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SYNTHESIS AND CHARACTERIZATION NOVEL EMERALDINE-SALT@PORANGCOMPOSITES THIN FILM:CONDUCTIVITY STUDIES

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RESEARCH ARTICLE

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Abstract

A synthesis of the emeraldine salt@porangcomposite was synthesized using the in situ chemical polymerization method to form the ES@Porang composite. Polymerization using ammonium peroxodisulphate as an initiator in the aniline oxidative polymerization process and using H_2SO_4 doping. The composite films were prepared with variations in dopant concentration and variations in the amount of glucomannan. Characterization of the ES@Porang composite using FTIR and conductivity testing using an LCR-meter. The optimum conductivity values of the ES@Porangcomposite thin film with variations in dopant concentration and amount of glucomannan were 24.3 x 10^{-3}, 58.2 x 10^{-3}, and 84,3 x 10^{-3} S/cm respectively.

Introduction:-

The sensor material is a material used as a medium to absorb water vapor. Sensor material that has absorbed water will change the electrical properties of the material. If the water vapor that has been absorbed is proportional to changes in its electrical properties, then this material can be used as an air humidity sensor material (Anggi et al., 2017**).**Cellulose is one of the materials used as a matrix in the process of making polyaniline composites because of its flexibility. The source of cellulose used in this study came from extracted bagasse. The choice of bagasse as a source of cellulose is due to its abundance and relatively high cellulose content. Bagasse is the remaining part of the sugarcane stem in the sugarcane extraction process which still contains moisture, fiber, and dissolved solids. In principle, bagasse fiber consists of cellulose, pentosan, and lignin, and the composition of the three components can vary in different sugarcane varieties (Anggi et al., 2017),The plant Porang" plant (Amorphophallus sp.) is a plant that lives in tropical forest and is widely found in Indonesia. "Porang" tubers contain a lot of glucomannan and are known as Konjac Glucomannan. Konjac Glucomannan is widely used as a "traditional" food in Asia such as noodles, tofu, and jelly. Some of the benefits ofkonjac flour are reducing blood cholesterol, and diabetics, as a substitute for agar-agar and gelatin (Aryanti et al., 2015). Based on (Athawale et al., 2006), nanocomposite Pd Polyaniline peaks at 3600-3000 cm^{-1} and 3000-2800 cm^{-1} correspond to the –NH and –CH at ES@Porang composites stretching vibration respectively. Bands due to stretching of aromatic CN vibrations appear at 1294 cm^{-1} while the absorption peaks at 1594 and 1490cm^{-1} represent the Quinoid(Q), and Benzenoid(B) structures of the emeraldine phase of ES@Porang.The plant Porang (Amorphophallus sp.) is a plant that lives in tropical forests andis widely found in Indonesia. "Porang" tubers contain a lot of glucomannan and are known as Konjac Glucomannan.

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Konjac Glucomannan is widely used as a traditional food in Asia such as noodles, tofu, and jelly. Some of the benefits of konjac flour are reducing blood cholesterol, and diabetics, as a substitute for agar-agar and gelatin (Aryanti et al., 2015). Polypyrrole is a typical conductive polymer functional material among many conduction polymers. Due to its unique properties, PPy has been widely applied, such as electrochemistry, electrode materials, optics, biotechnology, and conducting materials. However, the polypyrrole range of applications is limited in practice due to its non-melting, insoluble, and poor processing performance (Huixia et al., 2011).Based on (Athawale et al., 2006) Nanocomposite Pd Polyaniline peaks at 3600-3000 cm^{-1} and 3000–2800 cm^{-1} correspond to the –NH and –CH stretching vibration respectively. Bands due to stretching of aromatic CN vibrations appear at 1294 cm^{-1} while the absorption peaks at 1594 and 1490 cm^{-1} represent the Quinoid (Q) and Benzenoid (B) structures of the emerald phase of ES@Porangcomposite film. However, a comparison of ES@Porang composite film. However, a comparison of the FT-IR spectra of the exposed samples to the unexposed nanocomposites revealed two significant differences. The intensity as well as the sharpness of the peaks representing the NH and CH stretching vibrations were found to be enhanced (the effect became more pronounced at higher methanol concentrations, namely 2000 ppm). However, a comparison of the FT-IR spectra of the exposed samples to the unexposed nanocomposites revealed two significant differences. The intensity as well as the sharpness of the peaks representingthe NH and CH stretching vibrations were found to be enhanced (the effect became more pronounced at higher methanol concentrations, namely 2000 ppm). This could be due to the higher degree of interaction between these groups and the methanol molecule. Despite this, the quinoid peaks appear to shift by 40 cm^{-1}, from 1594 cm^{-1} to 1550 cm^{-1} on exposure to methanol vapor. This can be attributed to the interaction of methanol molecules with nitrogenous imines, causing a reducing effect. the effective positive charge on the imine nitrogen is reduced by the methanol molecule in the presence of Pd-nanoparticles by converting the imine nitrogen into amines, ie benzene. In this paper, Pd-polyaniline (Pd-PANI) nanocomposites have been used as sensing materials for nanotechnology which has become an area of research impetus, various alcohol vapors. Polyaniline can be used as a moisture sensor material because it has a good conductivity value, but because polyaniline is hydrophobic, it must be composited with hydrophilic or water-loving materials. Cellulose is a hydrophilic material, so it can increase the sensitivity of polyaniline-based moisture sensors. (Widiyanti et al., 2018) and the oxidized and reduced states of PAni, and their derivatives, are represented by a reduced “y” index unit (benzenoid ring) and an oxidized “1 – y” unit (quinoid ring),as in Figure 1.below: (Mazzeu et al., 2018)

