymer is polyaniline widely studied because it has a higher environmental stability and ease of synthesis process, compared to other conductive polymers

Polyaniline can be synthesized electrochemically produce products in the form of films and the usual chemical synthesis will produce polyaniline in powder form. Synthesis of polyaniline chemically ordinary is often done because the interfacial polymerization method is relatively easy in the provision of doping.

Polyaniline-based composites research has been done,including by[2-4].
Fe3O4@PANI nanocomposites [5]

ZnO is is a relatively soft material with a hardness of about 4.5 Mohs. Elastic constants smaller than the III-V semiconductors, such as GaN. Heat capacity and high thermal conductivity, low thermal expansion and high melting temperature ZnO is beneficial for ceramics. Among the tetrahedral semiconductors, ZnO has the highest piezoelectric tensor or at least comparable with GaN and AlN. These properties make this material is an important technology for many piezoelectric applications, which require a large electromechanical clutch.

ZnO has a relatively large band gap of ~ 3.3 eV at room temperature, because it is pure ZnO is colorless and transparent. Advantages associated with a large band gap, including a high voltage, the ability to sustain large electric fields, high temperatures and large operating power

In writing this article aims to determine the morphology and electrical conductivity of the film Polyaniline doped with ZnO, of the data is expected to be used as a superconducting material.

II. Experimental Procedures

Material

Aniline monomer ($C_6H_5NH_2$), Purity 15%, Merck Germany, Mr: 93.13 g / mol, 25% purity HCl GR Merck Germany, Mr: 36.46 g/mol, H_2O_2,Zn $(CH_3COO)_2.2H_2O$ Mr: 219 Merck Darmstadt Germany, Mr NH_4OH:42Merck Darmstadt Germany, Darmstadt Germany Merck Ethanol.

Instrument.

SEM - EDX JEOL5310 LV. LCR Meter

Synthesis of ZnO Particles with Coprecipitation Method

Zn $(CH_3COO)_2.2H_2O$ as much as 5 ml of 0.2 M NH_4OH mixed with about 45 ml. Then add 25 ml of ethanol, the solution distirer and heated to a temperature of 80^o C for 2 hours. After the cooling the solution at room temperature for a few minutes, then wash with a solution of water and ethanol to remove impurities to produce a white precipitate. Then dry in the oven for 24 hours.

Synthesis of Polyaniline Film / HCl / ZnO

H_2O_2 solution of 10 ml and 1 g of ZnO in the stirrer for 2 hours on a glass beaker. Mix 25 ml of aniline, and 50 ml of HCl in the solution of H_2O_2 and ZnO. And then the mixture was polymerized using galvanostat with nickel as the partner electrode (anode / CE) and carbon as working electrode (cathode / WE). The mixture will be polymerized with nickel electrodes shaped thin plate and a thin beam-shaped carbon electrode. PANI / HCl / ZnO were formed to be attached to the carbon electrode in the form of a layer having a thickness of tens of micrometers formed layer is a thin film PANI / HCl / ZnO .

III. Results and Discussions

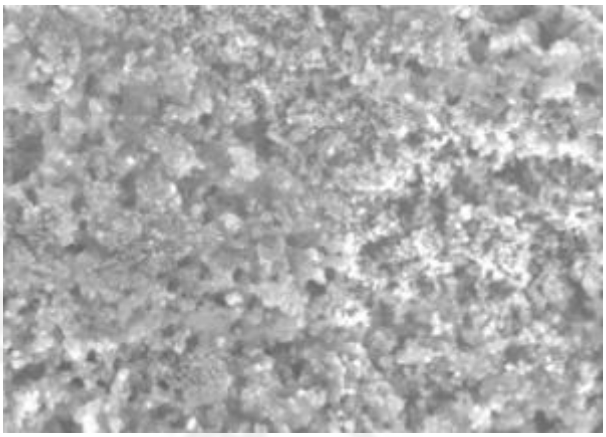

Fig .1 Polyaniline Film Morphology

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Polyaniline film surface morphology was observed directly by using an electron microscope (SEM). The surface morphology of polyaniline samples taken using SEM . The surface morphology is shown in Figure 1 appears SEM image shows the structure with a diameter of several tens of nanometers and several hundred nanometers long and highly porous. In the SEM image, it can be observed clearly nanofiber-this nanofiber intersect to form highly porous structure [4]

