Synthesis of palm oil-base zinc stearate and its application on manufacture of rubber component

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Abstract. Palm oil-based zinc stearate synthesis process has been developed. Palm oil-based stearic acid and zinc oxide were used as material. The synthesis was performed using an oil-jacketed reactor with a capacity of 50 kg per batch. The reaction was occurring at 95°C with atmospheric pressure and it took place about 10 minutes. The product characteristics was powder with a white appearance, 0.0 - 0.3 % fatty acid content, 0.2 - 1% moisture content, 118 – 125°C melting point and 0.5 - 1.1 g/cm³ specific gravity. The product of zinc stearate was also examined on an application of rubber component production process. Effects of using the zinc stearate to characteristics of the rubber component were tested. The characteristics included elongation, hardness and tensile strength of the rubber component. Application of 5 phr of zinc stearate on rubber component formulation showed that the rubber component processed using the zinc stearate has basic characteristic that meet the standard of rubber component of American Chemical Society (ACS) 1.

1. Introduction
Palm oil is the main vegetable oil produced in Indonesia. A huge amount of the palm oil production needs to be utilized as raw material of various products. One of the prospective products is zinc stearate. Zinc stearate has been applied in some process manufacture such as pigment and resin [1] as well as cosmetic [2] industry. Application of zinc stearate in rubber processing has been reported has some effect on natural rubber properties [3,4].

In the process production of natural rubber and synthetic rubber, using zinc oxide and stearic acid in the rubber component manufacture process is widely applied [5-8]. However, excessive use of zinc oxide in rubber compounding has been reported to disrupt the environmental balance [9-12]. Using a zinc stearate as an activator and accelerator in rubber processing could reduce excessive zinc oxide in rubber compounds [13]. This research proposed a palm oil-based zinc stearate synthesis method according to the previous reported work, with some modification and its application on rubber component manufacture [4].

2. Materials and method
2.1. Zinc stearate synthesis
In this work, the synthesis of zinc stearate was performed by adopting a fusion method [4]. The materials for zinc stearate synthesis were stearic acid (stearic acid content 69%) and zinc oxide (purity of 53%), which were obtained from PT Sumi Asih and Global Chemical Co., Ltd, respectively used as received. In this synthesis, 47.6% and 52.4% of stearic acid and zinc oxide were loaded in a 50 kg
capacity reactor equipped with an oil jacket and stirrer. The reaction conditions were at temperature 95°C and atmospheric pressure. The characteristics of the zinc stearate produced in the processes included its appearance, free fatty acid, moisture content, melting point and specific gravity were examined.

2.2. Zinc stearate application on the rubber compound
The zinc stearate developed in this work was examined on the application of rubber compound manufacture. The examinations were performed at the Indonesian Rubber Research Institute. The test was based on the manufacture of rubber compound according to the composition of the American Chemical Society (ACS) 1. Originally the composition of the formula of compound manufacture used zinc oxide and stearic acid as activator and accelerator on the rubber compound manufacture. In this examination, the zinc oxide and Stearic Acid were replaced with zinc stearate of BPPT produced in this work with a composition of 5 phr (per hundred rubber). The characteristics of the manufactured rubber compound, including hardness, tensile strength, elongation at break, tear strength, and specific gravity, were examined with the method of test as presented in table 2.

3. Result and discussion
3.1. Synthesis of zinc stearate
The synthesis of the zinc stearate was performed in an oil-jacketed reactor. First, stearic acid was loaded and melted in the reactor at 70°C and then zinc oxide was loaded to the reactor. The reactor temperature was adjusted to the reaction temperature (95°C) to allow the reaction occurred. The reaction process was observed visually. When the temperature of the material (zinc oxide and zinc stearate) reached about 95°C reaction was occurring and it was indicated by the formation of abundant water vapor in the reactor. The water vapor was a byproduct of the synthesis as shown in the reaction equation (1). The reaction was simple and it could be considered as zero-waste process. It was due to the byproduct of the reaction, which was solely water that has no bad impact on the environment. Moreover, it was observed that the process has been giving high yield with 98% (w/w) of the reactant converted to the product of zinc stearate. In addition, the completeness of the reaction was also indicated by the solidification of the raw material as a sign of zinc stearate formation, which had a melting point of about 120°C (figure 2a).

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2C_{17}H_{35}COOH + \text{ZnO} \rightarrow (C_{17}H_{35}COO)_2\text{Zn} + \text{H}_2\text{O} \quad (1)
\]

In the synthesis of zinc stearate, temperature change during the process is essential to be well controlled. The formation of zinc stearate can be observed by the temperature profile during the synthesis process. The reaction of zinc stearate synthesis occurred when material temperature increased dramatically during the process. In this situation, the increasing temperature of the material exceeded the temperature of the oil jacket as a heating medium caused by the heat resulting from the exothermic reaction.
Figure 1. A typical temperature profile of the reaction of zinc stearate synthesis.

A typical temperature profile of the reaction of zinc stearate synthesis can be observed in figure 1. In addition, completeness of the reaction was also indicated by the solidification of the raw material as a sign of formation of zinc stearate which has a melting point of about 120°C (figure 2a). To allow the reaction to complete and the excess of zinc oxide completely mix with the zinc stearate produced, the material was stirred continuously for 60 minutes at 130°C in which zinc stearate completely melted.

