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RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF NOVEL ES-PANI/GLUCOMANNAN COMPOSITE: STUDY OF CONDUCTIVITY

Busroni Busron¹, Anggoro Wicaksono¹, Moh Irfan Efendi¹, Devi Nalita¹, Safna Aullia Intan Mawarni¹, Arum Prasanty¹, Bambang Hidayat¹, Imaniah Wiyono Saputri¹, Bambang Piluharto¹, Dwi Indarti¹ and Chairil Anwar²

1. Departement of Chemistry, Faculty of Mathematics and Natural Sciences, Jember University
Jl. Kalimantan 37, Jember 681752.
2. Departement of Chemistry, Faculty of Mathematics and Natural Sciences, GadjahMada University Jl. Sekip Utara, Yogyakarta 55281.

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Abstract

The synthesized of emeraldine-salt-polyaniline composites were synthesized using in situ chemical polymerization to form the ES-PANI-glucomannan composites and the ES-PANI-cellulose composites using the batch, and spray method. Polyaniline can be composed of glucomannan and cellulose matrices because they are abundant, cheap, and environmentally friendly. The Ammonium peroxodisulfate (APS) is used as an initiator for the process of oxidative polymerization of aniline and using HCl and HCOOH doping. Characterization of ES-PANI-Glucomannan and ES-PANI-Cellulose composites using FT-IR and conductivity test using LCR-meter. The ES-PANI-Glucomannan composite used the HCl doped batch method, and the conductivity values optimum were $3,68 \times 10^{-3}$ S/cm, the ES-PANI-Glucomannan composites used the HCOOH doped batch method, the conductivity values optimum were $5,87 \times 10^{-4}$ S/cm, the ES-PANI-Cellulose composites used the HCl doped batch method, the conductivity values were $1,08 \times 10^{-1}$ S/cm, the ES-PANI-Glucomannan composites used the HCl doped spray method, the conductivity values optimum were $1,23 \times 10^{-7}$ S/cm, and the ES-PANI-Glucomannan composites used the HCOOH doped spray method, the conductivity values optimum were $9,20 \times 10^{-8}$ S/cm. The results of the study showed that the highest conductivity values were found for the ES-PANI-Glucomannan and ES-PANI-Cellulosa composites in the batch method of HCl, and HCOOH doping.

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Introduction:-

The “porang” plant (*Amorphophallus* sp.) is a plant that lives in tropical forests and is widely found in Indonesia. “Porang” tubers contain a lot of glucomannan and are known as Konjac Glucomannan (KGM). Konjac Glucomannan is widely used as a traditional food in Asia such as noodles, tofu, and jelly. Some of the benefits of konjac flour or KGM 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.

Corresponding Author:- Busroni Busron

Address:- Departement of Chemistry, Faculty of Mathematics and Natural Sciences, Jember University Jl. Kalimantan 37, Jember 681752.

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 $3600\text{--}3000\text{ cm}^{-1}$ and $3000\text{--}2800\text{ cm}^{-1}$ correspond to the --NH and --CH ES-PANI 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-PANI. However, a comparison of ES-PANI. 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). 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)

Figure 1:- Structure of Emeraldine Salt-Polyaniline (ES-PANI).

Figure 2:- Structure of Glucomannan

Polyaniline has the advantages of being easy to synthesize, stable in the environment, and resistant to corrosion (Huixia et al., 2011). that the glucomannan-ES-polyaniline composite model can be explained because of the hydrogen bonding between the hydroxyl groups of imine and Amina including in ES- polyaniline, as shown in Figure3 below:

Figure 3:- ProposedES-PANI/GlucomannanComposites Film.

The conductivity of polyaniline ranges from 1-100 S/cm, which is relatively low compared to the conductivity of other conductive polymers (Bhadra et al., 2009). Increasing the conductivity of polyaniline can be done by adding dopant compounds by reducing the electron density in the polyaniline chain so that polyaniline with a high electron distribution is obtained. Dopants are additional compounds used to increase the conductivity of conductive polymers, one of which is polyaniline. Generally, the dopant compounds used are strong acids that can donate or donate protons. Some dopant compounds that are often used in the polymerization of polyaniline are hydrochloric acid, sulfuric acid, phosphoric acid, and several types of metals (Jarad et al., 2016). Bacterial cellulose is cellulose formed from the secretion of the bacterium *Acetobacter xylinum*. Bacterial cellulose has a high surface area and porosity (Chawla et al., 2009). In addition, cellulose can interact with polymers to form new bonds found in cellulose fibers to allow cellulose as a composite matrix with polyaniline (Wang et al., 2012). polyaniline using in situ polymerization synthesis. So that the current research will develop a study on the effect of cellulose thickness and dopant type on the conductivity of PANI/BC composites. The results of the synthesis were characterized by electrical properties using an LCR meter and electrical structural characteristics using FT-IR

Methodology/ Approach:-

Materials:-

The equipment used includes beakers, Erlenmeyer glasses, measuring flasks, stirring rods, simple presses, dropper pipettes, Mohr pipettes, ball pipettes, desiccators, glass funnels, scissors, and automatic shakers. Characterization tools include an LCR meter as well as a set of Fourier Transmittance Infra-Red (FTIR) tools. The materials used include aniline, Tepung Glukomanan, chloroform p.a, ammonium Ammoniumperoxodisulfate p.a, distilled water, and hydrochloric acid (HCl), HCOOH, acetone, NaOH.