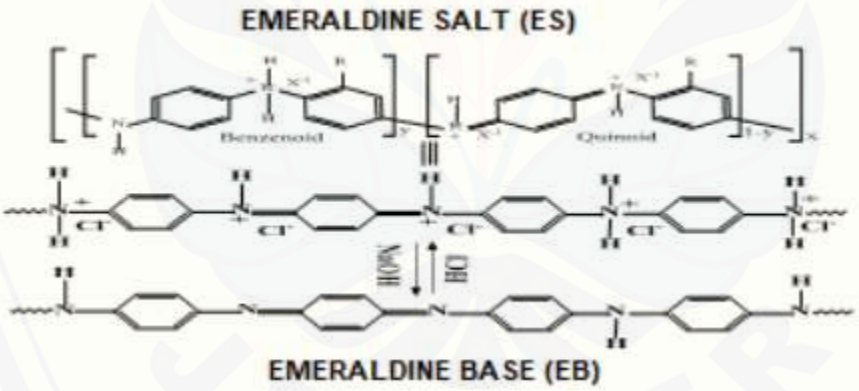

Figure 1:- Structure of Emeraldine Salt@Porang

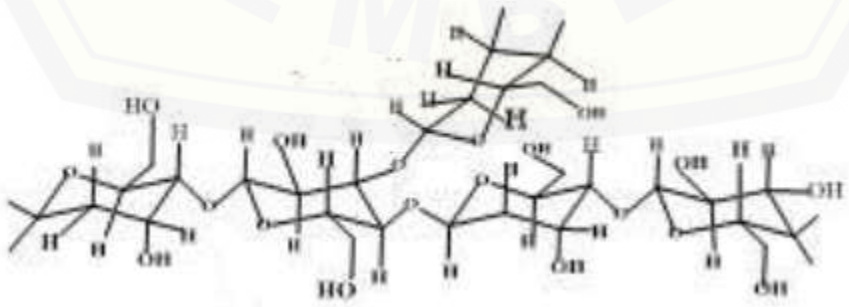

Figure 2:- Structure of Porang/Glucomannan.

Polyaniline has the advantages of being easy to synthesize, stable in the environment, and resistant to corrosion (Huixia et al., 2011). that the ES@Porangcomposite filmmodel can be explained because of the hydrogen bonding between the hydroxyl groups of imine and Amina including inthe ES@Porangcomposite filmas shown in Figure3 below:

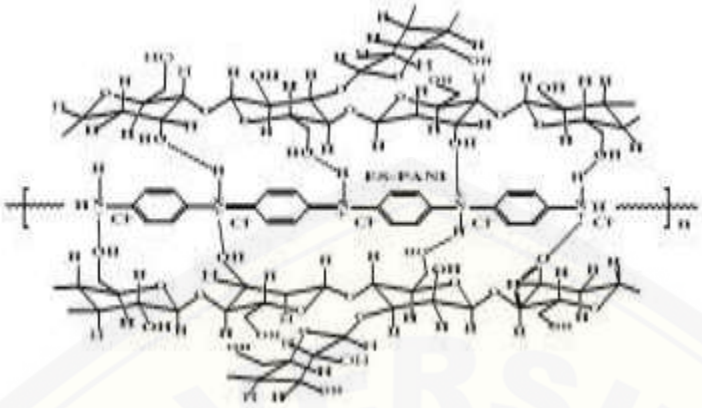

Figure 3:- ProposedES@Porang Composites Thin Film.

