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ANALYSIS OF THE OPTICAL PROPERTIES OF THE SYNTHESIS OF Fe_3O_4/PANi NANOCOMPOSITES WITH THE SOL-GEL METHOD USING SPIN COATING

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ABSTRACT

Research has been carried out on the analysis of the optical properties of Fe_3O_4/PANi nanocomposites synthesized by the sol-gel method using spin coating. The purpose of this study was to determine the crystal structure, crystal size, surface morphology, and optical properties of the Fe_3O_4/PANi nanocomposite thin films. One of the applications of optical properties is that it can be used as a semiconductor. In this study, the primary material used was iron sand obtained from Oyster Beach, Padang Pariaman Regency, West Sumatra. Iron sand contains a magnetite (Fe_3O_4) phase, which has the highest magnetic properties, and the polymer used is a type of polyaniline polymer (PANi) which has good chemical stability. Fe_3O_4 acts as a filler, while PANi acts as a matrix. In this study, five variations of the composition of Fe_3O_4 in PANi were used, namely 30%, 40%, 50%, 60%, and 70%. For the method used, namely the sol-gel method and sample testing using XRD, SEM, and UV-Vis Spectrophotometer characterization tools. The results of the XRD characterization showed that each addition of PANi into Fe_3O_4 did not affect the resulting crystal structure and had a crystal size below 100 nm. SEM testing revealed the presence of agglomeration and particle size at variations of 30%, 40%, 50%, 60%, and 70% were 27 µm, 53 µm, 61 µm, 84 µm, and 46 µm respectively. And the results of the UV-Vis Spectrophotometer test obtained energy gap values for variations in the composition of Fe_3O_4 in PANi, namely 30%, 40%, 50%, 60%, and 70%, respectively, namely 2.51 eV, 2.39 eV, 2.18 eV, 2.35 eV, and 2.30 eV. Based on the research results, it can be proven that the Fe_3O_4/PANi nanocomposite thin layer is a semiconductor material because it is in the range of 0-3 eV.

Keywords : *Fe_3O_4, PANi, Optical Properties, Thin Films, Spin Coating*

I. INTRODUCTION

At this time, one of the research that is developing very rapidly is research on magnetic oxides [1]. Magnetic oxide exists in nature and is often found in iron sand. According to a report from research conducted by Darvina, et al, 2017, iron sand has blackish color characteristics, which are often found along the coast, one of which is at Tiram Beach, Padang Pariaman Regency, West Sumatra. Where based on the results of XRD characterization shows that the iron sand of Oyster Beach contains iron oxide minerals in the form of magnetite in addition to other oxide minerals [1]. Iron sand is usually called black sand because of its own color [2]. The color is influenced by the content of iron oxides such as magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), and hematite (α-Fe_2O_3) [2]. Magnetite

(Fe_3O_4) has become a priority in research activities because it has many advantages compared to other mineral compounds [2].

At present, nanostructured materials have attracted the attention of scientists because of their tiny size and surface area to volume ratio, causing sizes that affect their chemical and physical properties which are very different from large materials with the same chemical composition [3]. The nanostructure of magnetic oxide is one of the essential nanomaterials in the development of several new functional and intelligent materials [4]. The field of nanocomposite materials has recently received serious attention from scientists [5]. Various studies that are carried out very carefully are continuously carried out [5]. The research was carried out based on an elementary thought/idea, namely composing a material consisting of blocks of homogeneous particles with nanometer size [5]. Nanocomposites can be considered solid structures with nanometer-scale dimensions that are repeated at distances between different structural constituents [5]. Bonding between particles that occur in nanocomposite materials plays a vital role in improving and limiting material properties [5]. In general, nanocomposite materials exhibit different mechanical, electrical, optical, electrochemical, catalyst, and structural properties compared to their constituent materials [5]. Nanocomposites are composite materials made by inserting nanoparticles into a macro-sized material as a filler in a matrix [6]. Composite materials are divided into two parts, namely filler (matrix reinforcement) and matrix (filler protection) [6].

Magnetic nanoparticles, iron oxide magnetite (Fe_3O_4), is an attractive material and has extensive applications [7]. In its bulk size, this material is a group of ferrimagnetic materials [7]. However, at nanometer size, this material becomes a superparamagnetic material and has better properties such as high saturation magnetization (90 emu/gram), biological compatibility, and environmental stability [7]. For the manufacture of nanocomposites, conductive polymers are needed [8]. Polyaniline (PANi), also known as aniline black, is one of the most common polymers in the manufacture of conductive polymers. PANi synthesis can be used in two ways, namely chemical or electrochemical synthesis [9] in chemical synthesis, which acts as a trigger for the polymerization process is an additional element (free radicals) in this study using ammonium peroxydisulfate (APS) while in electrochemical synthesis is electrical energy. Until now, many conductive polymers have been synthesized, but of all conductive polymers, PANi has several advantages, apart from the ease of synthesis and low cost, as well as excellent chemical and physical stability [10].