Synthesis of Glucomannan Film

Glucomannan film was prepared by dissolving 1.5 g; 2.5 g; 3.5 g of porang tuber flour in 100 mL of water. The mixture was then added with 5 mL of glycerol. This mixture is then heated for 45 minutes at 90°C. The glucomannan solution has been heated for 45 minutes is then printed with a certain thickness. The film was allowed to stand for 2 hours and then dried at 50°C for 24 hours. The dried film was then cut into 4 x 2.5 cm sizes.

Synthesis of ES-PANI/Glucomanan Composite Film using Batch Method.

The first glucomannan film was soaked for 15 minutes, then immersed in a solution of ammonium peroxodisulfate (APS) with a concentration of 1 M in the amount of 15 mL for 15 minutes, then the glucomannan film was put in and then immersed in dopant of HCl 1,2 M in the amount of 15 mL for 30 minutes. The oxidized film was then immersed in 15 mL of aniline monomers for 15 minutes. Second, polymerization was carried out based on research from (Busroni, et al., 2022 and Wang, et al., 2012) with the comparison of aniline: HCl: APS is 1:1,2:1. The soaked film was then shaken for 30 minutes. The soaked composite film was washed with 3 x 10 mL acetone and dried at room temperature for 2 x 24 hours then composites were characterized by FTIR and conductivity tests

Synthesis of ES-PANI/Glucomanan Composite Film using Spray Method.

The glucomannan films were soaked for 15 minutes, then immersed in ammonium peroxodisulfate (APS) solution with a concentration of 1 M in a volume of 15 mL for 15 minutes, then sprayed with Aniline (1 M) monomers with a total of 15 sprays in 1 minute on one side for 30 minutes. refer to (Shi et al., 2012). The composite that had been sprayed with 1 M aniline monomer was then washed using acetone. The soaked composite film was washed with 3 x 10 mL acetone and dried at room temperature for 2 x 24 hours. The composites were characterized by FTIR and conductivity tests

Characterization of ES-PANI-Glucomanan Composite Film

Characterization of ES-PANI-Glucomannan composites Film, the chemical structures of the ES-PANI-Glucomannan composites 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-PANI/Glucomanan Composite Film

The method used is in situ oxidative polymerization (Busroni, et al., 2022) where the polymerization is carried out in stages from aniline monomers to obtain ES-PANI/Glucomannan. The film glucomannan immersion in a mixture of APS and HCl or HCOOH does not show any color change. However, after soaking with aniline monomer for 30

minutes, there was color purple changed to dark green, tending to black in all parts of Glucomannan. This color purple change to dark green indicates that aniline polymerization has taken place into ES-PANI/Glucomannan in the form of emeraldine salt. The resulting material obtained after polymerization was then dried at room temperature for 2 x 24 hours to reduce the moisture content in the ES-PANI/Glucomannan composite Film. The reaction mechanism of polyaniline polymerization by oxidation can be shown in Figure 4. below. Proposed Mechanism Polymerization of Polyaniline Oxidative (Busroni, et al., 2022) below:

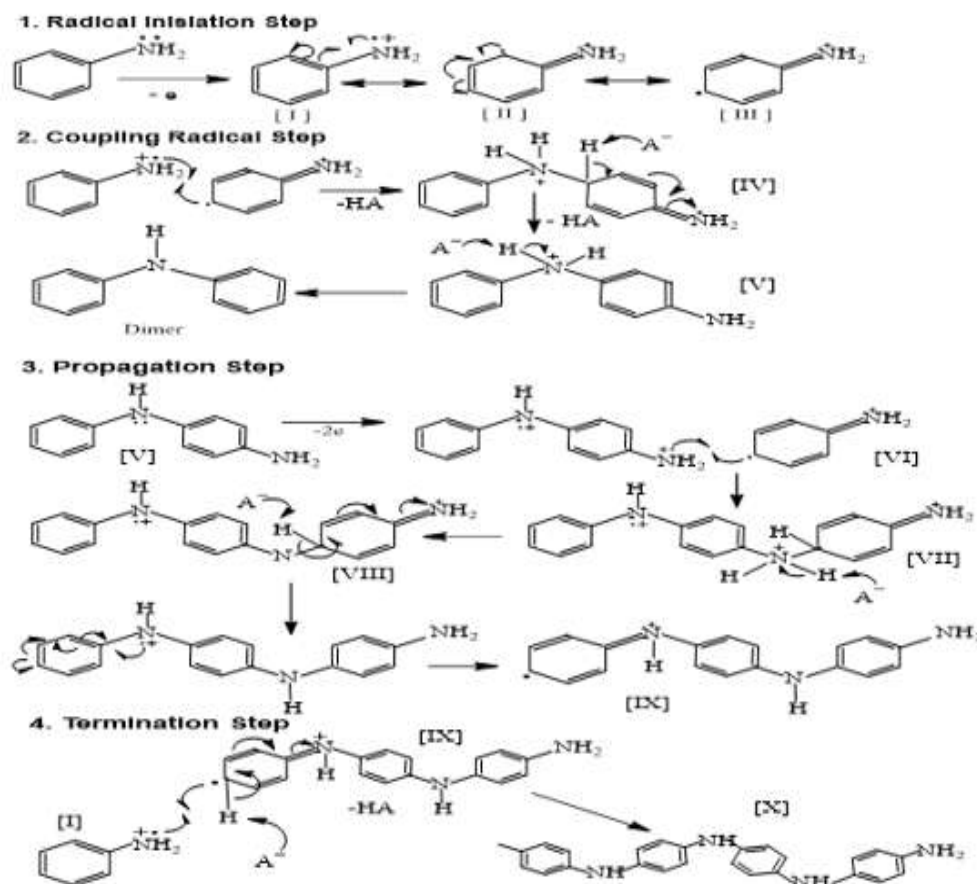


Figure 4:- Mechanism: Polymerization of Polyaniline Oxidative (Busroni et al., 2022).

Analysis of FT-IR spectra (Fourier Transform Infra Red) of composites: FTIR analysis was used to study and observe the effect of immersion time in the mixture of APS and HCl or HCOOH for each functional group possessed by ES-PANI. Before this step, it is necessary to analyze the changes in the absorption peaks contained in Glucomannan as a composite matrix, after the synthesis of the ES-PANI/Glucomannan composites. Referring to table 1 contains data on the absorption peaks of each type of vibration and Figure 4 Mechanism: Polymerization of Polyaniline Oxidative (Busroni et al., 2022) shows Figure 5 the FT-IR spectra of Glucomannan and PANI without dopants. There was a change in the absorption peak at 3340 cm^{-1} belonging to Glucomannan which experienced a widening of the spectra to 3220 cm^{-1} . This indicates that there has been an interaction between the nitrogen-have positive charge atom in ES-PANI, and the hydroxyl group of Glucomannan, resulting in a widening of the absorption peak but with a transmittance that tends to be sloping. Changes also occurred with the appearance of typical ES-PANI peaks at 1590 cm^{-1} and 1490 cm^{-1} which indicated the presence of quinoid and benzenoid groups. Meanwhile, the absorption peak is 1062 cm^{-1} which is ether as a glycoside bond in Glucomannan. On the influence of Batch and Spray methods of the mixture of APS and HCl or HCOOH quantitative analysis is needed on the absorption relative peaks of quinoids (Q) and benzenoids (B). This is necessary to determine the effect of the structure ES-PANI formed due to the Batch and Spray methods, where the effect comparison of quinoid and benzenoid composition affects the electrical properties of polyaniline (Hatchett, 1999). Figure 6, Figure 7, Figure 8, Figure 9, and Figure 10 shows the FTIR spectra of the ES-PANI/Glucomannan composite doped with HCl and

HCOOH with quinoid ($\sim 1590\text{ cm}^{-1}$) and benzenoid ($\sim 1490\text{ cm}^{-1}$) moieties. Through qualitative analysis of transmittance, data on the composition of quinoid and benzenoid on the conductivity produced by each Batch and Spray method were obtained

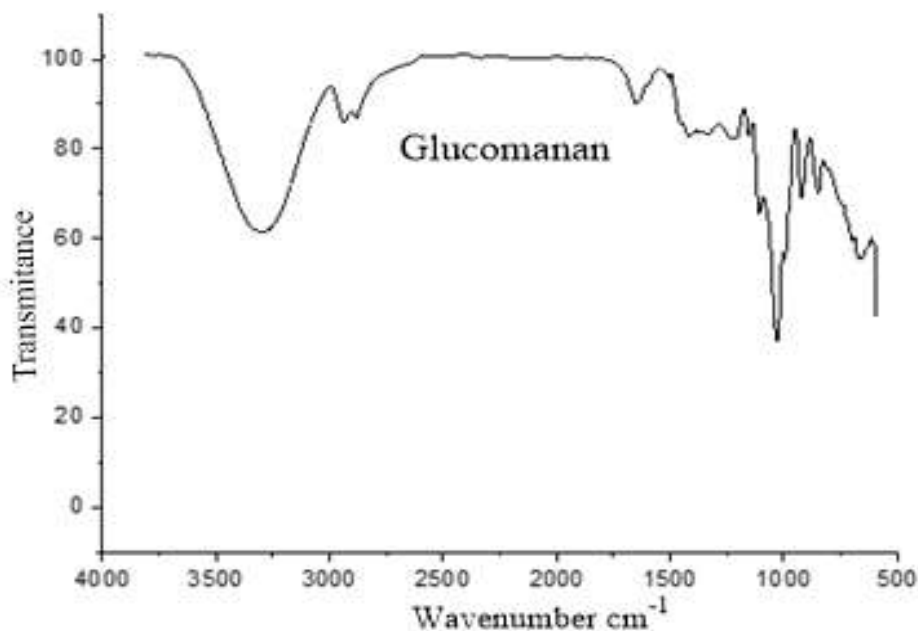


Figure 5:- FTIR Spectra of Film Glucomannan.