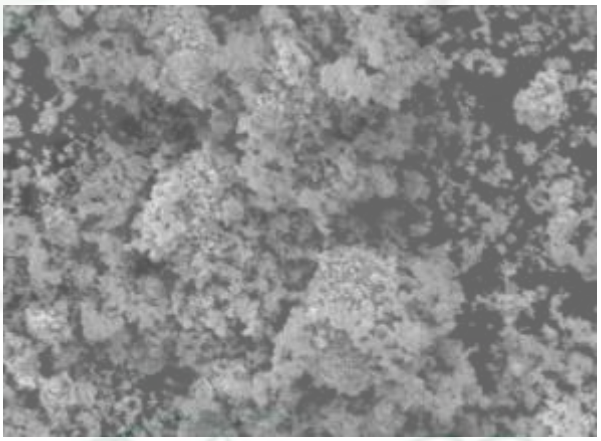

Fig .2 Morphology ZnO

In Figure 2, ZnO particles seem homogeneous and agglomeration. Agglomeration in the whole sample due before washing ZnO allowed to stand for 24 hours, experienced the process, the particles that have been formed back reacts with oxygen in the air. When the particles react with oxygen, there is the possibility of positively charged or negatively. This attraction causes the particles of the joining other particles to form larger particles [5]

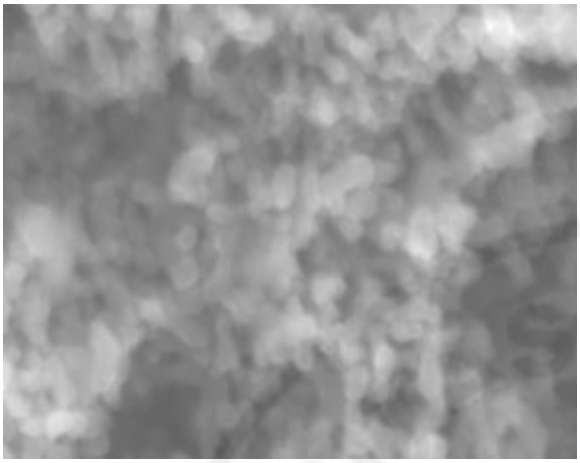

Fig .3 Polyaniline Film Morphology/HCl/ZnO

From Figure 3 it can be seen that there is a uniform porosity on the surface of the sample. The color difference in the image does not reflect the variety of elements in the sample. As for the composite PANi/HCl /ZnO, with different magnifications remains visible presence of pores in the area of the sample is black and white clots allegedly ZnO particles. The size and distribution of ZnO particles can not be detected on SEM results.

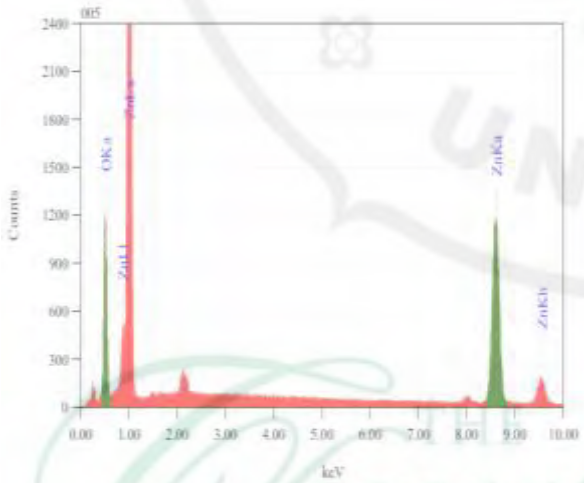

Fig 4. The test results on samples of ZnO elementary

From Figure 4 .. is the result of testing using X-ray fluorescence (XRF). The elementary analysis results indicate that the content of zinc and oxygen can be determined quantitatively. The elements contained in the sample is Zn and O. Based on the results of the elementary analysis, Figure 4 shows that the information obtained in the ZnO samples containing zinc (Zn) amounted to 77.77% and oxygen (O) of 22.23 %. This indicates that the ZnO nanoparticles contain more zinc (Zn) than the element oxygen (O).

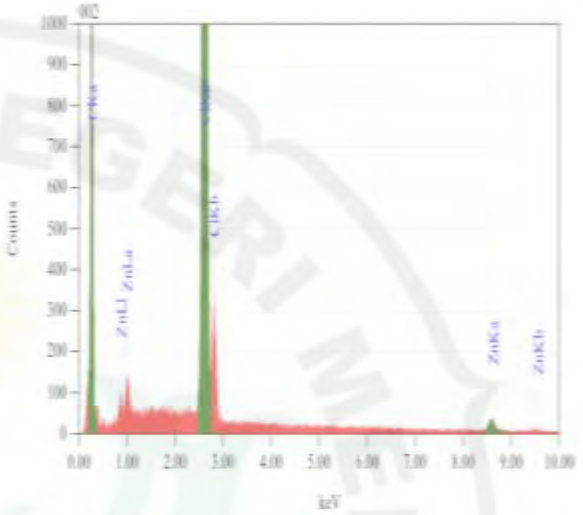

Fig 5. The test results on samples of elementary Polyaniline / HCl / ZnO

Figure 5 is the result of testing using X-ray fluorescence (XRF). The elementary analysis results indicate that the content of zinc and oxygen can be determined quantitatively. The elements contained in the sample is PANI / HCl / ZnO. The elementary analysis results, show that the information obtained in the sample PANI / HCl / ZnO containing as aniline ($C_6H_5NH_2$) and Oxygen, Element Cl and O_2 of 13.38% and zinc (Zn) and 1.28% oxygen. This indicates that the PANI / HCl / ZnO many contain as aniline, elements of H does not exist because the reduced along H_2O_2.