Methods that can be used to synthesize iron nanoparticles include the sol-gel method, flash combustion, coprecipitation, microemulsion, hydrothermal, and others [11]. The sol-gel method is a "wet method" because the process involves a solution as the medium [11]. In the sol-gel method, as the name implies, the solution undergoes a phase change to become a sol (colloid, which has solids) and then becomes a gel (colloid but has a more significant solid fraction than sol) [11]. The spin coating method can be interpreted as a method of forming thin layers through a spin or spin process [12]. The material to be included in a thin layer is made in the form of a solution or gel, which is then dripped onto a substrate and stored on a disc, which can rotate at a reasonably high speed [12]. Thin films have optical properties that are highly dependent on the energy gap of the properties of the thin layer material to be deposited [1].

II. METHOD

This type of research is experimental research, in which experimental research is one of the types of quantitative research methods that aim to test the success or failure of experimental variables. The research was carried out from May to October 2022 at the Laboratory of Materials Physics and Biophysics, FMIPA, Padang State University. Iron sand is the sample used in this study which was obtained from Oyster Beach, Padang Pariaman Regency, West Sumatra, Indonesia. This study discusses the analysis of the optical properties of the Fe_3O_4/PANi nanocomposites synthesized by the sol-gel spin coating method. Testing of optical properties was carried out using a UV-Vis Spectrophotometer test kit.

In this study, the characterization test tools used were X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), and UV-Vis Spectrophotometer. The XRD test kit provides information about the crystal structure and size of the crystals. SEM test results provide information on the structure of surface morphology and particle size. At the same time, the UV-Vis Spectrophotometer test equipment provides information about optical properties in terms of absorbance, transmittance, reflectance, and energy gap values. The XRD test tool used has a size specification of 75 × 75 × 60 cm with a jar quantity of 1 pcs volume of 100 mL power used 220 V phase 3 motor 0.4 kW speed 1410 with milling timer 1 minute - 99 hours 99 minutes and this tool is at the Physics Materials Laboratory, FMIPA, Padang State University. The SEM test kit has the JEOL JSM-6360LA specification with a magnification of 2000X, and this tool is located at the Marine Geology Research and Development Center Laboratory located in Bandung. While the UV-Vis Spectrophotometer test tool has the UV VIS 100 DA-X type, the lowest single bear wavelength is 190-210 nm, and the highest is 800-1000 nm, this tool is located in the

Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences, Padang State University. The form of the UV-Vis Spectrophotometer is shown in Figure 1.

Fig 1. UV-Vis Spectrophotometer

The study of the physical properties of material includes electrical, magnetic, optical, and mechanical properties. One of the electrical properties of materials is the energy gap. Study the electrical properties, primarily to determine the size of the energy gap, which is determined through the absorption spectrum of the compound material. The absorption spectrum of the material was obtained using a UV-Vis spectrophotometer [13]. The energy gap is the difference between the upper end of the valence band (Ev) and the lower end of the conduction band (Ec) or the minimum energy required to excite electrons from the valence band to the conduction band [13]. The Tauc plot method is a method of determining the optical band gap by looking at a linear graph of the relationship E (eV) on the x-axis and $(\alpha h\upsilon)^{1/m}$ on the y-axis. The relationship between photon energy ($h\upsilon$) and absorption coefficient (α) is determined by the equation:

$$(\alpha h\upsilon)^{1/m} = \mathrm{c}\ (h\upsilon - Eg) \qquad (1)$$

Where h = 6.63 × 10-34 J.s, c is the constant of the speed of light and Eg is the material energy gap, and the exponent m depends on the type of transition [14].

A search for the gap energy value is carried out to determine whether the material is an insulator, semiconductor, or conductor. Magnetite (Fe_3O_4) is a material with the properties of semiconductor material, this was reported by Ghandoor, H.E, et al, 2012 [15]. Semiconductor materials act as insulators at shallow temperatures and act as conductors at room temperature. The range of semiconductor gap energy values is at a value of 0-3 eV [15].

To achieve this research, several tools were used, namely HEM-E3D, X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), UV-Vis Spectrophotometer, spin coating, oven, magnetic stirrer, permanent magnets, digital scales, glasses beakers, stirring rods, measuring cups, glass substrates, dropping pipettes and laboratory tweezers. While the materials used are iron sand, alcohol, distilled water, ammonium persulfate, tapioca flour, ethylene glycol, polyaniline (PANi), nitric acid, and oxalic acid. There were three stages carried out in this study, namely the sample preparation stage, the sample making stage, and the sample characterization stage. The research steps can be seen in the form of a flowchart in Figure 2 below, namely:

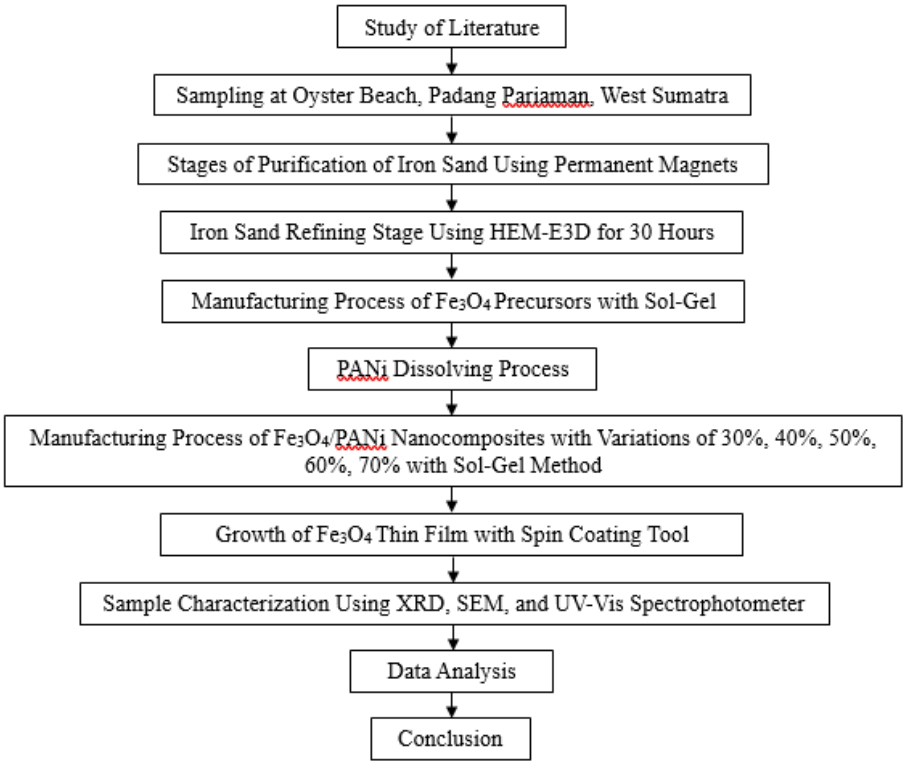


Fig 2. Flowchart

The initial stage is the sample preparation process starting with taking iron sand from Tiram Beach, Padang Pariaman Regency, West Sumatra, with the help of a permanent magnet, then the iron sand is pulled using a permanent magnet 30 times with the aim of separating the iron-containing sand from other materials. Then the iron sand is washed using distilled water to clean the iron sand and dried in the sun until dry. After that, the iron sand is pulled one more time using a permanent magnet 20 times to ensure that the iron sand is not mixed with other impurities [16]. Then the milling process was carried out for 30 hours. This milling time provision is based on the research of Darvina, et al, (2017), where the milling time of 30 hours shows the loss of the hematite phase so that only one phase is left, namely Fe_3O_4 [1]. The second stage is the process of making the sample, starting with creating the precursor ($Fe(NO_3)_3.9H_2O$) using the sol-gel method. The precursor was prepared by reacting 4.35 g Fe_3O_4 with 1.125 g oxalic acid ($C_2H_2O_4$) with 10.25 ml nitric acid (HNO_3) at 110℃ using a magnetic stirrer. Then 3.5 g of the dissolved sample was weighed and dissolved with 13.75 g of ethylene glycol in a beaker and stirred using a stirring rod. After that, heating the sample that has been stirred is heated at 80 ℃ while stirring using a magnetic stirrer at 250 rpm for 2 hours, and when finished, a gel is formed.

The next step is the process of dissolving PANi by dissolving 0.93 g of PANi and 1.15 g of ammonium persulfate in 100 ml of HNO_3 and stirring for 2 hours. After that, the process of making Fe_3O_4/PANi nanocomposites was carried out by determining five variations of the Fe_3O_4 composition in PANi, namely 30%, 40%, 50%, 60%, and 70%. Then weigh the two materials according to the variation that has been determined, then PANi according to the concentration in the stirrer together with ammonium persulfate as much as 0.05066 g for 15 minutes at 20℃. After that, mix the Fe_3O_4, which has been weighed based on the composition and stirrer for 48 minutes at 80℃ according to research conducted by Khairy (2014) [17]. The process of making Fe_3O_4/PANi nanocomposites forms a gel that will be needed in the process of making thin films using spin coating. A thin layer was grown using a spin coating tool on a glass substrate that had been cleaned with ethanol. Then, the solution was dripped using a dropper onto a glass substrate measuring 2 cm × 3 cm and rotated with a spin coating. The spin coating speed used is 1000 rpm for 60 seconds (Rianto D, et al., 2018). The coating time is in accordance with previous research conducted by Eken (2009). And the thin layer of Fe_3O_4/PANi nanocomposite that had been formed was heated at 60℃ for 6 hours [1].