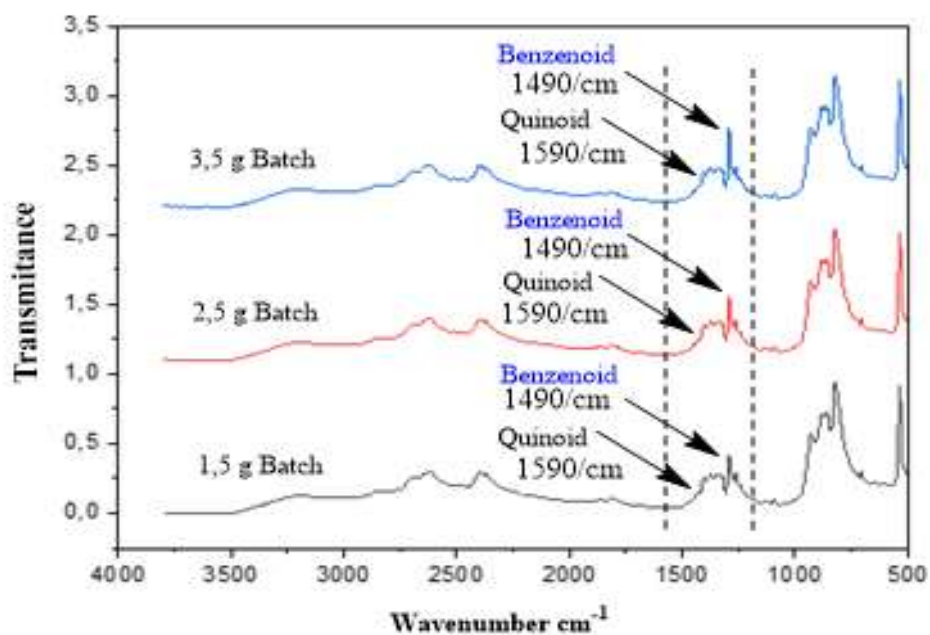


Figure 6:- FTIR Spectra of Glucomannan-ES-PANI Composites Film (Quinoid /Benzenoid) use Batch Method (Dopant HCl).

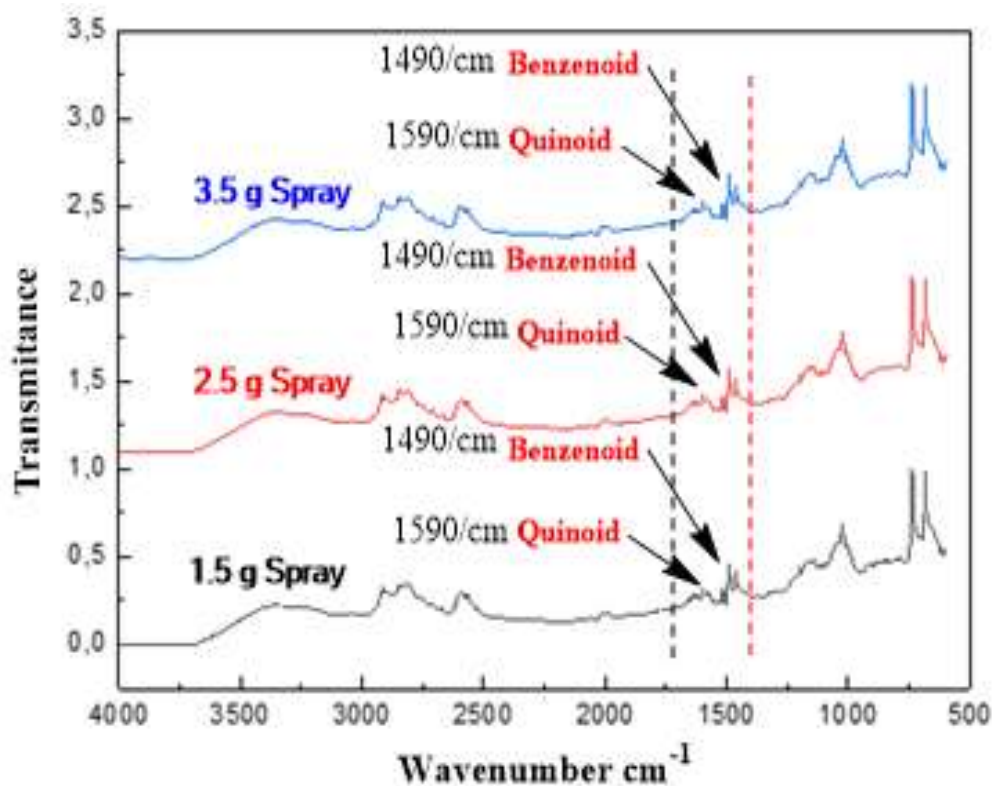


Figure 7:- FTIR Spectra of Glucomannan-ES-PANI Composites Film (Quinoid / Benzenoid) use Spray Method (Dopant HCl).