Increasing the conductivity of polyaniline can be done by adding dopant compounds by reducing the electron density in the polyaniline chain to obtain polyaniline with a high electron distribution. Dopants are additional compounds used to increase conductivity, one of which is polyaniline. Glucomannan is cellulose that is formed in the "porang" plants (Aryanti, et al, 2018). In addition, cellulose can interact with polymers to form new bonds through hydrogen bonds found in glucomannan, making it possible as a composite matrix with polyaniline. Polyaniline composites use the in situ polymerization method. So that this research will develop a study of the effectof variations in the amount of glucomannan and variations in dopant concentration on the conductivity of the ES@Porangcomposites. The results of the synthesis are characterized by electrical properties using an LCR meter and structural characteristics using FT-IR

Methodology:-

Materials:

The equipment used includes beakers, Erlenmeyer cups, volumetric flasks, stirring rods, dropper pipettes, Mohr pipettes, ball pipettes, desiccators, glass funnels, scissors, and shakers. The characterization tools include LCR meters and FT-IR, SEM, The materials used include aniline, Porang flour, chloroform, distilled water, acetone, sulfuric acid, ammonium peroxodisulphate

Synthesis of Porang Film

Glucomannan film was prepared by dissolving serials 1.5 g, 2.5 g, and 3.5 g of Glucomannan flour ("porang" flour) in 100 mL of water. This mixture is then heated for 45 minutes at 90℃. The glucomannan solution that has been heated for 45 minutes is then printed with a certain thickness. The film was left for 2 hours and then dried at room temperature for 24 hours. The dried film was then cut into 4 x 4 cm sizes

Synthesis of ES@PorangComposite Thin Film

The first glucomannan thin film was soaked for 15 minutes, then batched in a solution of ammonium peroxodisuphate (APS) with a concentration of 1 M as much as 15 mL for 15 minutes, then the film was put in and then immersed in various H_2SO_4 concentrations of 0; 1; 1,5, 2 and 2,5 M as much as 15 mL for 30 minutes. The oxidized film was batched in 15 mL of aniline monomer for 15 minutes. Second, polymerization was carried out based on research from (Busroni et al., 2022) with aniline, H_2SO_4, and APS ratio of 1:1, 2:1.

Characterization of ES@PorangComposite Thin Film

Characterization ofES@Porang composites thin film, the chemical structures of the ES@Porangcomposites were studied using Fourier Transform Infrared (FTIR) spectroscopy, Scanning Electron Microscopy was used to analyze the morphology and the composition of the specimens. The electrical quantities and the conductivity of specimens both use LCR-Meter

Result and Discussion:-

Synthesis of ES@Porang Composite Thin Film

The method used is in situ oxidative polymerization (Busroni, et all., 2022) where the polymerization is carried out in stages from aniline monomers to obtainthe ES@Porangcomposite. Immersion of Porang film in a mixture of APS and H_2SO_4. Then after soaking with aniline monomer for 30 minutes, a purple to dark green color change occurred, tending to black on all parts of the glucomannan thin film. This change of purple to dark-green color change indicates that the polymerization of aniline has occurred to become ES@Porangcomposite in the form of emeraldinesalt. The resulting material obtained after polymerization was then washed with 3 x 10 mL acetone and dried at room temperature for 2 x 24 hours to reduce the water content in the ES@Porangcomposite film. The mechanismfor the oxidation reaction of polyaniline polymerization can be shown in Figure 4 (Busroni et al., 2022) below:

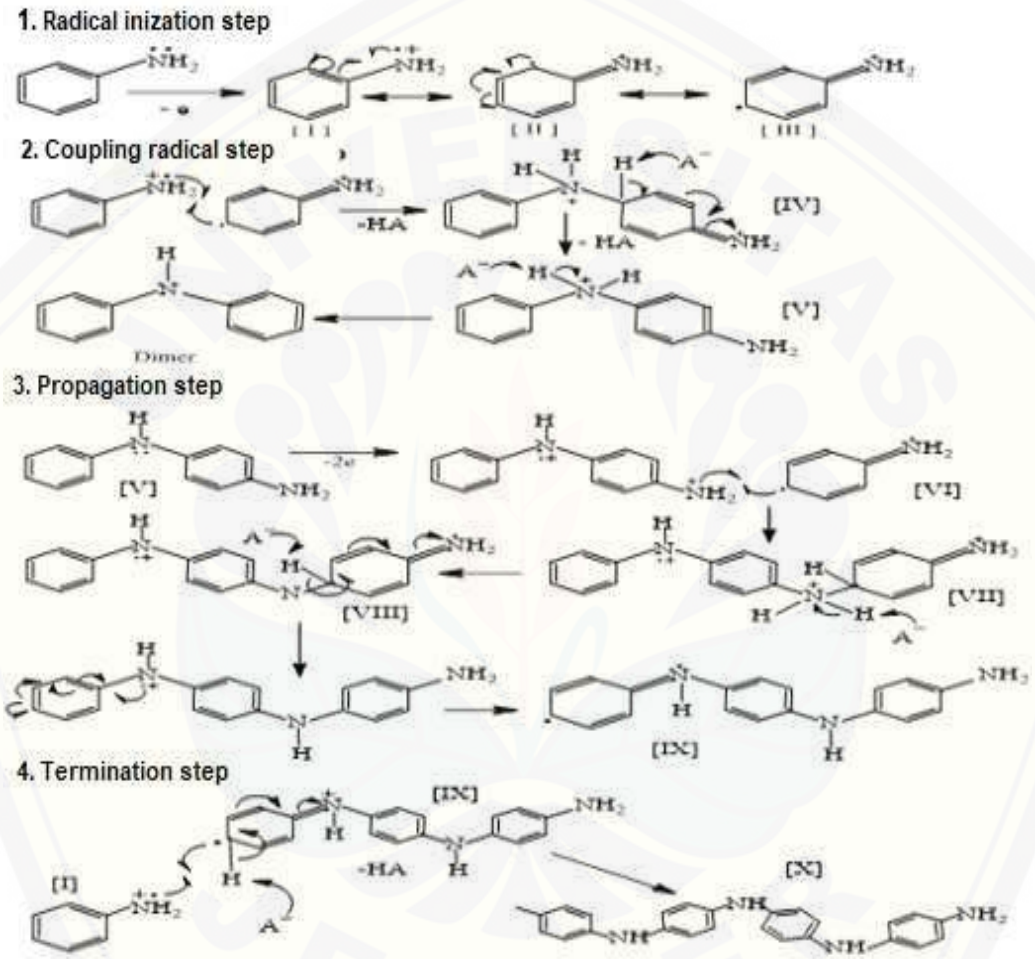

Figure 4:- Mechanism: Polymerization from monomer of aniline(Busroni et al., 2022).

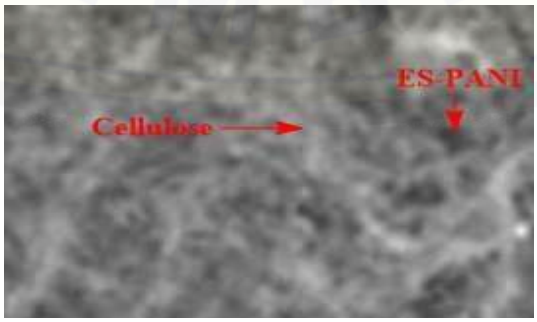

Figure 5:- Spectra SEM of ES@Porang composite thin film.

