

EFFECT OF ALKALINE TREATMENT ON THE PROPERTIES OF OIL PALM EMPTY FRUIT BUNCH FIBER-REINFORCED POLYPROPYLENE COMPOSITE

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ABSTRACT

Oil palm empty fruit bunch (OPEFB) is one of the waste products of oil palm plantations and has not been optimally used in Riau Province, Sumatera, Indonesia. OPEFB is reduced by incineration, which causes pollution problems. However, the combustion of OPEFB generates ash, which is rich in potassium. Moreover, OPEFB fiber has good strength, low cost, low density, and biodegradability, and it can be used as composite reinforcement. However, the natural fibers in composites have poor compatibility with the matrix and relatively high moisture absorption. Hydrolysis of OPEFB ash creates a base solution that can be utilized in an alkaline treatment process to increase the mechanical properties of natural composites. The aim of this study was to investigate the effect of various extracts of OPEFB ash on the tensile strength, flexural strength, and water absorption of an OPEFB fiber-polypropylene composite. The experimental design used was the Response Surface Method-Central Composite Design (RSM-CCD). The results showed that the tensile strength increased with an increase of fiber length and concentration of the OPEFB ash extract solution, but tensile strength decreased with a longer soaking time. Flexural strength increased with an increase in fiber length but decreased with an increase in the concentration of the OPEFB ash extract solution and longer soaking time. Water absorption increased with lower and higher concentrations of OPEFB ash extract solution and fiber length and with shorter and longer soaking times. The highest tensile strength (20.100 MPa) was achieved at 5%wt alkaline concentration, 36 h soaking time, and 3 cm fiber length. The highest flexural strength (30.216 MPa) was achieved at 5%wt alkaline concentration, 12 h soaking time, and 3 cm fiber length. The lowest water absorption (0.324%) was achieved at 10% wt alkaline concentration, 24 h soaking time, and 2 cm fiber length.

Keywords: Alkaline treatment; Flexural strength; Oil palm empty fruit bunch; Tensile strength; Water absorption

1. INTRODUCTION

The use of natural fibers as reinforcement of synthetic fiber composites has increased rapidly and widely in the construction material and automotive component industries (Ayrilmis et al., 2011; Ton-That & Denault, 2007). Currently, construction materials that are made of synthetic fiber composite materials are used for car dashboards and other interior parts. However, the use of synthetic fiber as reinforcement for these composites has become less attractive over the past few years because of its non-degradable properties. The use of natural fibers for the

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reinforcement of composites has become a popular research topic due to natural fibers' availability and sustainability. Natural fiber has several advantageous properties such as good strength, low density, biodegradability and low cost of production (Ayrilmis et al., 2011; Pracella et al., 2010). Moreover, natural fiber-reinforced composites have a lower density than synthetic fiber-reinforced composites, which ensures the production of lighter composites (Akil et al., 2011; Leao et al., 2010).

Similar to other natural fibers, oil palm empty fruit bunch (OPEFB) fibers are lignocellulosic fibers where the cellulose and hemicellulose are reinforced in a lignin matrix. OPEFB has a cellulose content of about 44% (Anggraini & Roliadi, 2011). For this reason, OPEFB is a potential source of natural fiber for reinforcing composites. OPEFB fibers have been studied as potential manufacturing composite materials using different synthetic polymers such as polypropylene copolymer (Razak & Kalam, 2012), homopolymer polypropylene (Khalid et al., 2008), poly(vinyl chloride) (Abu Bakar et al., 2005), epoxy (Yusoff et al., 2010). OPEFB fibers have also been studied as a component incorporated with glass fiber in polyester (Karina et al., 2008).

