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FOURIER TRANSFORM INFRA RED CHARACTERIZATION OF RESIPRENE – POLYPROPHYLENE BLEND WITH STEARIN AS COMPATIBILIZER

Nina Hartati[1*], Basuki Wirjosentono[2], Erika Dwi Oktaviani[1], Melantina Oktriyanti[1], Meyci Trisna[1], Metta Wijayanti[1]

[1]Chemical Engineering Major, Politeknik Negeri Sriwijaya, Indonesia

[2]Chemistry Department, Mathematic and Science Faculty, Universitas Sumatera Utara, Indonesia

nina.hartati@polsri.ac.id

The research on Fourier Transform Infra Red Characterization of Resiprene – Polyprophylene Blend with Stearin as Compatibilizer has been conducted. The Polypropylene-Resipren mixture was created by dissolving each in xylene solvent at 200 °C, followed by pressing at 170 °C in accordance with ASTM 368 M Type 4. Subsequently, its mechanical properties were characterized using a Tensile Strength Test to determine the optimum mixture. From the mechanical property characterization, the optimal mixture was found to be a combination of Polypropylene and Resipren with an 80:20 ratio (phr: phr). Next, the optimal mixture was supplemented with Stearin as a Compatibilizer with variations of 0.2; 0.4; 0.6; 0.8; and 1 phr, and its mechanical properties were re-characterized to obtain the optimal mixture, which was achieved with the addition of 0.2 phr Stearin. The FT-IR spectrum analysis of Resiprene reveals the existence of a double carbon-carbon bond, as evidenced by an absorption peak at approximately 1600 cm-1. Additionally, distinctive absorption bands for CH_3, CH_2, and –CH are observed at 2924 cm^{-1} and 2723 cm^{-1}, respectively. In the analysis of the Polypropylene-Resipren mixture, various absorption peaks are identified, including 748.38 cm^{-1}, 1373-1458 cm^{-1}, 1651 cm^{-1}, 1701 cm^{-1}, 2723 cm^{-1}, 2862 cm^{-1}, 2924-2954 cm^{-1}, and 3425 cm^{-1}. These findings confirm the presence of the double carbon-carbon bond in Resiprene and indicate the introduction of stearin, as demonstrated by absorption peaks at 3400 cm^{-1} (O-H group) and 1701 cm^{-1} (carbonyl group). Furthermore, the FT-IR spectrum of the Polypropylene-Resipren-Stearin mixture exhibits persistent Resiprene features, with sharper peaks for the O-H group at 3400 cm-1 and the carbonyl group at 1701 cm^{-1}.

Keywords : *Resiprene, Polyprophylene, Stearin, ASTM 368*

1. IINTRODUCTION

The Directorate General of Agro and Chemical Industry of the Ministry of Industry of the Republic of Indonesia has set forth in its vision and mission to enhance the production of rubber and rubber processed goods, aiming to position Indonesia as a leading producer of rubber goods and processed rubber before the year 2020. However, up until now, approximately 85% of Indonesia's total rubber exports consist of raw materials. In other words, only about 15% of the national rubber production is processed into finished goods by domestic industries. This proportion indicates the need for the Indonesian rubber industry to improve both quantity and quality, given the high dependency on imported rubber raw materials. Therefore, efforts, special attention, and innovation are necessary for the development of raw rubber materials into processed rubber goods. One approach to increasing the economic value of rubber is through modification, such as through cyclization (Eddyanto et al., 2013).

Natural rubber can be chemically modified through the cyclization process, resulting in cyclized natural rubber (Cyclised Natural Rubber/CNR). Cyclized natural rubber is obtained by treating natural rubber with strong acids (such as sulfuric acid, p-toluenesulfonic acid) or Friedel-Crafts catalysts (such as $FeCl_3$, $SnCl_4$, $TiCl_4$). Cyclized natural rubber has double bonds in its polymer chain and is a nonpolar polymer. Resiprene is CNR’s resine. Compatibilizers function by incorporating one chemical component to bond with a polymer and another chemical component to bond with a different polymer, forming covalent bonds between the two polymer phases. The utilization of compatibilizers minimizes the separation of the two polymer phases by improving the adhesion at their interface. Typically, compatibilizer materials consist of block copolymers or grafts with distinct segments that chemically match the different phases of the matrix.