The results of the characterization Figure 5 of the ES@Porangcomposite film with SEM showed that the glucomannan or cellulose surface was covered by polyaniline in the form of a thin film. SEM micrograph of the ES@Porangcomposite films shows that the synthesized composite has an aggregate structure that matches that reported by Yusningsih et al., 2018.Analysis of composite used FTIR (Fourier Transform InfraRed) was used to study and observe the effect of immersion time in a mixture of APS and H_2SO_4 on each functional group belonging toES@Porangcomposite composites Prior to this step, it is necessary to analyze the absorption peaks contained in glucomannan as a composite matrix, after the synthesis of theES@Porang composites that have been formed. There was a change in the absorption peak at 3340 cm^{-1} of the hydroxy group in glucomannan which experienced a broadening of the spectrum to 3220 cm^{-1}. This indicates that there has been an interaction between the positively charged nitrogen atoms inES@Porangcomposite composites and the hydroxyl groups in Glucomannan, resulting in a broadening of the absorption peak but with a sloping transmittance. changes also occur with the appearance of a typical ES@Porangcomposite peak at 1580 and 1450 cm^{-1} which means the presence of quinoid and benzenoid groups. While the peak of 1042 cm^{-1} is the ether peak as a glycosidic bond in Glucomannan. The influence of the Batch method on the mixture of APS and H_2SO_4 requires a qualitative analysis of the peak absorption of the relative quinoid (Q) and benzenoid (B) compositions.Figures 6, Figure 7, Figure 8, Figure 9, Figure 10, Figure 11, Figure12, and Figure 13 below, show the FTIR spectra of theES@Porangcompositedoped with H_2SO_4 with quinoid (~1580cm^{-1}) and benzenoid (~1450 cm^{-1}). Through qualitative analysis, data on the relative composition (Aquinoid/Abenzenoid) to conductivity was obtained according to the facts in Table 1.

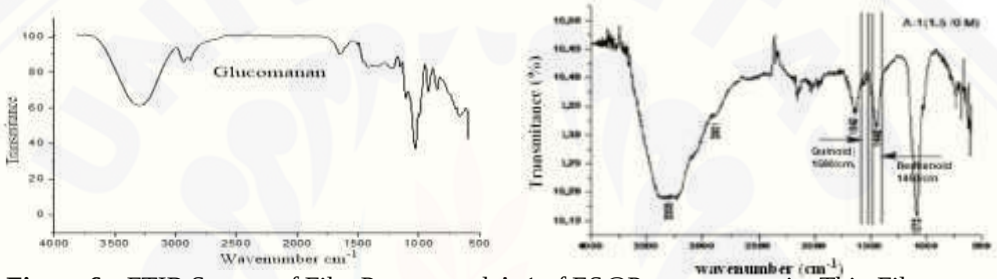

Figure 6:- FTIR Spectra of Film Porang, and A-1 of ES@Porangcomposite Thin Film

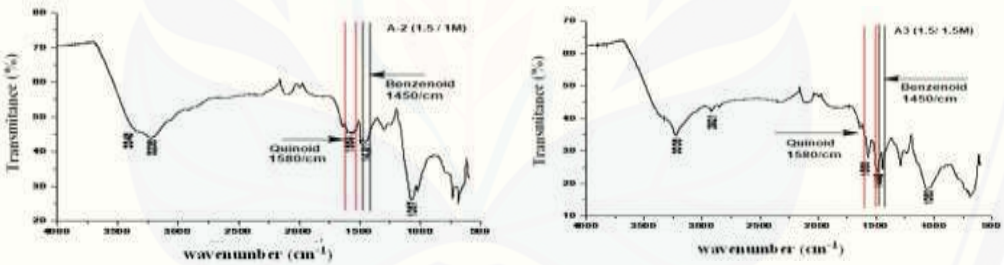

Figure 7:- FTIR Spectra A-2, and A-3ofES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$)(Dopant H_2SO_4).

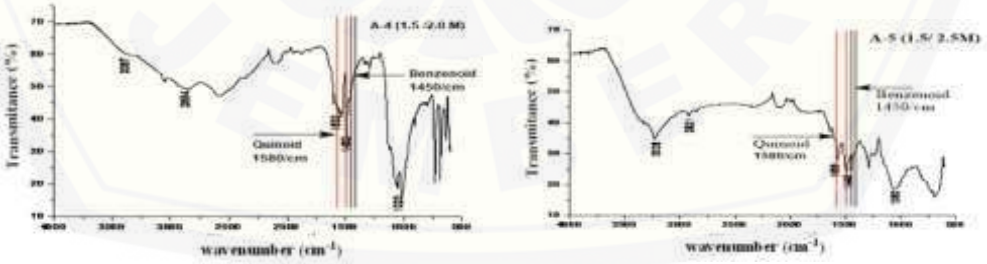

Figure 8:- FTIR Spectra A-4, and A-5 of ES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$) (Dopant H_2SO_4)

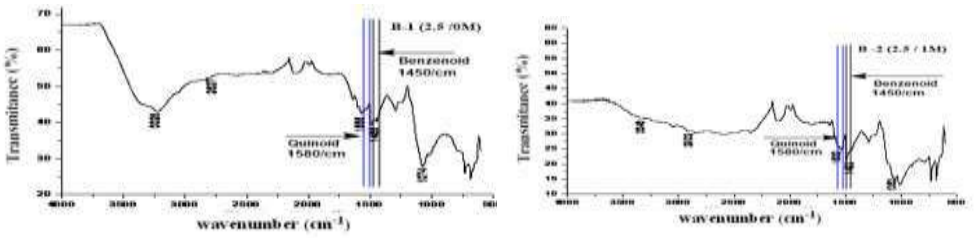

Figure 9:- FTIR Spectra B-1, and B-2ofES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$) (Dopant H_2SO_4).

