Analysis of natural rubber compounds with filler Of Oil Palm Empty Bunches Powder and Carbon Black

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Abstract. The study aimed to determine the effect of the addition of carbon black (CB) and Palm Empty Bunch Powder (OPBEP) as a filler in natural rubber compounds on thermal and transmittance properties. The composting method is done at the Open mill. The process of mixing natural rubber with additives, curative agents, activators, accelerator materials and nano OPBEP and CB with variations (0; 2; 4; 6; 8)% wt. The results of the study were obtained. FTIR results from compound with CB and OPBEP fillers as a whole showed almost the same results. This is due to modifications so there is a possibility of overlapping with uptake on other bands. There is a peak of 2912 C–H asymmetric stretching vibrations of octadecylamine, respectively. Bending vibration of C–H in the = CH group of 3,4- or 1,2-units. The thermal properties of the DSC thermogram obtained an increase in the melting point and cross-linking with an increase in the content of the OPBA element compared with no filler as well as the CB / filler. In general, a compound with OPBEP filler and CB can be used as a technical product that has more mechanical properties.

Keywords: OPBEP, CB, natural rubber compounds, Thermal properties

1. Introduction

The natural rubber has unique characteristics such as high strength, flexibility and high elasticity [1], so it becomes one of the important materials that is widely used. However, natural rubber needs to be added with fillers and additives to improve natural properties that are not desired so that a product is obtained as expected. The mixture of rubber, fillers, and additives is known as a compound. The rubber compound is a mixture of raw rubber, chemicals, and the way it is carried out by grinding in an open mill machine at certain temperatures and formulations. [2] the formation of the rubber compound is the initial stage of the production of rubber finished goods.

Rubber technology began to emerge when a product of natural latex was discovered, known as natural rubber or NR, which has a molecular structure of cis-1,4-polyisoprene and is not resistant to ozone, oil, and high temperatures. If it has been vulcanized, it turns into a thermoset and cannot be processed again either by heating or dissolving. Indonesia is the second-largest NR producing country in the world after Thailand, with its main product type.

Oil Palm Empty Bunch Powder (OPBEP) is the residual combustion of OPBEP in palm oil processing. OPBEP has the potential to be developed into goods that have a high use value if mixed with natural rubber [3] compounds contained in OPBEP where obtained Fe2O3 0.2%, Na2O 0.34%, MnO 0.56%, MgO 0.78%, P2O5 1.95%, Al2O3 13.87%, CaO 18.20% and SiO2 58.60% [4–5] has been
done because OPBEP produces strong fibers it can be used as a filler in fibrous rubber products, including gloves, car seats, mattresses, and composite boards [6].

Carbon black (CB) is the type of filler most commonly used in making rubber compounds. Carbon black filler has a strengthening effect on the physical properties of volcanic acid especially those with small grain size [7, 8, 9].

The addition of carbon black will affect compound properties, the viscosity and strength of the compound will increase, but the use of carbon black has a weakness, ie the compound adhesion will be reduced. This makes carbon black not compact with other constituent materials at the time of mixing. Much research has been done on the mixture of OPBEP with natural rubber, among others [10, 11].

This study aims to determine the thermal properties and functional groups as well as the molecular bonds of alan rubber compound with OPBEP and CB fillers with variations in composition. (0; 2; 4; 6; 8)% by weight.

2. Experimental

2.1 Material

OPBEP Nano Particles were used from the results of the study [5] with a size of 68.63 nm. The main material used in this study is natural rubber (NR). (SIR-20 with Mooney Viscosity 70 at 100° C), carbon black (N330). Additives used include Sulfur 3, Mercaptodibenzo-thiazoledisulfide (MBTS), Zinc Oxide, Stearic Acid, Tetro Metil Thiuram Disulfide (TMTD), sulfur and dibenzothiazyl disulfide, (Iso Propylamino Diphenylamine( IPPD ).