Due to the low surface energy of recyclable cyclized natural rubber resin, resulting in low interfacial interaction and adhesive properties, a stearin-based compatibilizer is necessary. Researchers anticipate that stearin can enhance the physical and mechanical properties of the mixture due to the presence of stearic acid. This improves the homogeneity of the resin with polypropylene, expanding its potential applications. A study conducted by Nasution et al. (2014) investigated the grafting of methyl methacrylate onto cyclized natural rubber using dicumyl peroxide as an initiator. The resulting CNR-g-MMA product was identified through the appearance of absorption peaks at 1731 cm -1, characteristic of carbonyl groups from methyl methacrylate. Grafting methyl methacrylate onto cyclized natural rubber at 90 °C produced MMA-g-CNR, and an extended grafting reaction increased the degree of grafting.

Siregar (2015) explored the grafting of maleic anhydride onto cyclized natural rubber in an internal mixer, examining its physical properties and compatibility with polyamide. Grafting maleic anhydride onto cyclized natural rubber produced CNR-g-AM, with a higher concentration of maleic anhydride resulting in more grafted maleate groups on the CNR polymer chain. The grafting product exhibited minimal changes in physical properties except for an elevated glass transition temperature and improved compatibility with polyamide.

Mirzataheri (2000) examined the cyclization of natural rubber in a toluene solution at 100 °C, indicating variations in cyclization rate and the presence of 8% SnCl4 in 27.4% total unsaturation in cyclized natural rubber, as evidenced by the degree of unsaturation. The degree of cyclization and the formation of mono-, bi-, or poly-cyclic structures were calculated based on the reduction in the characteristic intensity of the unsaturation absorption band and the decrease in the amount of unsaturation. Sahly (2006) investigated the production of rubber adhesive on metal using a blend of cyclo rubber and natural rubber. The optimal formula for MS-30 adhesive displayed characteristics such as a Brookfield viscosity of 1750 cP, density of 1.021 g/cm3, shear strength of 3.54 kg/cm2, and tensile strength of 5.65 kg/cm2. The study concluded that the blend of cyclo rubber and natural rubber in the MS-30 adhesive is well-formulated as a raw material for rubber adhesives on metal, demonstrating superior adhesive strength compared to commercial adhesives. However, the Brookfield viscosity value remains relatively high compared to commercial adhesives.

Bahruddin et al. (2007) examined the morphology and properties of a mixture of natural rubber/polypropylene dynamically vulcanized in an internal mixer. The study found that increasing the ratio of natural rubber to polypropylene can lead to a uniform distribution of natural rubber particles with small sizes. The ratio of natural rubber to polypropylene and the composition of the cross-linking agent can enhance the mechanical properties of the thermoplastic elastomer.

2. METHOD

Tools and Material

The tools used in this study were three neck rounded flask, hotplate, condensor, glass beaker, scale glass, thermometer, a set of press printing tools. The materials used in this study were xylena, polypropylene, resiprene 35 from PTPN 3, and stearin.

Preparation of Polypropylene Mixture with Resiprene

The preparation of Polypropylene Mixture with Resipren begins by introducing 90 phr of polypropylene into a beaker glass and adding 450 ml of xylene. This mixture is heated on a hotplate at a temperature of 180°C. Subsequently, 10 phr of Resipren is placed into a three-necked flask, and 50 ml of xylene is added. A condenser is connected to the left opening of the three-necked flask, while the middle part is sealed with a rubber stopper, and the right part is equipped with a thermometer. The mixture is heated on a hotplate at 200°C and stirred using a magnetic stirrer. After both components dissolve completely, the polypropylene solution is poured into the Resipren solution and stirred again with a magnetic stirrer for approximately 15 minutes.

Next, the mixture is poured into a petri dish and dried at room temperature. The same procedure is repeated for polypropylene: Resipren mixtures with ratios of 100:0 (phr:phr), 80:20 (phr:phr), 70:30 (phr:phr), and 60:40 (phr:phr). The mixtures, finely ground using a pestle and mortar, are placed between 15x15 cm aluminum plates covered with aluminum foil to conform to ASTM 368 M Type 4. These plates are then inserted into a hydraulic press set at 175°C for 15 minutes. Subsequently, the plates are lifted and cooled.

Stearin Addition

The optimal Polypropylene Mixture with Resipren is supplemented with Stearin in variations of 0.2 phr, 0.4 phr, 0.6 phr, 0.8 phr, and 1 phr.

Fourier Transform Infra Red Characterization

Firstly, the film resulting from the mixture is clamped onto the sample holder and then placed in the direction of the infrared light in the instrument. The outcomes will be recorded on a scaled paper in the form of a curve depicting wave number against intensity.