III. RESULTS AND DISCUSSION

Research on the optical properties of the Fe_3O_4/PANi nanocomposite results can be tested using several characterization test equipment. XRD test equipment can determine the crystal structure and crystal size. While the SEM test tool can determine the surface morphology of the sample, and the UV-Vis Spectrophotometer test tool can determine the absorbance, transmittance, reflectance, and energy gap values.

To determine the crystal structure and crystal size of the Fe_3O_4/PANi nanocomposite, tests were carried out using an XRD test kit. The following is a graph of the Fe_3O_4/PANi nanocomposite XRD characterization with five variations of Fe_3O_4 composition in PANi, namely 30%, 40%, 50%, 60%, and 70% can be seen in Figure 3.

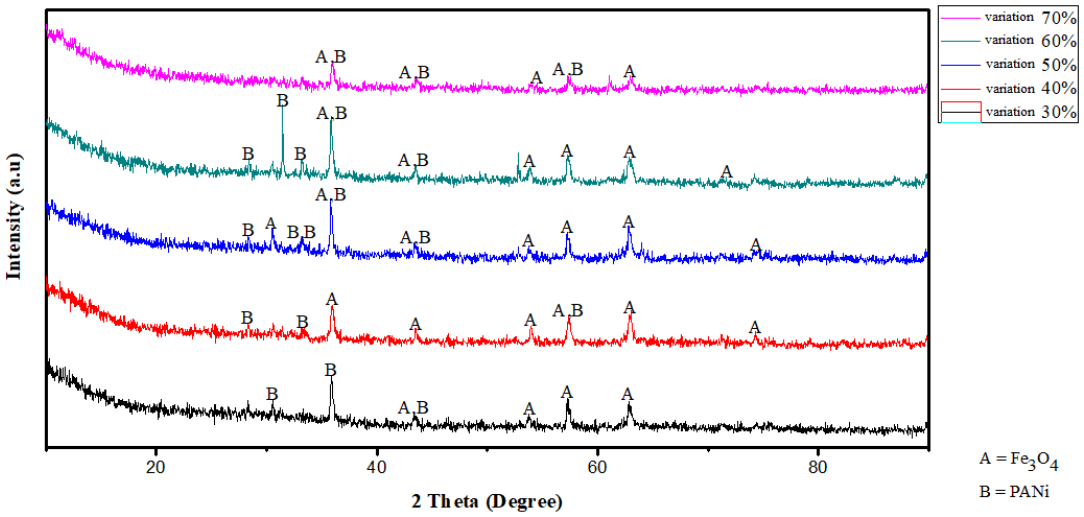
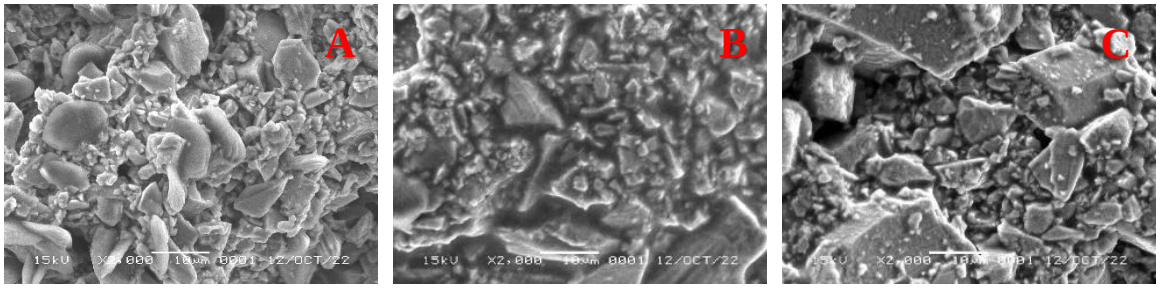


Fig 3. Fe_3O_4/PANI Nanocomposite XRD Characterization Results with 5 Composition Variations

It can be seen in Figure 3, shows the results of the XRD characterization in the form of X-Ray Diffraction patterns from Fe_3O_4/PANi nanocomposite thin films with five composition variations, namely 30%, 40%, 50%, 60%, and 70%. For the results of XRD characterization, the crystal structure of the Fe_3O_4/PANi nanocomposite was obtained, namely the cubic structure for the Fe_3O_4 phase and orthorhombic for the PANi phase in all variations. This is in accordance with the research of Simamora et al., 2018 [18], which stated that the crystal structure obtained by Fe_3O_4/Polypyrrole was cubic [18]. For crystal size variations in the composition of Fe_3O_4 in PANi, namely 30%, 40%, 50%, 60%, and 70%, respectively, were 49.1 nm, 52.2 nm, 51.7 nm, 51.0 nm, and 50.6 nm. As seen in Figure 3, the peaks that stand out in each variation are 35°, 43°, and 57°, and the miller indices in the Fe_3O_4 plane are (311), (400), (511), and the PANi miller indices are (031), (201), (420) and (421). With the addition of variations in the composition used, the resulting crystallite size decreased. This is because the atoms have low energy which makes it difficult for the atoms to move and begin to adjust their positions relative to other atoms so that the crystal size obtained is small [19].