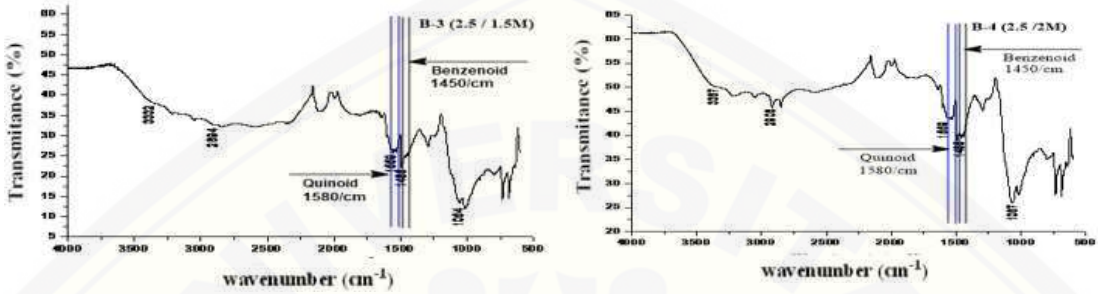

Figure 10:- FTIR Spectra B-3, and B-4 ofES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$) (Dopant H_2SO_4).

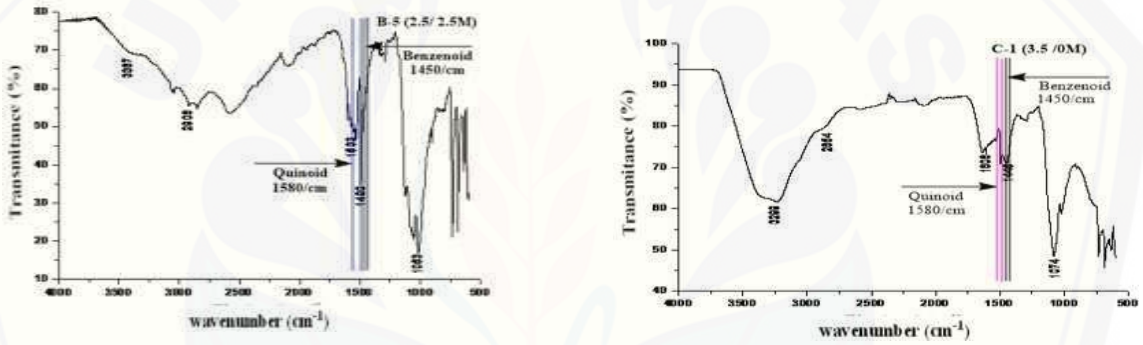

Figure 11:- FTIR Spectra B-5, and C-1 ofES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$) (Dopant H_2SO_4)

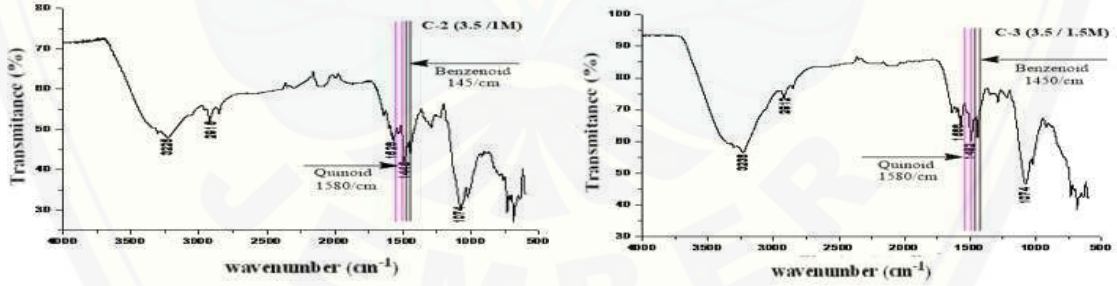

Figure 12:- FTIR Spectra C-2, and C-3 of ES@PorangComposites Thin Film ($A_{Quinoid}/A_{Benzenoid}$) (Dopant H_2SO_4) .