Electrical conductivity

Conductance thin films was measured by using LCR-meters. Measurements were made at room temperature with the lamp lighting the room. Electrical conductance data is used to calculate the value of electrical conductivity of the film Polianialin. Conductance data obtained will be compared with data theory is formed including the conductor, semiconductor or insulator material. Based on the electrical properties of materials distinguished by: conductors, semiconductors and insulators. Conductors ($\sigma = 10^7\ \Omega$ / m), Isolator ($\sigma\ \Omega = 10^{-20}$- 10^{-10} / m), Semiconductors ($\sigma = 10^{-6}$-$10^4\Omega$ / m).Conductivity Measurement Polyaniline

Film / HCl obtained konduktivisnya value of 0.52 x 10^{-3} Ω / m.
These results were obtained from Polyaniline Film conductivity value with the value of a frequency of 1 Hz - 100 kHz. Polyaniline films were synthesized showed that the material is a semiconductor.

Polyaniline based its electrical properties are divided into two: the conductive polyaniline and polyaniline isolatif. Based on the level of oxidation, polyaniline can be synthesized in several forms, namely isolatifnya leucomeraldine base (LB) reduced full, emeraldine base (EB) is oxidized half and pernigranilin base (PB) which fully oxidized. Of these three forms, EB palingngan stable and also the most widely studied because of its conductivity can be set from 10^{-10} S/cm to 100 S/cm through a doping process, while LB and PB forms can not be made conductive. Forms of EB can be made conductive by doping process protonik acid such as HCl, in which protons are added to sites -N =, while the number of electrons in the chain. Conductive shape of EB-called emeraldine salt (ES) [6]

Results obtained from the value of conductivity of ZnO is 0,8x10^{-4}Ω / m. Values greater than the conductivity of ZnO film conductivity PANI / HCl. Film PANI/HCl/ZnO of measurement have conductivity values 1,45x10^{-3} Ω/ m. Conductivity value accreting. This is because the value of Polyaniline doped with ZnO., The characterization of the electrical properties of Polyaniline Film / HCl / ZnO obtained that polyaniline and ZnO is a semiconductor material. So it is suitable in applications Semiconductor and p-n junction diode.

Table 1-3 shows the results of measurements with LCR to determine Inductance (L) Capacitance (C) Resistance (R) Impedance (Z) of PANI material, ZnO and Polyaniline / HCl / ZnO, whereas Table 4 shows the conductivity value of each material

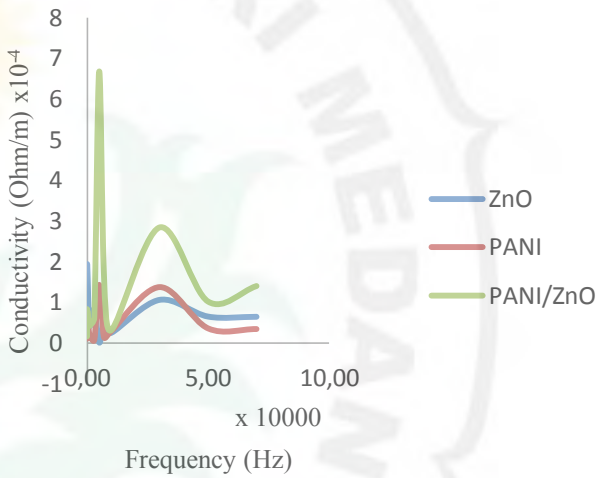

Fig 6. The Relationship Between The Conductivity Of The Frequency

Figure 6 shows the value of the conductivity of PANI -ZnO greatest value.

Table .1.
Measurement Results With LCR Film Polyaniline (PANI)

	PANI			
f (Hz)	Inductance (L)	Capacitance (C)	Resistance (R)	Impedance (Z)
1	5,58 MH	4,54 uF	7,02 kOhm	6,88kOhm
10	402,93 kH	628,65 uF	6,99 kOhm	6,74 kOhm
100	22,13 kH	114,46 uF	13,52 kOhm	9,69 kOhm
1000	848,24 H	29,86 uF	6,24 kOhm	4,05 kOhm
3000	88,03 H	31,97 uF	2,74 kOhm	1,42 kOhm
5000	41,90 H	24,18 uF	2,76 kOhm	1,18 kOhm
7000	20,42 H	25,31 uF	2,19 kOhm	831,47 Ohm
10000	12,15 H	20,85 uF	1,99 kOhm	713,07 Ohm
30000	1,81 H	15,54 uF	1,10 kOhm	326,02 Ohm
50000	688,81 H	14,71 uF	837,13 Ohm	209,51 Ohm
70000	370,04 H	13,97 uF	730,24 Ohm	158,85 Ohm
100000	190,03 H	13,33 uF	499,47 Ohm	116,13 Ohm