Characterization using the SEM tool of the Fe_3O_4/PANi nanocomposite thin layer aims to determine the surface morphology of the sample. For sample testing, the tool used was SEM with JEOL JSM-6360LA specifications, and sample testing was carried out at the Marine Geology Research and Development Center Laboratory located in Bandung and using 2000X magnification. The following is the result of the SEM characterization of the Fe_3O_4/PANi nanocomposite with five composition variations, namely 30%, 40%, 50%, 60%, and 70% can be seen in Figure 4.



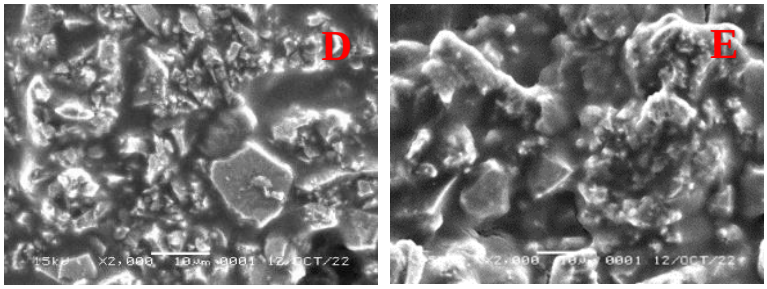


Fig 4. SEM Characterization Result Data from (a)30% (b)40% (c)50% (d)60% (e)70%

It can be seen in Figure 4, The results of the SEM characterization test of Fe_3O_4/PANi nanocomposite thin films with five variations and using 2000X magnification can be seen on the surface morphology of the samples. In each sample, it looks like lumps, there are cavities in several variations, and the surface is uneven. For each composition variation, there are lumps that are not evenly distributed, and there is a buildup of particles or substances together which is often referred to as agglomeration. The results of the morphology of the PANi/Fe_3O_4 composite showed that agglomeration occurred in several parts [20]. This is understandable because Fe_3O_4 is a ferromagnetic material, so it tends to attract each others between its particles [20]. To find out the average particle size for each composition variation, you can use the Analyze Particles feature in the Image-J application. In this study, the particle sizes obtained for each composition were 30%, 40%, 50%, 60%, and 70%, namely 27 µm, 53 µm, 61 µm, 84 µm, and 46 µm. It can be seen that the more addition of Fe_3O_4, the larger the particle size but at a variation of 70%, the particle size has decreased. Irregularity of particle size can be caused by the accumulation of several substances [21].

And the results of the characterization using a UV-Vis spectrophotometer of Fe_3O_4/PANi nanocomposite thin films aim to determine the absorbance, reflectance, transmittance, and energy gap values. For sample testing, the tool used is a UV-Vis Spectrophotometer with UV VIS 100 DA-X type, the lowest single bear wavelength is 190-210 nm, and the highest is 800-1000 nm, and sample testing is carried out at the Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences. Science is housed at Padang State University. The following is the result of the Fe_3O_4/PANi nanocomposite UV-Vis spectrophotometer characterization with five composition variations, namely 30%, 40%, 50%, 60%, and 70% for the absorbance values can be seen in Figure 5.

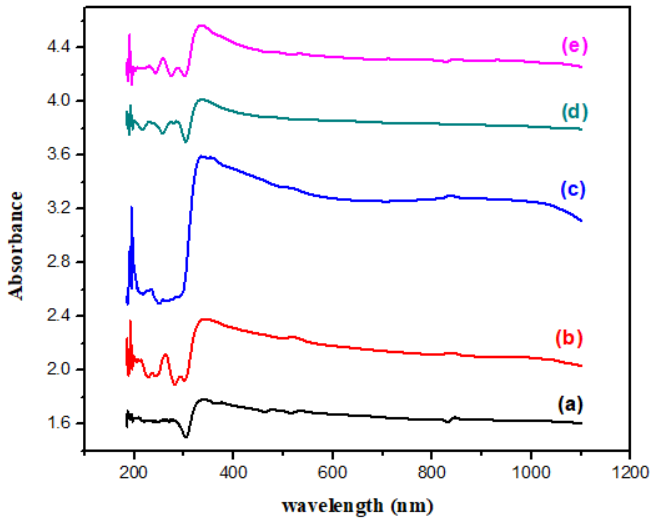


Fig 5. Absorbance of (a)30% (b)40% (c)50% (d)60% (e)70%

In Figure 5, it can be seen that the highest Fe_3O_4/PANi nanocomposite absorbance values for variations of 30%, 40%, 50%, 60%, and 70% were 1.78, 2.59, 3.32, 2.17, 2.32. If the greater the addition of Fe_3O_4, the greater the absorbance value, but at 60% variation, it decreases and increases again at 70% variation. Based on the results, the highest absorbance value was found in the 50% variation, namely 3.32 at a wavelength of 335 nm, this indicates that the absorbed light intensity is greater than other variations. At this maximum absorbance point, it indicates that electrons cannot absorb energy at that wavelength, so the power provided is only passed through [22]. At a wavelength of 335 nm, the maximum absorbance peak appears at a variation of 50%, indicating the presence of