The liquid form of zinc stearate caused the reactor's unloading process became possible to perform easily (figure 2b). Subsequently, the zinc stearate was exposed to an aeration room for cooling and solidified. The solid zinc stearate then was ground into powder form. The consistency of the process and the controlled quality of the zinc stearic produced in this synthesis were examined by characterization of the zinc stearate. The characteristics of the zinc stearate produced are presented in the table 1.

Figure 2. a) Zinc stearate formation indicated by solidification of the material at the temperature of 95 - 120°C. b) Liquid zinc stearate was unloaded in liquid condition at the temperature of 130°C.
Table 1. Characteristics of zinc stearate which is produced in the synthesis.

| Parameter                  | Characteristic       |
|----------------------------|----------------------|
| Appearance                 | Homogenous white color, powder |
| Free Fatty Acid content (%)| 0 – 3                |
| Moisture content (%)       | 0.2 - 1              |
| Melting point (°C)         | 118 - 125            |
| Specific Gravity (g/cm³)   | 0.5 – 1.1            |

3.2. Zinc stearate application on the rubber compound
Zinc stearate resulted from this synthesis was applied in a rubber manufacturing process. The zinc stearate was applied to substitute zinc oxide and stearic acid, which was used as activator and accelerator in the process of rubber compound vulcanization. As a control, the vulcanization of the rubber compound was also performed using zinc oxide and stearic acid as activator and accelerator. The rubber compound vulcanization was performed by applying ACS 1 method. The examination of the application of zinc stearate and the control experiment, which used zinc oxide and stearic acid to rubber vulcanization, had applied activator/accelerator of 5 phr. According to the results of this test, as presented in table 2, all the basic characteristics of rubber products have met the ACS 1 rubber compound standard. Moreover, replacing zinc oxide and stearic acid with zinc stearate improved the quality of the resulted rubber compound. The improvement of the quality might be observed in table 2. It could be noticed that the application of the zinc stearate produced in this work increased the value of hardness of 9.0 - 12.1%, tensile strength of 30.4 - 66.4%, and elongation at break of 1.3 - 4.0%.

Table 2. Characteristics of the rubber compound.

| Parameter                  | Parameter value         | Test method          |
|----------------------------|-------------------------|----------------------|
|                            | Control (ACS 1)a)       | Using zinc stearate  |
|                            |                         | (5 phr*)b)           |
| Hardness, Shore A          | 33                      | 36 – 37              | D.2240-05(ra2010) |
| Tensile Strength, Mpa      | 12.5                    | 16.3 – 20.8          | D.412-06ae2       |
| Elongation at break, %     | 750                     | 760 – 780            | D.412-06ae2       |
| Tear Strength, kN/m        | 22.22                   | 22.2 – 22.6          | D.624-00(ra2002)  |
| Specific Gravity, g/cm³    | 0.983                   | 0.942 – 0.958        | D.297-93(ra2002)  |

phr(*) = Per Hundred Rubber
a) accelerator/activator was zinc oxide and stearic acid as control process
b) accelerator/activator was zinc stearate produced in this work

4. Conclusion
A Synthesis method of zinc stearate based on palm oil was developed. zinc stearate produced by the method was applied as accelerator on a rubber manufacturing process. The examination of the rubber compound indicated that replacement of zinc oxide and stearic acid with zinc stearate in a vulcanization of rubber compound could improve the rubber compound characteristic.
5. Reference

[1] Gursel A, Akca E and Sen N 2018 *Period. Eng. Nat. Sci.* 6 p 154
[2] Matthew C, Emmanuel O and Lawrence E 2019 *Glob. Sci. J.* 7 p 296
[3] Cunningham J 1992. Color cosmetics. *In: WILLIAMS D.F. & W.H, S. (eds.) Chemistry and Technology of the Cosmetics and Toiletries Industry* (Dordrecht: Springer)
[4] Basu D, Das A, Stöckelhuber KW, Wagenknecht U & Heinrich G 2014 *Prog. Polym. Sci.* 39 p 594
[5] Ismail H, Kamal SK and Mark SE 2001 *J. Elastomers Plast.* 33 p 100
[6] Ma H, Williams PL and Diamond SA 2013 *Pollut.* 172 p 76
[7] Lower, E. S. 1982 *Pigment Resin Technol.* 11 p 9
[8] Maciejewska M, Sowinska A and Kucharsk, J 2019 *Polymers (Basel)* 11(10)
[9] Gönen M, Balköse D, İnal F and Ülkü S 2005 *Ind. Eng. Chem. Res.*, 44 p 1627
[10] Fathurrohman MI, Soegijono B and Budianto E 2013 *J. Mater. Sci. Eng. B.* 3(9) p 575
[11] N. Hayeemasae N, W. G. I. U. Rathnayake WGIU and Ismail H 2018 *Rubber, Plast. Recycl. Technol.* 34 p 1
[12] Nasruddin & Bondan AT 2019 *J. Phys. Conf. Ser.* 1282(1)
[13] Helalya FM, El Sabbagh SH, El Kinawy OS and Sawya, SME 2011 *Materials & Desig.* 32 2835

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