Despite several advantages, the main drawbacks of using natural fibers, including OPEFB, in composites are the poor compatibility, because the poor adhesion between fiber and matrix, and the relatively high moisture absorption. Therefore, chemical treatments have been considered for modifying the fiber surface properties. Researchers have reported several chemical treatments for use in natural-fiber reinforced composites including alkali, silane, acetylation, benzylation, acrylation, maleated coupling agents, isocyanates, permanganate and other chemical modifications of natural fibers. Alkaline treatment, or mercerization, is one of the most commonly used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets (Li et al., 2007). In studies using the alkaline treatment; OPEFB fibers were immersed in 5% solution of sodium hydroxide (NaOH) for a given period of time (Sreekala et al., 2000; Sreekala, 2002; Sreekala, 2003); dipped in 5% sodium hydroxide solution for about 48 h (Agrawal et al., 2000); soaked in 2% NaOH solution at 100°C for 30 min (Karina et al., 2008); soaked in 17.5% NaOH at 20°C for about 2 h (Khalid et al., 2008); immersed in various NaOH concentrations, exposure times and treatment temperatures respectively in the ranges of 30–90 min and 30–100°C (Moshiul et al., 2012); and immersed in a solution of 3% hydroxy ethyl acrylate (HEA) and 1% dicumyl peroxide in methanol for 5 min (Jawaid et al., 2014). In our previous study, alkaline treatment using an extract of OPEFB ash has been carried out on oil palm fronds (OPF). OPF soaked in 3:1–5:1 water to ash dissolving ratio at temperature 30–40°C for 4–12 hours soaking time. The result showed that alkaline treatment using extract of OPEFB ash significantly affected the mechanical and physical properties of the OFB fibers reinforced composite (Fatra et al., 2015).

Riau Province is the largest crude palm oil (CPO) production area in Indonesia, yielding 6,646,997 tons of CPO, or 23.93% of total national production, in 2013 (Directorate General of Estate Crops, 2014). The oil palm is a major source of edible oil, which is extracted from the palm fruit; however, the extraction process produces a large amount of solid waste that causes environmental problems, such as a pile of trash and smell of. Oil palm empty fruit bunch (OPEFB) is the second largest amount of this solid waste (about 1.1 tons for each ton of the CPO) generated from the palm oil industry after the oil palm frond. Moreover, OPEFB is commonly used as mulch for oil crops, and it is partially reduced by incineration, which has also caused pollution problems.

The OPEFB ash generated by incineration is often used as a substitute for potash fertilizer. OPEFB ash contains potassium in the form of potassium carbonate (43.15%wt), potassium hydroxide (15.91%wt), sodium carbonate (0.36%wt) and sodium hydroxide (0.13%wt), that

may increase the alkalinity of an OPEFB ash extract solution (Taiwo & Osinowo, 2001). Potassium carbonate is very soluble in water, and the dissociation reaction will produce potassium ions (K^+) and carbonate ions (CO_3^{2-}). Hydrogen ions (H^+) from water will react with carbonate ions which will cause the number of hydroxide ions (OH^-) to be greater than H^+ in the solution.

In this study, the extraction solution of OPEFB ash was used as an alkaline treatment of OPEFB instead of aqueous sodium hydroxide (NaOH). The purpose of this study was to investigate the potential of OPEFB ash as an alkaline treatment to increase tensile and flexural strength and to reduce water absorption of OPEFB fiber-polypropylene composites.

2. MATERIALS AND METHODOLOGY

2.1. Materials and Apparatus

OPEFB and OPEFB ash were obtained from the National Plantation V (PTPN V) palm oil mill in Sei Galuh, Riau Province. OPEFB was cleaned to attain the OPEFB fiber from the fruit stem. The fiber was extracted by a 96 h water-retting process and then sun-dried for three days. The fiber was cut at lengths of 1, 2, and 3 cm and then stored in a plastic container in order to avoid fungi. The OPEFB ash was sifted using 60 and 80 mesh sieves to produce an extracted alkaline solution.

Polypropylene copolymer was obtained from The Polyolefin Company (Singapore) Pte. Ltd. The universal testing machine at the Laboratory of Polymer Testing, Indonesian Institute of Sciences (LIPI), Bandung was used to conduct the tensile and flexural tests. A morphological analysis of the OPEFB fiber-polypropylene composite was carried out using scanning electron microscopy (SEM) at the Laboratory of Geological Resources Center, Bandung.

2.2. Alkaline Treatment

The OPEFB ash was dissolved in distilled water with different ash to water ratios (5, 10, and 15% wt), and the solution was stirred for five minutes. Then, the solution was allowed to settle for 48 h in order for the ash to precipitate. The extraction alkaline solution was obtained by separating the solution and the sediment. Next, the OPEFB fiber was soaked in the extraction alkaline solution for 12, 24, and 36 hours. After treatment, the fibers were stored in a desiccator before composite molding.