light at the wavelength of UV (ultraviolet) light. The result of measuring absorbance against wavelength is that with increasing wavelength, the absorbance spectrum gets smaller [1]. For the wavelength range of ultraviolet (UV) light, namely 100-400 nm.

The following is the result of the characterization of the Fe_3O_4/PANi nanocomposite UV-Vis Spectrophotometer with five composition variations, namely 30%, 40%, 50%, 60%, and 70% for the reflectance values can be seen in Figure 6.

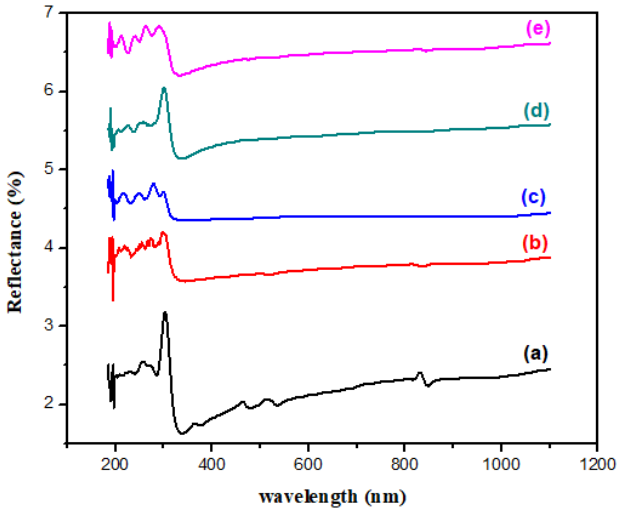


Fig 6. Reflectance of (a)30% (b)40% (c)50% (d)60% (e)70%

In Figure 6, it can be seen that the highest reflectance values of Fe_3O_4/PANi nanocomposites for variations of 30%, 40%, 50%, 60%, and 70% are 3.18 %R, 0.9 %R, 0.53 %R, 1.61 %R, 0.94 %R. Judging from the reflectance data on the Fe_3O_4/PANi nanocomposite, the greater the addition of Fe_3O_4, the lower the reflectance value, but there is an increase at 60% variation and a decrease at 70% variation. Based on the results, the highest reflectance value is found in the 30% variation, namely 3.18 %R at a wavelength of 303 nm, this indicates that the intensity of the reflected light is greater than other variations. At a wavelength of 303 nm, the maximum reflectance peak appears at a variation of 30%, indicating the presence of light at the wavelength of UV (ultraviolet) light. For the wavelength range of ultraviolet (UV) light, namely 100-400 nm.

The following is the result of the Fe_3O_4/PANi nanocomposite UV-Vis spectrophotometer characterization with five composition variations, namely 30%, 40%, 50%, 60%, and 70% for the transmittance values can be seen in Figure 7.

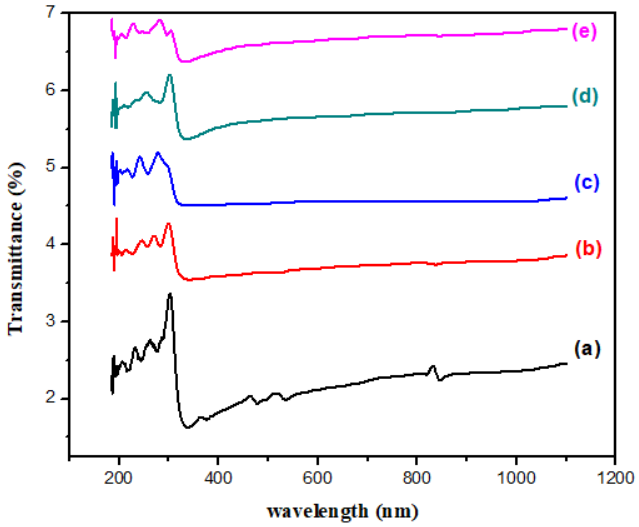
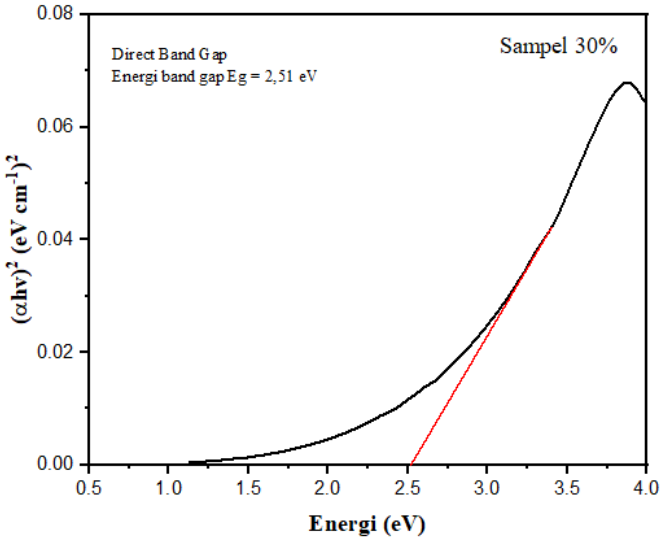