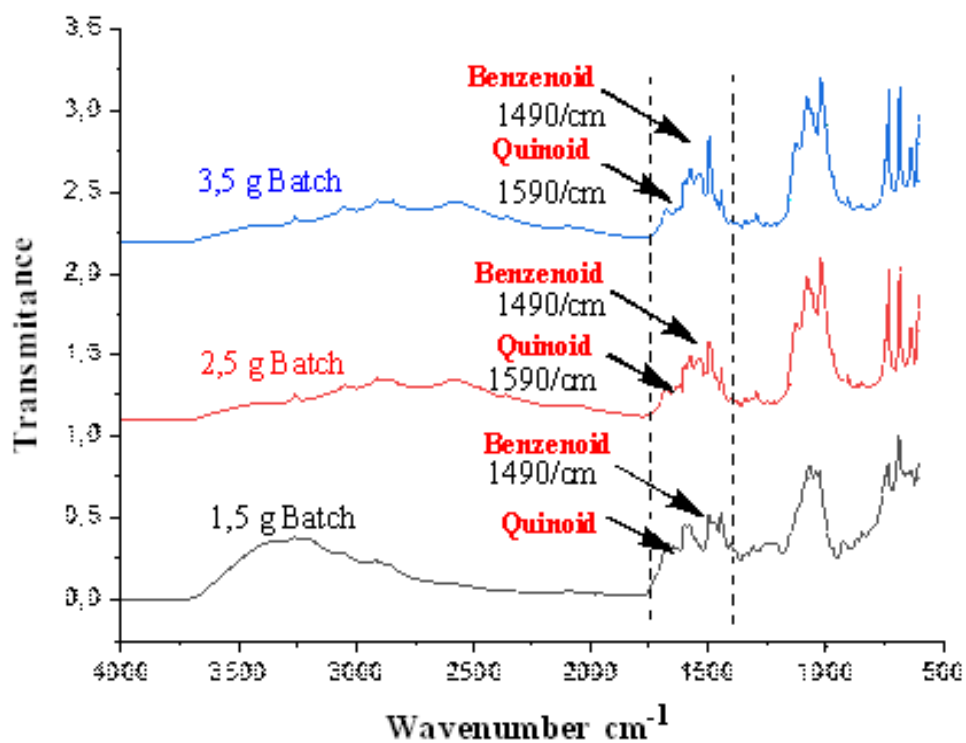


Figure 8:- FTIR Spectra of Glucomannan-ES-PANI Composites Film (Quinoid / Benzenoid) use Batch Method (Dopant HCOOH).

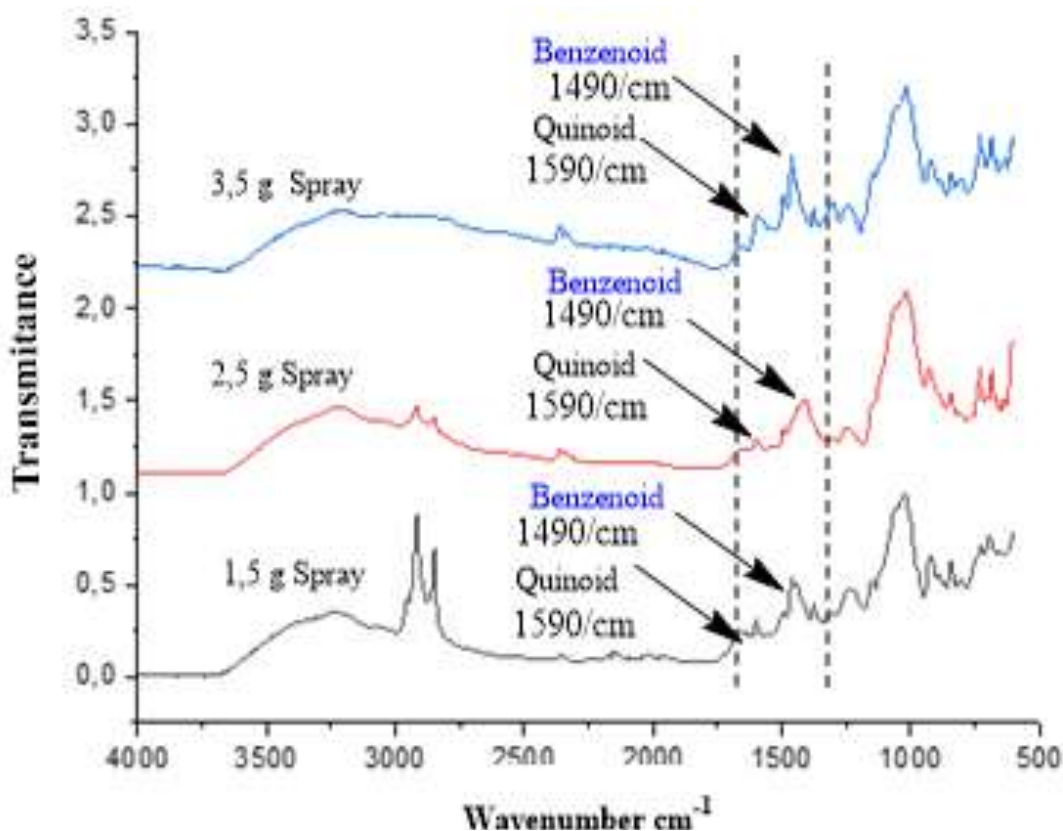


Figure 9:- FTIR Spectra of Glucomannan-ES-PANI Composites Film (Quinoid /Benzenoid) prepared by Spray Methods (Dopant HCOOH).