3. RESULTS AND DISCUSSION

Functional group analysis of the mixture containing 80 phr Polypropylene: 20 phr Resipren

The results of the functional group analysis of the mixture containing 80 phr Polypropylene: 20 phr Resipren using infrared spectroscopy (IR) have been conducted. Infrared spectroscopy is a commonly used method to investigate the structure, bonds, and functional groups present in a compound based on the absorption of wavelengths. The analysis of this infrared spectrum aims to determine the changes in functional groups experienced by the Polypropylene-Resipren mixture, including the addition of Stearin. Table 1 shows the absorption bands in the IR spectrum of the 80 phr Polypropylene: 20 phr Resipren mixture.

Table 1. Infra Red Adorbance of 80 phr Polipropilena : 20 phr Resiprene Mixture

Wave Number (cm^{-1})	Functional Group
748,38	C-H long chain
1373,32-1458,18	C-H bending
1651,07	C=C
2723,49	CH
2862,36	CH_3
2924,09-2954,95	CH_2

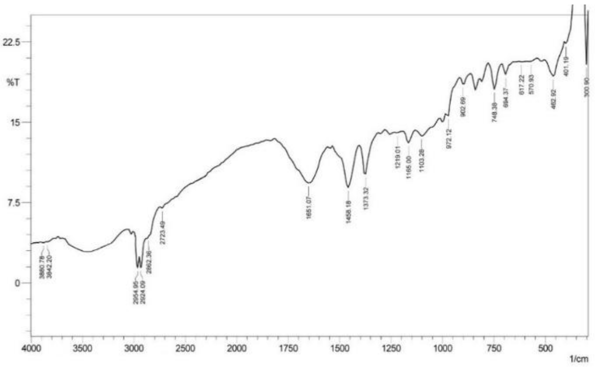

Figure 1 Infra Red Adorbance of 80 phr Polipropilena : 20 phr Resiprene

Based on the FT-IR spectrum as shown in Figure 1, it can be stated that there is a double carbon-carbon bond in the structure of Resiprene, confirmed by the absorption peak around 1600 cm-1, which aligns with the findings reported by Lee, D.F. et al., 1963. Additionally, absorption bands are observed around the wavenumbers of 2924 cm^{-1} for CH_3, 2924 cm^{-1} for CH_2, and 2723 cm^{-1} for –CH, in accordance with the report by Karo, 2007.

Functional Group Analysis for the Mixture of 80 phr Polypropylene: 20 phr Resipren with the Addition of 0.2 phr Stearin

Table 2 below shows the absorption bands in the FT-IR spectrum of the 80 phr Polypropylene: 20 phr Resipren mixture with the addition of 0.2 phr Stearin.

Wave Number (cm^{-1})	Functional Group
748,38	C-H long chain
1373,32-1458,18	C-H bending
1651,07	C=C
1701,95	C=O
2723,49	CH
2862,36	CH_3
2924,09-2954,95	CH_2
3425,58	O-H

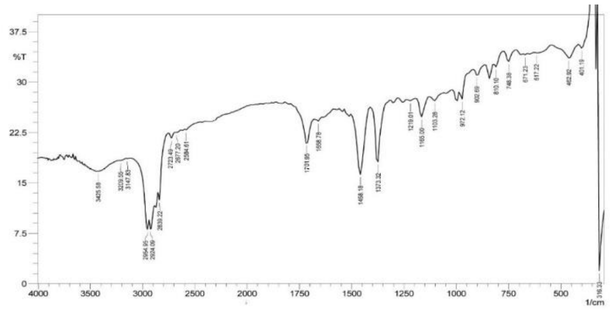

Figure 2. FTIR Spectrum of 80 phr Polipropilena : 20 phr Resipren mixture with 0,2 phr Stearin additio

The FT-IR spectroscopy analysis of the Polypropylene-Resipren mixture has a spectrum with absorption peaks in the wavenumber regions of 748.38 cm^{-1}, 1373-1458 cm^{-1}, 1651 cm^{-1}, 1701 cm^{-1}, 2723 cm^{-1}, 2862 cm^{-1}, 2924-2954 cm^{-1}, 3425 cm^{-1}, as depicted in Figure 2. Based on the FT-IR spectrum as shown in Figure 2, it can be concluded that there is a double carbon-carbon bond, confirmed by the appearance of an absorption peak around 1600 cm^{-1}, indicating the structure of resiprene, which aligns with the findings reported by Lee, D.F. et al., 1963. Additionally, absorption bands are observed around the wavenumbers of 2924 cm^{-1} for CH_3, 2924 cm^{-1} for CH_2, and 2723 cm^{-1} for –CH, consistent with the report by Karo, 2007. The emergence of an absorption peak around 3400 cm^{-1} indicates the presence of the O-H group from stearin, further reinforced by the absorption at 1701 cm^{-1}, indicating the carbonyl group from Stearin.