2.2 Preparation of Rubber Compounds with OPBEP and Carbon Black nanoparticle Fillers

The chemicals of each compound formula are weighed according to the formulation that has been determined as in Table 1. The amount of each ingredient in the compound formula in Phr (weight per hundred rubber) taking into account the conversion factor. Materials such as in Table 1 are mixed using an open mill until all ingredients are well mixed. Each mixed variation of SIR 20 natural rubber is put into the open mill machine, then ground until the rubber is completely solid. While the rubber milling process is running, the ingredients are inserted one by one into the open mill machine for each variation of the OPBEP and CB filler mixture. The compound mixing process by cutting from the edge of the mill, is rolled and then ground again until homogeneous. Compounds that have been made in the form of sheets are removed from the mill and put into plastic bags and labeled according to the sample code, then left for 24 hours.
Table 1. Composition of Compound Mixtures of Materials with OPBEP and CB Filler in phr.

| Materials           | Formula Compound (Per Hundred Ratio) | OPBEP | Carbon Black |
|---------------------|--------------------------------------|-------|--------------|
|                     |                                      | $S_0$ | $S_1$ | $S_2$ | $S_3$ | $S_4$ | $S_{cb1}$ | $S_{cb2}$ | $S_{cb3}$ | $S_{cb4}$ |
| NR SIR 20           |                                      | 100   | 98    | 96    | 94    | 92    | 98     | 96     | 94     | 92     |
| Wax                 |                                      | 1.5   | 1.5   | 1.5   | 1.5   | 1.5   | 1.5    | 1.5    | 1.5    | 1.5    |
| Filler (OPBEP and CB) |                                    | 0     | 2     | 4     | 6     | 8     | 2      | 4      | 6      | 8      |
| ZnO                 |                                      | 5     | 5     | 5     | 5     | 5     | 5      | 5      | 5      | 5      |
| SA                  |                                      | 2     | 2     | 2     | 2     | 2     | 2      | 2      | 2      | 2      |
| Sulfur              |                                      | 3     | 3     | 3     | 3     | 3     | 3      | 3      | 3      | 3      |
| IPPD                |                                      | 2     | 2     | 2     | 2     | 2     | 2      | 2      | 2      | 2      |
| TMTD                |                                      | 1.5   | 1.5   | 1.5   | 1.5   | 1.5   | 1.5    | 1.5    | 1.5    | 1.5    |
| MBTS                |                                      | 2.5   | 2.5   | 2.5   | 2.5   | 2.5   | 2.5    | 2.5    | 2.5    | 2.5    |

3. Results and discussion

3.1 Analysis of Fourier Transform Infra Red (FTIR)

The results of the FTIR characterization of natural rubber compound nanocomposite samples with OPBEP filler are shown in Figure 1 of the spectra to indicate the existence of several vibrational bonds [12]. C-H bonds (with hydrogen attached to carbon) absorb in the area between 2853 - 2962 cm$^{-1}$. Peak 1475-1300 also shows C-H bending. Peak 1000-650 shows C = C, Ar-H bending. FTIR spectra between natural rubber compound nanocomposites with the addition of OPBEP fillers and without fillers showed no significant difference, this is likely because the OPBEP fillers are spread evenly on the rubber compound and also possibly due to the number of fillers that are not large enough to affect the bond in natural rubber.

![FTIR Spectra](image_url)

Figure 1. FTIR Spectra of natural rubber compound with OPBEP filler combined (0-8)% by weight
The results of the FTIR characterization in Figure 2 on nanocomposite samples of natural rubber compound with Carbon Black (CB) fillers indicate the existence of several vibrational bonds. C-H bonds (with hydrogen attached to carbon) absorb in the area between 2853 - 2962 cm\(^{-1}\). Peak 1475-1300 also shows C-H bending. Peak 1000-650 shows C = C-H, Ar-H bending. FTIR spectra between natural rubber compound nanocomposites with the addition of CB fillers and without fillers showed no significant difference.