2.3. Composite Preparation

The composites were prepared by melt blending in an internal mixer using the Labo Plastomill (Toyo Seiki Seisaku-sho, Ltd., Tokyo, Japan), wherein the OPEFB fiber to polypropylene ratio was 40:60 (% v/v). In the preparation procedure, the polypropylene was first loaded into the internal mixer and preheated for two minutes without rotation of the roller rotor blade. Next, the OPEFB fiber was added to the melted polypropylene, and the rotor blade was rotated at a 60 rpm rotational speed for 15 min at 180°C.

2.4. Mechanical and Physical Properties

The compounded composites were then compression molded using a hydraulic hot press for five minutes under a pressure of 50 kgf/cm² at 180°C. The next step was cutting the sheet of composites into three standard test specimens (tensile strength, flexural strength, and water absorption specimens) using a dumbbell cutting machine. All specimens were stored at 23°C and 50% humidity before being tested. The number of experimental runs was 20, with one replicate of each run, which was determined by Response Surface Method-Central Composite Design (RSM-CCD) (Montgomery, 1991). Design-Expert® 7.0.0 software was used to analyze the variance model and to calculate and check the lack of fit of the current model based on the *F*-value and *P*-value. Tensile and flexural tests were conducted by using the universal testing

machine model UCT-5T (Orientec Co., Ltd., Japan). Tensile strength was tested in accordance with the JIS K 6781 for a specimen dimension of $115 \times 100 \times 2 \text{ mm}^3$. Flexural strength was tested in accordance with ASTM D 790-03 for a specimen dimension of $100 \times 127 \times 2 \text{ mm}^3$. *SEM* was utilized for observing the surface of selected specimens. Water absorption was tested in accordance with ASTM A 570, whereby specimens were immersed in distilled water for 24 h at room temperature. After immersion, the specimens were removed from the water, wiped off with a dry cloth, and weighed immediately.

3. RESULTS AND DISCUSSION

Table 1 shows the ANOVA summary of each response for tensile strength, flexural strength and water absorption under process conditions of alkaline concentration (X_1), soaking time (X_2), and fiber length (X_3).

Table 1 ANOVA Summary of Each Response

	Tensile Strength		Flexural Strength		Water Absorption	
	<i>F</i> -value	<i>P</i> -value	<i>F</i> -value	<i>P</i> -value	<i>F</i> -value	<i>P</i> -value
Model	22.1254	<0.0001	54.2826	<0.0001	17.3266	<0.0001
X_1	0.0091	0.9246	110.0913	<0.0001	13.4174	0.0010
X_2	0.0312	0.8610	154.7401	<0.0001	7.0143	0.0128
X_3	69.3333	<0.0001	82.3112	<0.0001	47.3822	<0.0001
X_1X_2	44.2904	<0.0001	67.2554	<0.0001	4.0997	0.0519
X_1X_3	42.9801	<0.0001	5.2491	0.0292	0.4096	0.5270
X_2X_3	1.4830	0.2328	46.1062	<0.0001	0.1382	0.7172
X_1^2	14.2903	0.0007	11.5092	0.0020	48.9961	<0.0001
X_2^2	15.7837	0.0004	2.8541	0.1015	5.1072	0.0312
X_3^2	8.7597	0.0060	7.8441	0.0088	41.5126	<0.0001
Lack of Fit	10.8046	<0.0001	8.2153	0.0001	32.5762	<0.0001

Note: X_1 : Alkaline Concentration; X_2 : Soaking time; X_3 : Fiber length

As depicted at Table 1, the significant level (the *F*-value) of the models were greater than $F_{0.05,9,30}$ (2.2107), and the *P*-value of the models also supported the significance of the model with a value less than 0.0001. It means that the models were significant to the all responses.

3.1. Compatibility

Alkaline treatment using sodium hydroxide (NaOH) affects the physical and mechanical properties of natural fiber composites, such as increasing surface roughness, so that mechanical interlocking (between the fiber and the matrix) can be improved. This treatment removes certain hemicellulose, lignin, and oils on the surface of fiber to increase the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites. In addition, the alkaline treatment increases the tensile and flexural strength (Li et al., 2007).