Functional Group Analysis Results for the Mixture of 80 phr Polypropylene: 20 phr Resipren with the Addition of 1 phr Stearin

The functional group analysis results for the mixture of 80 phr Polypropylene: 20 phr Resipren with the addition of 1 phr Stearin produced absorption bands as presented in Table 3.

Table 3. Absorption Bands in the FT-IR Spectrum of 80 phr Polypropylene: 20 phr Resipren with the Addition of 1 phr Stearin

Wave Number (cm^{-1})	Functional Group
748,38	C-H long chain
1373,32-1458,18	C-H bending
1651,07	C=C
1743,66	C=O
2723,49	CH
2862,36	CH_2
2924,09-2954,95	CH_3
3425,58	O-H

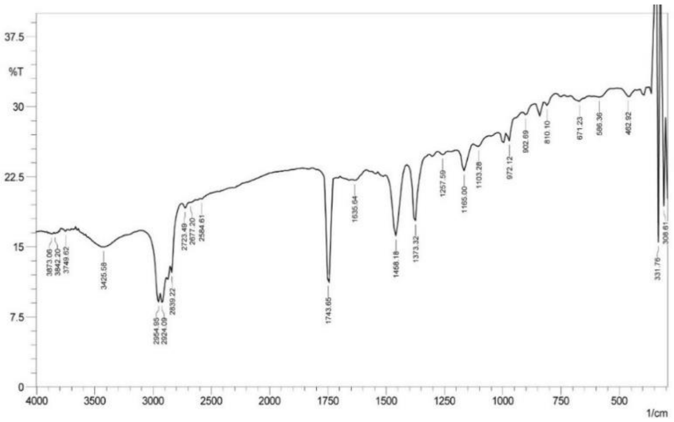

Figure 3. FTIR Spectrum of 80 phr Polipropilena : 20 phr Resipren mixture with 1 phr Stearin addition

Based on the FT-IR spectrum as shown in Figure 3, it can be stated that there is a double carbon-carbon bond, confirmed by the appearance of an absorption peak around 1600 cm^{-1}, indicating the structure of RESIPRENE, which aligns with the findings reported by Lee, D.F. et al., 1963. Additionally, absorption bands are observed around the wavenumbers of 2924 cm^{-1} for CH_3, 2924 cm^{-1} for CH_2, and 2723 cm^{-1} for –CH, consistent with the report by Karo, 2007. The emergence of an absorption peak around 3400 cm^{-1} indicates the presence of the O-H group from stearin, where the peak produced is sharper compared to the addition of only 0.2 phr Stearin. The interaction between Polypropylene and Resipren is reinforced by the absorption at 1701 cm^{-1}, indicating the carbonyl group from Stearin. Comparing the spectrum with the mixture of polypropylene, resipren, and stearin, there is a shift in the wavenumber for C=O, but no new functional groups are added, indicating only a physical interaction. Thus, it can be said that the addition of stearin has not fully formed a chemical bond.

4. CONCLUSION

The FT-IR spectrum analysis of Resiprene indicates the presence of a double carbon-carbon bond, supported by an absorption peak around 1600 cm^{-1}, Additionally, characteristic absorption bands for CH_3, CH_2, and –CH at 2924 cm^{-1}, 2924 cm^{-1}, and 2723 cm^{-1}, respectively. The analysis of the Polypropylene-Resipren mixture reveals various absorption peaks, including 748.38 cm^{-1}, 1373-1458 cm^{-1}, 1651 cm^{-1}, 1701 cm^{-1}, 2723 cm^{-1}, 2862 cm^{-1}, 2924-2954 cm^{-1}, and 3425 cm^{-1}, confirming the double carbon-carbon bond in Resiprene and the presence of stearin. The latter is indicated by an absorption peak at 3400 cm^{-1} (O-H group) and 1701 cm^{-1} (carbonyl group). Furthermore, the Polypropylene-Resipren-Stearin mixture's FT-IR spectrum demonstrates the continued presence of Resiprene features, with sharper peaks for the O-H group at 3400 cm^{-1} and the carbonyl group at 1701 cm^{-1}. A comparison with the mixture containing only 0.2 phr of Stearin suggests an enhanced interaction between Polypropylene and Resipren. However, the absence of new functional groups and only a shift in the wavenumber for C=O indicates that a complete chemical bond has not formed, implying a physical interaction between the components.

5. REFERENCES

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