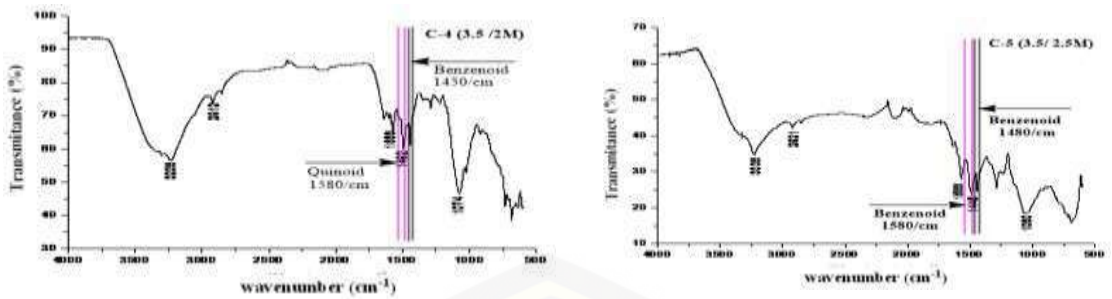

Figure 13:- FTIR Spectra C-4, and C-5 of ES@PorangComposites Thin Film($A_{Quinoid}$**/**$A_{Benzenoid}$**)** (Dopant H_2SO_4).

The results of the FTIR spectrum ofES@Porangcomposite thin film have the following peaks: at Figure 6, Figure 7, Figure 8, Figure 9, Figure 10, Figure 11, Figure 12, and Figure 13, while the ES@Porangcomposite with the concentration of 2.5M showing peaks at 3220.39 cm^{-1}; 1637.70 cm^{-1},1580.92 cm^{-1}; 1450.14 cm^{-1}; 1440.95 cm^{-1}; 1288.79 cm^{-1}; 1187.11 cm^{-1}; 1050.92 cm^{-1}; and 739.83 cm^{-1}; The composite FTIR spectrum contained a peak of 3401.12 cm^{-1}; as a stretch of O-H and a peak of 3220.39 cm^{-1}; as a type of bonding of N-H. The O-H stretching peak is the identity of the glucomannan cellulose present in the composite and the N-H peak is evidence of polyaniline inclusion in the composite matrix. Based on Athawale et al., 2006, the peaks at 3600–3000 cm^{-1},and 3000–2800 cm^{-1}correspond to the stretching vibrations of –N H and –CH PANI respectively. Bands due to aromatic stretching of CN vibrations appear at 1294 cm^{-1}, while the adsorption peaks at 1584 and 1450 cm^{-1} represent the Quinoid (Q), and Benzenoid (B) of the emerald phase ofES@Porang. The FTIR spectra of the ES@Porangcomposite doped with H_2SO_4 showed the presence of quinoid and benzenoid peaks is the main constituent of polyaniline which plays an important role in its electrical properties. However, the addition of variations in the concentration of dopant H_2SO_4 will affect the relative composition of the quinoid and benzenoid rings inES-PANI@Glucomannan composites. According to the dopant reaction mechanism with polyaniline, the imine site on the quinoid peak allows protonation by a strong acid. Protonation by variations in the concentration of strong acids at imine sites accompanied by increasing changes in the electrical properties ofES@Porangcomposite. The greater the relative composition ($A_{quinoid}/A_{benzenoid}$) in the polymer chain. the greater the conductivity according to the result of research by (Yuningsih et al., 2018, and Busroni et al., 2022) However, not all reactions between dopants and the number of imine sites can form quinoid structures. This is influenced by the acid concentration and soaking time (Busroni et al., 2022).Based on (Anggi et al., 2018;Yuningsih et al., 2018; Busroni et al., 2022), an increase in the dopant concentration of H_2SO_4in polymerization is proven to increase the conductivity value caused by an increase in the ratio ($A_{quinoid}/A_{benzenoid}$) as shown in Figure 5, Figure 6, Figure 7, Figure 8, Figure 9, Figure 10, Figure 11, and Figure 12, effects of variation of H_2SO_4 dopant to the structure of emeraldine salt and the increase in conductivity is higher by dopant which is a strong acid, the increase in conductivity values can be seen at Table 1. Based on research conducted by (Yuningsih et al., 2018., Busroni, et al., 2022), when using acid doping with strong acids at a concentration of 0 until 2,5 M H_2SO_4 the conductivity decreases and the polyaniline hydrolysis reaction occurs, because the system has the ability to absorb 100% of waters from the thin layer, thus allowing the occurrence of thermodynamic equilibrium in solution using room temperature, the more water content is substituted by APS and dopant. In this study, the cellulose from the glucomannan film was immersed in a mixture of APS and dopant of H_2SO_4 for 30 minutes to replace the water content in glucomannan and H_2SO_4so that the conductivity would be higher