The results of the FTIR spectrum of ES-polyaniline have the following peaks: 3220.31 cm^{-1} ; 1590.6 cm^{-1} ; 1491.18 cm^{-1} ; 1297.28 cm^{-1} ; 1152.84 cm^{-1} ; 1019.53 cm^{-1} , and 875.39 cm^{-1} ; while the bacterial polyaniline-cellulosic composite with a concentration of 3.5M HCl resulted in the FTIR spectrum at Figures 5, 6, 7, 8, and 9 showing peaks at 3401.12 cm^{-1} ; 3220.39 cm^{-1} ; 1637.70 cm^{-1} ; 1580.92 cm^{-1} ; 1490.14 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. The results of the spectrum of polyaniline and polyaniline-glucomannan composites in the study can be seen in Figures 6, Figure 7, Figure 8, Figure 9, and Figure 10. The FTIR spectra of the ES-PANI/Glucomannan composite doped with HCl and HCOOH show two peaks of a quinoid, and a benzenoid. At the same oxidation state (from emeraldine base/EB change to emeraldine salt/ES), the quinoid and benzenoid rings are cross-linked. These two parts are the main constituents of polyaniline which play an important role in its electrical properties. However, the addition of HCl and HCOOH dopants will affect the composition of the quinoid and benzenoid ring in ES-PANI. According to the reaction mechanism of dopant with polyaniline, the imine site (-N=) in the quinoid ring allows protonation of strong acids to occur. The protonation of a strong acid at the imine site results in a change in the quinoid ring to benzenoid. The change from quinoid to benzenoid is accompanied by changes in the electrical properties of ES-polyaniline which are increasing. The greater the composition of benzenoid in the polymer chain, the greater the conductivity. However, not the overall reaction between the dopant and the amount of the imine site in the quinoid will form a benzenoid. This is influenced by the acid concentration, the strength of the acid initiation, and the ability of the acid to protonate the imine site. Depending on the comparisons square in quinoid/benzenoid approximately 0.8 (Busroni et al., 2022)

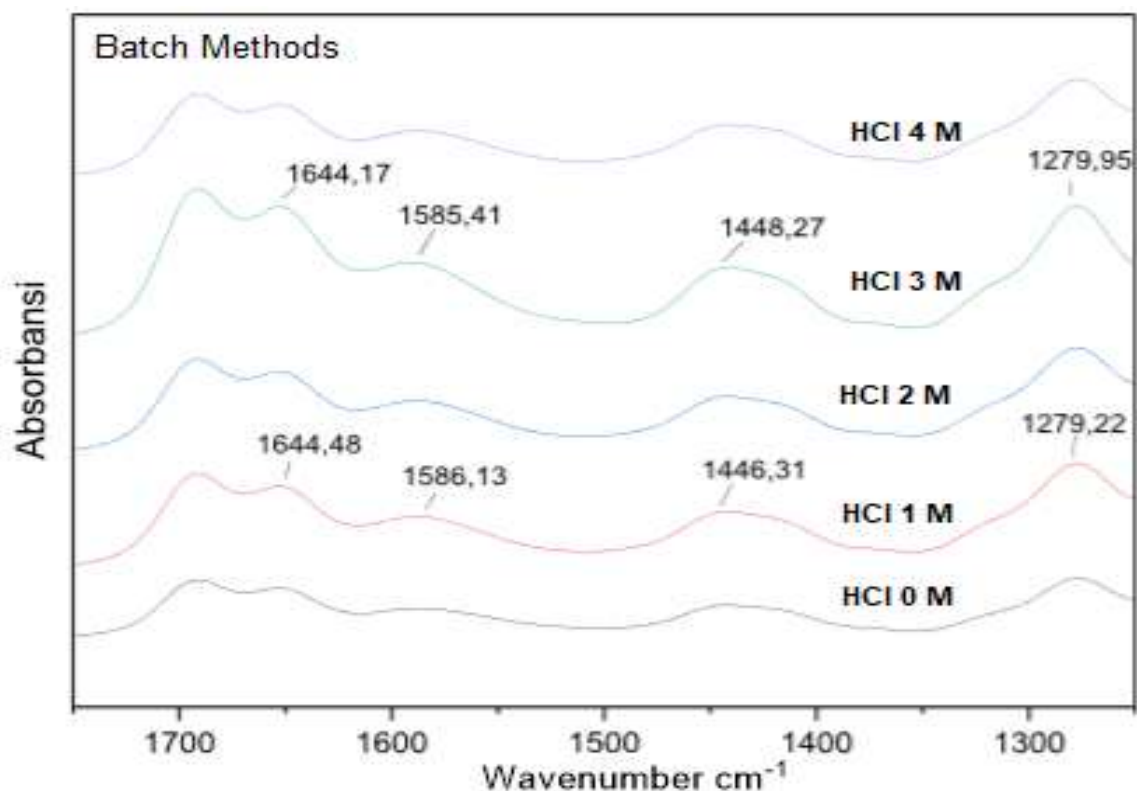


Figure 10:- FTIR Spectra of ES-PANI-BC Composites Film (Benzenoid and Quinoid) by Batch Methods use Dopant HCl.

Table 1:- Effect of Variation of Concentration of Dopant (M) onto Conductivity Value of ES-PANI-BC Composites.