The SEM micrograph taken from the highest performance of tensile and flexural fracture surface specimens is shown in Figure 1. The images show that there is no fiber pull-out from the matrix, indicating that the adhesion between the fibers and the matrix is strong enough. This means that the alkaline treatment disrupted the hydrogen bonding in the network structure, thereby increasing surface roughness, thus improving mechanical interlocking (Akil et al., 2011).

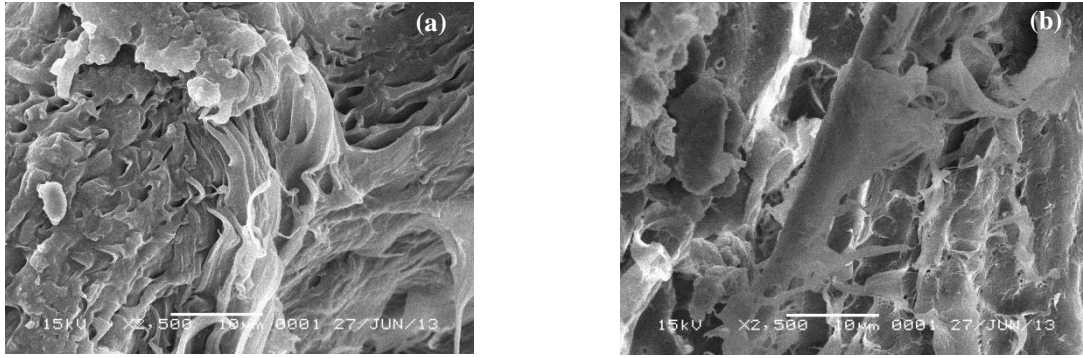


Figure 1 SEM micrograph of fracture surface: (a) tensile strength specimen; (b) flexural specimen

3.2. Mechanical Behaviour

Figure 2 shows the effect of soaking time, fiber length, and concentration of alkaline solution on tensile strength. From this figure, it is evident that the tensile strength increased with the increase of soaking time and fiber length at a low concentration of alkaline solution (5% wt). The longer soaking time at a lower alkaline concentration resulted in more lignin and hemicellulose being removed from the fiber surface. On the other hand, at a higher alkaline concentration and longer soaking time, the tensile strength of the composite decreased because of excess delignification of the OPEFB fiber, resulting in weakening of or damage to the fiber. These findings comply with those of Mishra et al. (2003). They reported that sisal fiber-reinforced polyester composite treated with 5% NaOH had better tensile strength than 10% NaOH treated composites. They concluded that, for a higher concentration, a longer soaking time should not be used, but that a longer soaking should be used for a lower concentration (Mishra et al., 2003).

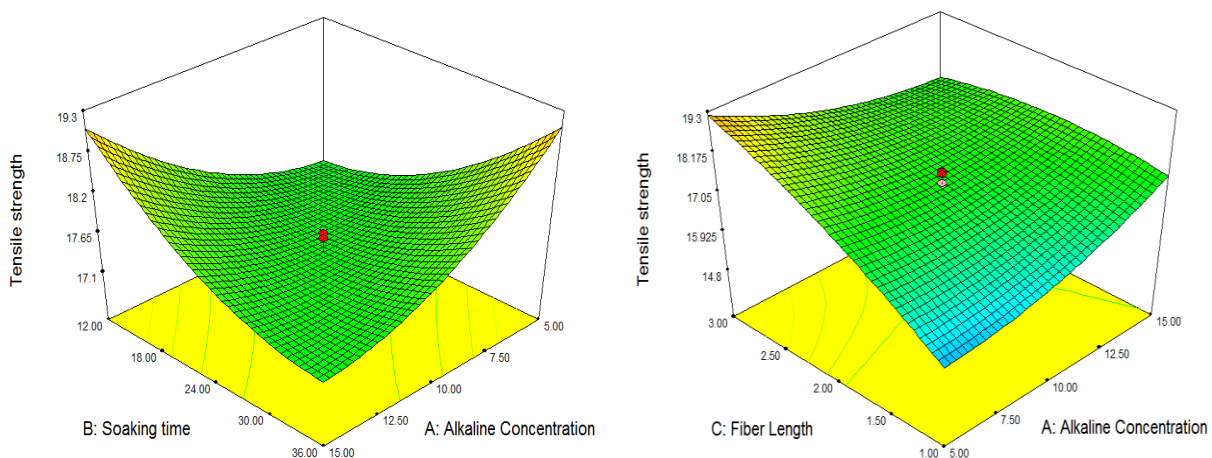


Figure 2 Tensile strength as a function of soaking time, fiber length and alkaline concentration