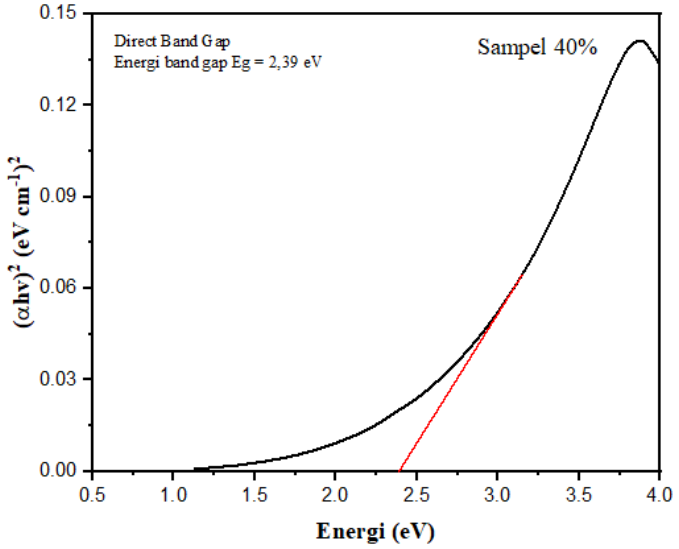
Fig 7. Transmittance of (a)30% (b)40% (c)50% (d)60% (e)70%

In Figure 7, it can be seen that the highest transmittance values of Fe_3O_4/PANi nanocomposites for variations of 30%, 40%, 50%, 60%, and 70% were 3.37 %T, 0.99 %T, 0.73 %T, 1.52 %T, 0.83 %T. Judging from the transmittance data on the Fe_3O_4/PANi nanocomposite, the greater the addition of Fe_3O_4, the lower the transmittance value, but there is an increase at 60% variation and a decrease at 70% variation. Based on the results obtained, the highest transmittance value is found in the 30% variation, namely 3.37 %T at a wavelength of 302 nm, this indicates that the intensity of the transmitted light is large compared to other variations. At a wavelength of 302 nm, the maximum transmittance peak appears at a variation of 30%, indicating the presence of light at the wavelength of UV (ultraviolet) light. For the wavelength range of ultraviolet (UV) light, namely 100-400 nm.

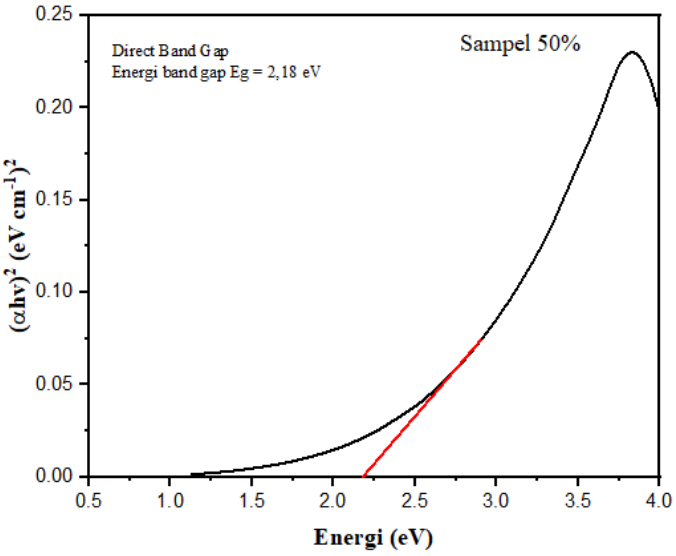
The following is the result of the Fe_3O_4/PANi nanocomposite UV-Vis spectrophotometer characterization with five composition variations, namely 30%, 40%, 50%, 60%, and 70% for the energy gap values obtained using the tauc plot method, which can be seen in Figure 8.