Table .2.
Measurement Results With LCR ZnO

	ZnO			
f (Hz)	Inductance (L)	Capacitance (C)	Resistance (R)	Impedance (Z)
1	1,80 MH	140,09 uF	20,21 kOhm	9,904 kOhm
10	179,19 kH	141,36 uF	14,11 kOhm	8,8029 kOhm
100	14,36 kH	156,32 uF	44,33 kOhm	8,845 kOhm
1000	3,53 kH	71,68 uF	87,94 kOhm	21,527 kOhm
3000	212,87 H	13,22 uF	38,77 kOhm	3,9911 kOhm
5000	51,56 H	19,65uF	6,84 kOhm	1,5765 kOhm
7000	42,66 H	12,11uF	7,49 kOhm	1,8204 kOhm
10000	35,86 H	7,95 uF	7,72 kOhm	1,9381 kOhm
30000	5,19 H	5,42uF	6,20 kOhm	966,87 kOhm
50000	2,04 H	4,94uF	5,08 kOhm	638,11 kOhm
70000	1,286 H	4,02 uF	3,89 kOhm	559,97 kOhm
100000	610,06 H	4,15 uF	2,00 kOhm	376,48 kOhm

Table .3
Measurement Results With LCR Film Polianilin/HCl/ZnO

	PANI/HCl/ZnO			
f (Hz)	Inductance (L)	Capacitance (C)	Resistance (R)	Impedance (Z)
1	3,90 MH	6,49 uF	4,66 kOhm	4,58 kOhm
10	349,88 kH	723,98 uF	3,99kOhm	3,92 kOhm
100	282,97 kH	8,95 uF	3,40 kOhm	3,39 kOhm
1000	514,58 H	49,22 uF	2,93 kOhm	2,17 kOhm
3000	53,98 H	52,13uF	1,65kOhm	866,89 kOhm
5000	29,047 H	34,88 uF	1,34 kOhm	755,93 kOhm
7000	16,618 H	31,10 uF	1,518 kOhm	658,63 kOhm
10000	8,33 H	30,39 uF	1,31kOhm	486,35 kOhm
30000	1,09 H	25,72 uF	665,30 Ohm	198,98 kOhm
50000	440,1 0 H	23,023uF	460,11 Ohm	132,41 kOhm
70000	236,01 H	21,90 uF	354,77 Ohm	99,62 kOhm
100000	123,10 H	20,57 uF	272,23 Ohm	74,4 0kOhm

Table .4
Conductivity with LCR – METER

f(Hz)	ZnO	PANI	PANI-ZnO
	σ (Ohm./m) x 10^{-4}	σ (Ohm/m) x 10^{-4}	σ (Ohm./m) x 10^{-4}
1	1,1453	0,1197	0,8339
10	1,4887	0,0992	0,1617
100	1,8940	0,7057	0,7953
1000	0,4460	0,2023	0,4194
3000	0,8232	0,0675	0,6231
5000	0,0158	1,4276	6,6691
7000	0,4126	0,1340	1,6542
10000	0,2518	0,2894	0,3158
30000	1,0604	1,3723	2,8410
50000	0,6501	0,3544	1,0184
70000	0,64524	0,3379	1,4001
100000	0,45533	1,1627	0,6377

IV. CONCLUSION .

Film Morphology PANI/ HCl / ZnO, with different magnifications remains visible presence of pores in the area of the sample is black and white colored blobs allegedly ZnO particles. Based on the results of the elementary analysis by XRF show that the information obtained in the sample PANI / HCl / ZnO containing aniline ($C_6H_5NH_2$) and Oxygen, Element Cl and O_2 of 13.38% and Zn and 1.28% oxygen. This indicates that the PANI / HCl / ZnO many contain aniline.The existence of an element of H is not due to reduced joint H_2O_2. Conductivity Measurement Results Polyaniline Film/HCl obtained conductivity values of the order of 10^{-4} Ω/m.
These results were obtained from Polyaniline Film conductivity value with the value of a frequency of 1 Hz - 100 kHz. Film Polyaniline synthesized showed that the material is a semiconductor. The conductivity of ZnO value is 10^{-4}Ω / m. Values greater than the conductivity of ZnO film conductivity PANI / HCl. Film PANI / HCl / ZnO of measurement have conductivity values of the order of 10^{-4} Ω/ m.Nilai Conductivity accreting. This is because the value of Polyaniline doped with ZnO.

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