Concentration of Dopant HCl (M)	C=C Benzenoid	-C=N Quinoid	$A_{\text{Quinoid}}/A_{\text{Benzenoid}}$	Value of Conductivity (S/cm)
0	0.65	0.60	0.92	2.19×10^{-9}
1	0.95	0.95	1.00	4.55×10^{-2}
2	1.00	1.00	1.00	5.61×10^{-2}
3	1.40	1.60	1.14	1.08×10^{-1}
4	0.85	0.95	1.12	8.37×10^{-2}

Based on (Yuningsih et al., 2017; Yuningsih et al., 2018; Busroni et al., 2022), Increasing the concentration of the dopant of HCl in the polymerization has been shown will increase the value of conductivity caused by the increase in the ratio of ($A_{\text{Quinoid}} / A_{\text{Benzenoid}}$) has been shown in Figure 10, by the filled HCl dopant to the emeraldine salt structure and conductivity increase is higher by dopant HCl is a strong acid, the increase in the conductivity value can be seen in Table 1. According to research conducted by (Yuningsih et al., 2017; Yuningsih, et al., 2018; Busroni, et al., 2022), when using acid doping with strong acid at a concentration of 4 M the conductivity decreased and a polyaniline hydrolysis reaction occurred because the system has a water absorption ability of 100% of the thin film, thus allowing a thermodynamic equilibrium to occur in ES-PANI-BC (Busroni, et al., 2022). The longer the immersion time of glucomannan in a solution using room temperature, the more water content is substituted by APS and dopants. In this study, bacterial cellulose was immersed in a mixture of APS and HCl for 0,5 hours to replace the presence of water content in glucomannan and HCl or HCOOH the higher the conductivity will. However, if the quinoid composition predominates in the ES-PANI structure chain to a certain extent, a lower conductivity is obtained. The phenomenon in Table 1, may be due to hydrolysis in the presence of strong acids such as HCl at 4 M the conductivity decreased as shown by the proposed Mechanism of hydrolysis of ES-PANI/Cellulose in Figure 11. below:

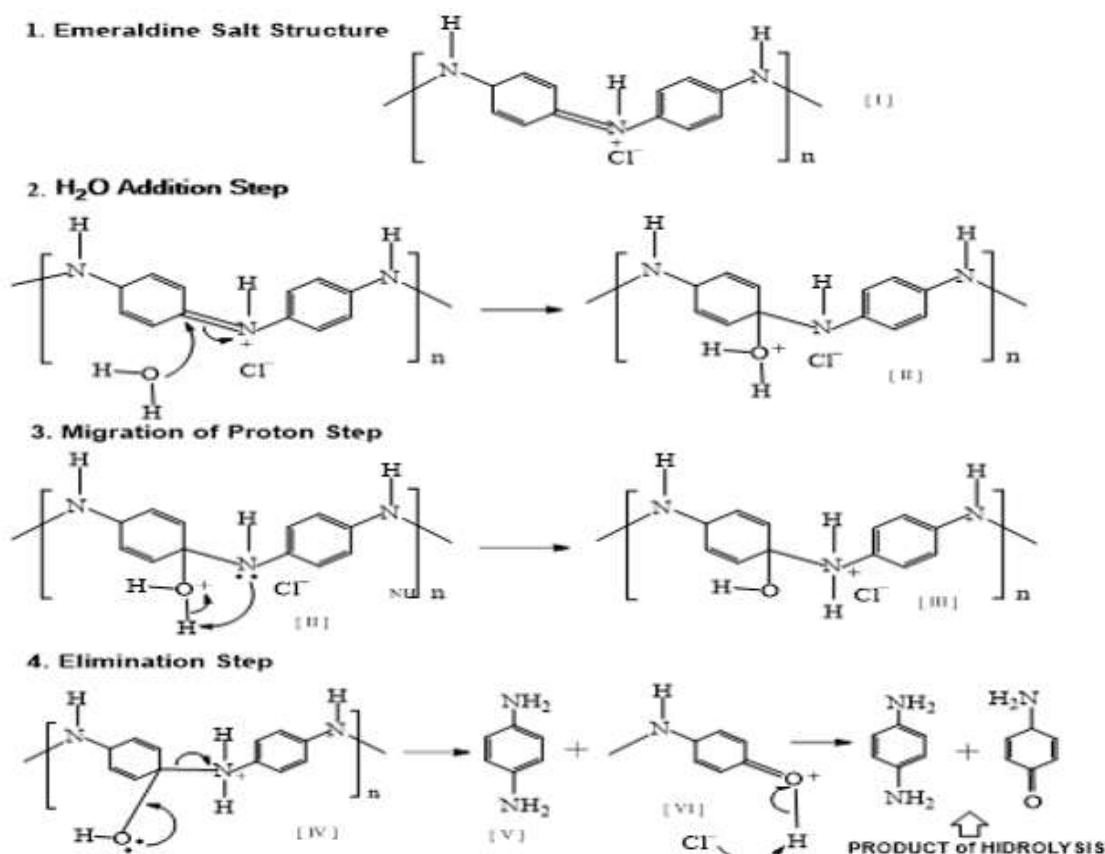


Figure 11:- Proposed Mechanism of Hydrolysis Emeraldine-Salt by Acid (Busroni et. al., 2022).