Figure 3 depicts a graphical representation of the flexural strength as a function of soaking time, fiber length and alkaline concentration. The longer soaking time and increased fiber length at lower concentrations of OPEFB ash extract solution increased the flexural strength and reduced the hemicellulose and lignin that cause the roughness of the fiber surface which, in turn, causes the mechanical interlocking to strengthen. Higher alkaline concentration and longer soaking time decreases flexural strength because more cellulose is dissolved with the hemicellulose and lignin during the alkaline treatment process. The result is that the strength of the fiber decreases due to brittleness.

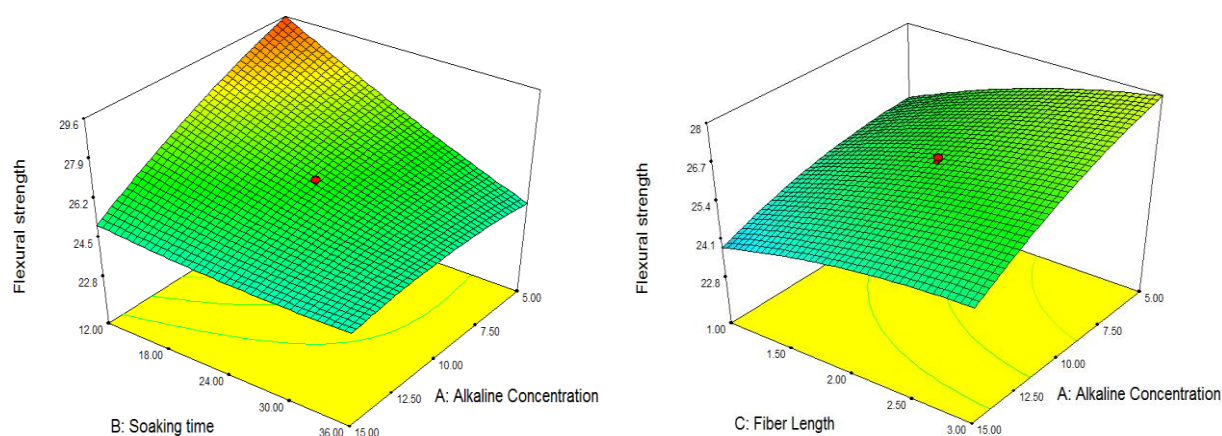


Figure 3 Flexural strength as a function of soaking time, fiber length and alkaline concentration

The highest composite flexural strength (30.216 MPa) was obtained at 5% wt alkaline solution, 12 h soaking time, and 3 cm fiber length. The flexural strength increased with increased fiber length and soaking time. Analysis of variance of the flexural strength model resulted in P -values of the terms X_1 , X_2 , X_3 , X_1X_2 , X_1X_3 , X_2X_3 , X_1^2 and X_3^2 that were less than 0.05, signifying that each mentioned term had a significant effect on the model.