Table 1:- Effect Mass of Porang, and Concentration of Dopant Onto of ES@Porang Composites Thin Film.

Variation Concentration of Dopant H_2SO_4 (M)	Conductivity Value (S/cm)		
	Variation Amount of Glucommanan (Dopant H_2SO_4)		
	Composites of Film A (1,5 g of Glucomannan) S/cm	Composites of Film B (2.5 g of Glucomannan) S/cm	Composites of Film C (3.5 g of Glucomannan) S/cm
0	6.81×10^{-5}	7.09×10^{-5}	8.26×10^{-5}
1	17×10^{-3}	21.2×10^{-3}	26.9×10^{-3}
1.5	21×10^{-3}	27.8×10^{-3}	31.4×10^{-3}
2.0	$24{,}1 \times 10^{-3}$	48.5×10^{-3}	50.7×10^{-3}
2.5	24.3×10^{-3}	58.2×10^{-3}	84.3×10^{-3}

First, increasing the mass of glucomannan, and in the polymerization will increase the degree of conductivity caused, second the increasing concentration of dopant equals increasing of protons (H^+) by the filled H_2SO_4 as a dopant to the emeraldine salt structure and high concentration of dopant rather than low concentration of dopant, the increase in the conductivity value can be seen in Table 1. according to research conducted by (Anggi, et al., 2018; Yuningsih, et al., 2018; Busroni, et al., 2022), Table 1, has been shown, the highest conductivity value in this study in the batch system was at mass glucomannan 1,5 g, 2,5 g, and 3,5 g using doping H_2SO_4 of 24,3 x 10^{-3}, 58,2 x 10^{-3}, and 84,3 x 0^{-3} S/cm in respectively has been shown in Table 1 Synthesis of the ES@Porangcomposite film obtained emeraldine salt and along with an increasing mass of glucomannan (1.5 g; 2,5 g, and 3,5 g mass of glucomannan), the conductivity value was higher. Judging from the range of conductivity values,ES@Porang is a conductor. This result is supported by the FTIR characterization in Figure 6, Figure 7, Figure 8, Figure 9, Figure 10, Figure 11, Figure 12, and Figure 13, which shows, that in the sample with the addition of 3.5 g of glucomannan mass the ES@Poranggraft will result in more interactions with amine and imine groups, so the resulting polymer chain will be longer. The longer the polymer chain. the more filler the binding matrix. The more charge carrier ions so the conductivity values of the material increase. In Figure 6, Figure 7, Figure 8, Figure 9, Figure 10, Figure 11, Figure 12, and Figure 13, it can be seen that qualitatively the amount relative of ($A_{Quinoid}/A_{Benzenoid}$) groups has high in theES@Porang film form using high concentration H_2SO_4 which is of higher rather than using low concentration H_2SO_4, and the amount of glucomannan mass and the amount relative of ($A_{Quinoid}/A_{Benzenoid}$) is highest This conditiongreatly affects the conductivity values of the ES@Porang composite thin film. Due to the relative quality of the best ($A_{Quinoid}/A_{Benzenoid}$) using a high concentration of the doped and high amount of mass Glucomannan is to contributingto increasing conductivity

Conclusion:-

Synthesis of ES@Porangcomposite film from glucomannan with the variation of dopant $H_2SO_4$and variation ofthe amount of glucomannan is a high concentration of dopant and high amount of glucomannan, the produces higher conductivity. The composite films were prepared with variations in dopant concentration and variations in the amount of glucomannan. Characterization of the composite using FTIR and conductivity. The optimum conductivityvalues of the ES@Porangcompositefilm with variations in dopant concentration and amount of glucomannan were 24.3 x 10^{-3}, 58.2 x 10^{-3}, and 84,3 x 10^{-3} S/cm respectively.

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