Table 2:- Effect of Mass of Glucomannan, Variation of Dopant, and Variation of Methods onto Conductivity Value of ES-Polyaniline-Glucomannan Composites.

Variation of Glucomannan	mass	Conductivity Value (S/cm)			
		Dopant HCl		Dopant HCOOH	
		Batch Method	Spray Method	Batch Method	Spray Method
1.50 g		2.36×10^{-3}	7.57×10^{-8}	3.01×10^{-4}	5.51×10^{-8}
2.50 g		2.87×10^{-3}	1.04×10^{-7}	4.36×10^{-4}	6.98×10^{-8}
3.50 g		3.68×10^{-3}	1.23×10^{-7}	5.87×10^{-4}	9.20×10^{-8}

Increasing the mass of glucomannan in the polymerization will increase the degree of conductivity caused by the increase in protons (H^+) by the filled HCl dopant to the emeraldine salt structure and conductivity increase is higher than HCOOH, because HCl is a strong acid and HCOOH is low acid, the increase in the conductivity value can be seen in Table 2. according to research conducted by (Yuningsih et al., 2017; Yuningsih, et al., 2018; Busroni, et al., 2022), when using acid doping with strong acid, the conductivity decreased and a ES-polyaniline hydrolysis reaction occurred because the system has a water absorption ability of 99% of the thin film, thus allowing a thermodynamic equilibrium to occur in ES-PANI-glucomannan (Busroni et al., 2022). The longer the immersion time of glucomannan in a solution using room temperature, the more water content is substituted by APS and dopants. In this study, Glucomannan was immersed in a mixture of APS and HCl, and HCOOH for 0,5 hours to replace the presence of water content in glucomannan and HCl or HCOOH the higher the conductivity will be. Table 2 has been shown, the highest conductivity value in this study in the batch system was at mass glucomannan 3,5 g using doping HCl of $3,68 \times 10^{-3}$ S/cm in Table 1 and mass glucomannan 3,5 g used doping HCOOH of $5,87 \times 10^{-4}$ S/cm because with the addition of acid concentration resulted in the emergence of free radical cations due to the oxidation polymerization reaction, and the highest conductivity value in this study in the spray system was at mass glucomannan 3,5 g using doping HCl of $1,23 \times 10^{-7}$ S/cm in Table 1 and mass glucomannan 3,5 g used doping HCOOH of $9,20 \times 10^{-8}$ S/cm because with the addition of acid concentration resulted in the emergence of free radical cations due to the oxidation polymerization reaction. This radical cation causes the double-bonded ES-

polyaniline to form an energy level between the valence bands. This causes the presence of free electrons which are charge carriers so the addition of the acid concentration will cause more free electrons to be bound to the ES-polyaniline and cause the conductivity to increase. The results of this characterization are the results of the oxidation polymerization process on aniline monomer which produces polyaniline doped with HCl as a proton source highest rather than HCOOH, resulting in a change in its structure from Emeraldine Base change to Emeraldine Salt which is highly conductive. Synthesis of the ES-polyaniline-glucomannan composite 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-PANI-glucomannan is a conductor. Table 2. shows that the resulting condition is greater if the addition of more glucomannan mass. This result is supported by the FTIR characterization in Figure 6, Figure 7, Figure 8, and Figure 9, and Figure 10, which shows, that in the sample with the addition of 3.5 g of glucomannan mass the ES-PANI-glucomannan graft 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. When applied as a humidity sensor, the ES-PANI glucomannan filler film functions as a binder for water molecules. The more charge carrier ions so the conductivity values of the material increases. In Figure 6, Figure 7, Figure 8, Figure 9, and Figure 10, it can be seen that qualitatively the amount of ($A_{\text{Quinoid}}/A_{\text{Benzenoid}}$) groups has high quality in the emeraldine salt form using HCl doping which is of higher quality than using HCOOH doping, and the amount of glucomannan mass and the quality of ($A_{\text{Quinoid}}/A_{\text{Benzenoid}}$) is highest in both HCl and HCOOH doping on a batch method is higher than spray system. This condition greatly affects the conductivity values of the ES-PANI-Glucomannan composite film. Due to the relative quality of the best ($A_{\text{Quinoid}}/A_{\text{Benzenoid}}$) using doped HCl rather than HCOOH

Conclusion:-

Synthesis of ES-PANI-Glucomannan composite film from glucomannan with dopant HCl produces higher conductivity rather than using dopant HCOOH batch method higher rather than spray methods. The optimum conductivity value of ES-PANI-Glucomannan dopant HCl is $3,68 \times 10^{-3}$ S/cm. and HCOOH is $5,87 \times 10^{-4}$ S/cm in the batch method and the optimum conductivity values of ES-PANI-Glucomannan dopant HCl is $1,23 \times 10^{-7}$ S/cm. and HCOOH is $9,20 \times 10^{-8}$ S/cm in spray method and The conductivity values of ES-PANI-BC dopant HCl 0 M; HCl 1 M; HCl 2 M; HCl 3 M is $2,193 \times 10^{-9}$; $4,551 \times 10^{-2}$; $5,605 \times 10^{-2}$; $1,077 \times 10^{-1}$ S/cm respectively.

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