3.2 Analysis of Differential Scanning Calorimetric (DSC)

DSC measurements were carried out on a sh differential scanning calorimetric model DSC-60 plus series. The weight of the samples was 10 – 15 mg. The heat of vulcanisation was determined by integrating the area under the exothermal calorimetric signal. DSC testing uses a temperature range of 25°C to 600°C, where the heating rate setting is set at 5 °C / min. DSC test results are thermogram curves that can be used to determine melting temperature and temperature. DSC analysis is generally used to study phase transitions, such as melting, glass transition temperature (Tg), or exothermic decomposition, and to analyze the stability of oxidation and heat capacity of a material. Glass transition temperature (Tg) is one of the important physical properties of polymers that causes the polymer to have resistance to heat or varying temperatures. Where when the outside temperature approaches the glass transition temperature, a polymer undergoes a change from a hard rigid state to a soft one like rubber.
Table 2. Melting point natural rubber compound with OPBEP filler

| Sample (wt %) | Onset (C) | Tm Peak (C) | Endset (C) | Heat Area (mJ) | Heat Delta (J/g) |
|--------------|-----------|-------------|------------|----------------|------------------|
| 0            | 370.44    | 374.44      | 384.14     | 65.29          | 8.37             |
| 2            | 337.62    | 360.66      | 383.49     | 493.58         | 63.28            |
| 4            | 335.18    | 359.76      | 384.32     | 442.08         | 54.58            |
| 6            | 333.35    | 361.25      | 376.37     | 449.40         | 59.52            |
| 8            | 342.21    | 364.59      | 373.27     | 373.27         | 45.52            |

From Figure 3 we can see the melting point from the onset temperature of 333°C to the endset temperature 384°C the change in peak temperature is not significant from the OPBEP filler composition 2 to 8% by weight from temperature 360.66°C to 364.5°C, this can be seen in Table 2, but the change in the Heat area decreases with increasing OPBEP content. Compared to the non-filler compound, the largest Delta (J/g) at 2% composition.

Figure 4 shows the melting point from the onset temperature of 338°C to the endset temperature 384°C changes in peak temperature are not significant from the composition of CB fillers 2 to 8% by weight from temperatures 366.25°C to temperatures 370.78°C, this can be seen in Table 3 but the change in Heat occurs with increasing CB content. from 403.38 to 360.68 mJ. When compared to the OPBEP heat filler, it is greater than the natural rubber compound with CB filler. For CB Heat delta (J/g) is greatest in composition 4% by weight.
Figure 4. DSC thermogram natural rubber compound with CB filler combined (0 -8)% by weight

Table 3. Melting point natural rubber compound with CB filler

| Sample (wt %) | Onset (C) | Tm Peak (C) | Endset (C) | Heat Area (mJ) | Heat Delta (J/g) |
|--------------|-----------|-------------|------------|----------------|-----------------|
| 0            | 370.44    | 374.44      | 384.14     | 65.29          | 8.37            |
| 2            | 338.82    | 366.25      | 384.03     | 403.28         | 51.70           |
| 4            | 342.15    | 372.05      | 381.34     | 482.11         | 63.44           |
| 6            | 356.81    | 367.67      | 381.70     | 372.05         | 45.93           |
| 8            | 340.97    | 370.78      | 380.48     | 360.68         | 48.09           |

The results from this DSC analysis can be used to determine the enthalpy by measuring the differential heat flow required to keep the sample material and the inert reference at the same temperature. One important information that can be obtained in semi-crystalline polymers is the material's crystallinity content. Both mechanical, physical and chemical properties, this depends on the composition of the mixture and crystallization conditions such as temperature, pressure, orientation, molecular weight, and thinner [13]. Melting temperature is an important parameter for thermoplastics because it represents the minimum temperature required for polymer processing. The melting behavior is highly dependent on the chemical structure of the material along with the size and regularity of crystallization found in the crystalline phase [14].

4. Conclusion

FTIR results from the compound with CB and OPBEP fillers as a whole showed almost the same results, FTIR spectra between natural rubber compound nanocomposites with the addition of OPBEP fillers and without fillers showed no significant difference. This is due to modifications so there is a possibility of overlapping with the uptake on other bands. The thermal properties of the DSC
thermogram obtained an increase in the melting point and cross-linking with an increase in the content of the OPBA element compared with no filler as well as the CB / filler

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