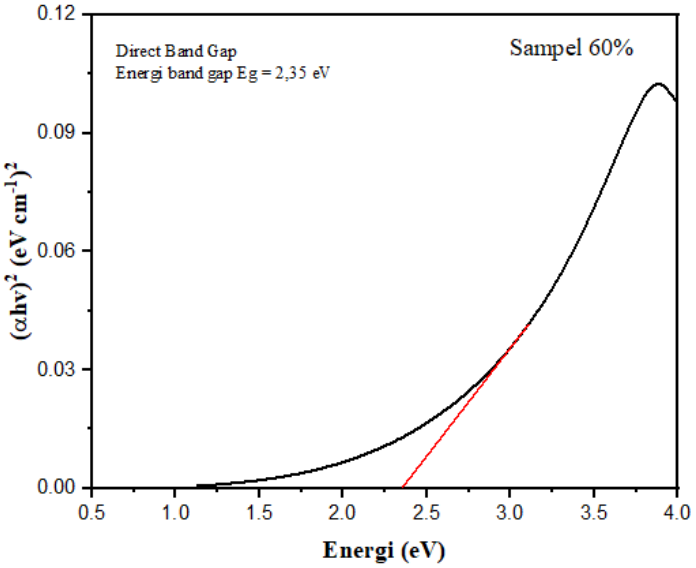
(a) 30% Fe_3O_4 - 70% PANi



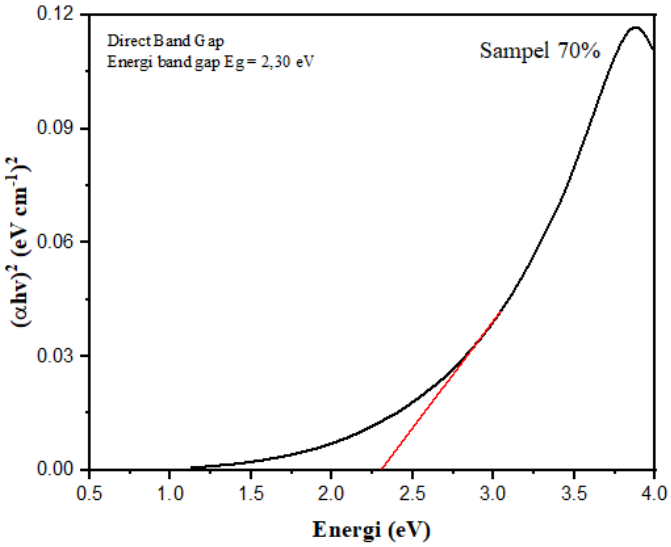
(b) 40% Fe_3O_4 - 60% PANi



(c) 50% Fe_3O_4 - 50% PANi



(d) 60% Fe_3O_4 - 40% PANi



(e) 70% Fe_3O_4 -30% PANi

Fig 8. Energy Gap of (a) 30% Fe_3O_4 - 70% PANi, (b) 40% Fe_3O_4 - 60% PANi, (c) 50% Fe_3O_4 - 50% PANi, (d) 60% Fe_3O_4 - 40% PANi, (e) 70% Fe_3O_4 - 30% PANi

In Figure 8, it can be seen that the energy values of the Fe_3O_4/PANi nanocomposite gap for variations of 30%, 40%, 50%, 60%, and 70% are 2.51 eV, 2.3 eV, 2.18 eV, 2.35 eV, and 2.30 eV. Based on the results obtained, the more the Fe_3O_4 composition, the lower the energy gap value. The higher the percentage of Fe_3O_4 composition in PANi, the smaller the energy gap obtained. This was also proven in research (Gomathi et al., 2018) that the addition of Fe_3O_4 nanoparticles to PANi caused the energy gap value to decrease [23]. The smaller the energy gap value, the smaller the energy band gap (between the conduction band and the valence band), so that electrons in the valence band more easily jump (excite) to the conduction band [22]. For each variation, it is concluded that the gap energy obtained is in the gap energy range of semiconductor materials. The range of semiconductor gap energy values is at a value of 0-3 eV [15].

IV. CONCLUSION

Based on the research that has been carried out regarding the analysis of the optical properties of the Fe_3O_4/PANi nanocomposite synthesized by the sol-gel spin coating method, it can be concluded that the XRD characterization results show that each addition of PANi into Fe_3O_4 does not affect the crystal structure and the average crystal size is below 100 nm so that the Fe_3O_4/PANi nanocomposite thin layer is nano-sized. For testing using SEM, the Fe_3O_4/PANi nanocomposite thin layer for all samples contained particles called agglomeration. Particle sizes at variations of 30%, 40%, 50%, 60%, and 70% were 27 µm, 53 µm, 61 µm, 84 µm, and 46 µm respectively. And for the value of the energy gap gain, the sample was tested using a UV-Vis Spectrophotometer, where the method used to obtain the energy gap is the tauc plot method. The application of Fe_3O_4/PANi nanocomposite thin films as a semiconductor is indicated by the energy gap value of each sample which has a value of < 3 eV. In semiconductor materials, the value of the energy gap is in the range of 0-3 eV. And also, the results obtained show that the increasing composition of Fe_3O_4, the lower the energy gap value.

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