3.3. Absorptivity

Water absorption was used to measure the effect of the process variables of the alkaline treatment as shown in Figure 4. Water absorption depends on factors such as fiber volume fraction, fiber orientation, interfacial bonding, fiber and matrix bonding, surface protection, and void. The main factor that affects water absorption in natural fiber reinforced composites is the hydrophilicity of the natural fiber. The poor resistance of fiber to water absorption has an undesirable effect on the mechanical properties such as lowering tensile and flexural strength and the dimensional stability of composites. The main process of water absorption consists of diffusion of water molecules inside the microgaps or microcracks between the polymer chains. The other common mechanism is capillary transport into the gaps and flaws in the interface between the fiber and polymer because of incomplete wettability, which is formed during the compounding process (Srinivasa & Bharath, 2012). Lignocellulosic fibers are hydrophilic and absorb moisture. Many hydrogen bonds (hydroxyl groups - OH) are present between the macromolecules in the fiber cell wall. When moisture from the atmosphere comes into contact with the fiber, the hydrogen bond breaks and the hydroxyl groups form new hydrogen bonds with water molecules. The cross-section of the fiber becomes the main access point for water penetration. The interaction between the hydrophilic fiber and the hydrophobic matrix causes fiber swelling within the matrix. This results in weakening of the bonding strength at the interface, which leads to dimensional instability, matrix cracking, and poor mechanical properties of the composites (Zakaria & Poh, 2002). Therefore, the removal of moisture from the fiber is an essential step in the preparation of composites. The moisture absorption of fibers could be reduced by eliminating hydrophilic hydroxyl groups from the fiber's structure through different chemical treatments (Wang et al., 2007). Fibers surface treatment increases water absorption except in alkali treated composites (Sreekala et al., 2002). Alkaline treatment promotes ionization, which substitutes the hydrogen ions in the hydroxyl groups of cellulose and lignin with alkoxide and changes the hydrophilicity of the fiber (Akil et al., 2011).

The trend of water absorption is in agreement with the trend of tensile strength, which are desirable properties achieved at lower soaking time and higher alkaline concentration. This result relates to the reduction of hydrophilic components from the fiber's surface, which

contributes to decreasing the water absorption of the composites. The lowest water absorption was 0.324%, which was obtained at 10% concentration of the OPEFB ash extract solution, 24 h soaking time, and 2 cm fiber length. Analysis of variance of the water absorption model result in P -values of the terms X_1 , X_2 , X_3 , X_1^2 , X_2^2 and X_3^2 that were less than 0.05, which means that the terms had a significant effect on the model. The interaction of soaking time with alkaline concentration (X_1X_2), soaking time with fiber length (X_2X_3) and alkaline concentration with fiber length (X_1X_3) were not statistically significant effect on the model because the P -values were greater than 0.05.

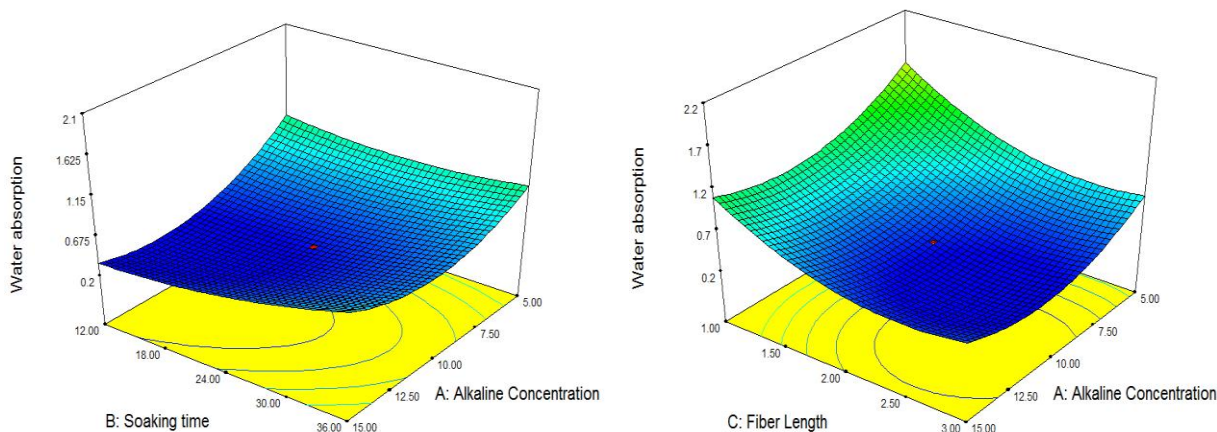


Figure 4 Water absorption as a function of soaking time, fiber length and alkaline concentration

4. CONCLUSION

The results of this study showed that an alkaline treatment using an OPEFB ash extract solution could reduce lignin and hemicellulose content in fibers, thereby increasing the surface roughness of the fibers to produce better adhesion between the fiber and the matrix. All of the variables in the alkaline treatment significantly affect the tensile strength, flexural strength, and water absorption of the composite. The alkaline concentration and soaking time influence tensile and flexural strength, which the longer soaking times and lower alkaline solution concentrations result in greater tensile and flexural strength as well as increased water absorption of the composite.

5. ACKNOWLEDGEMENT

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