

NATURAL RUBBER FLOORING GLOWING IN THE DARK

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Abstract

In this work, we studied the preparation of phosphorescent natural rubber (PNR) compounds from natural rubber (STR 5L). The internal mixer was used to blend the natural rubber and chemicals additives, the sequence of phosphor doping, and variations in the filler (calcium carbonate (CaCO_3), silica (SiO_2) and high styrene resin (HSR)), vulcanizing agent (DCP), antioxidant (BHT), and phosphor ($\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}$, Dy^{3+}) were studied. Furthermore, the phosphorescence, mechanical, and morphology properties of phosphorescent natural rubber (PNR) compounds were investigated. We discovered that the intensity of the phosphorescence was proportional to the amount of phosphor, the optimal filler mass ratio was SiO_2 to HSR (20:20 phr), BHT (1 phr), DCP (2 phr), and phosphor (20 phr). This resulted in maximum emission at 490 nm with a phosphorescence intensity of 292 a.u., tensile strength at 10.23 MPa, and hardness at 50.67 Shore A. As a result, indicating of the phosphorescent natural rubber compounds can pass the rubber flooring type tile with a hardness of 50 shore A according to Thai Industrial Standard 2377-2551 (TIS2377-2551).

Keywords: Glowing in the Dark; Natural Rubber; Phosphorescent Product

Introduction

Conventional rubber flooring, often derived from synthetic rubber, poses significant environmental challenges, including resource depletion, chemical emissions, and waste accumulation. Many synthetic rubber products are made from petroleum-based materials (Shah *et al.*, 2013), contributing to carbon emissions and long-term environmental harm due to their non-biodegradable nature. Thailand is the world's largest producer of natural rubber. In 2023, the country produced more than 4.7 million

tonnes of natural rubber. Majority of Thai natural rubber exports are currently in the intermediate rubber industries, taking rubber produced on plantations and converting this into semi-finished products, such as rib smoked sheet, blocked rubber, and concentrated latex (Chanthes *et al.*, 2023). Over the years, the drop in the price of natural rubber in Thailand has caused considerable financial strain for rubber farmers in the country due to overproduction and slowdowns in key industries like automotive

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manufacturing (which is a major consumer of rubber) (Saleh and El Mogy, 2020).

Natural rubber is a versatile material used in the production of a wide range of products. Here are some common products made from natural rubber: tires, rubber gloves, rubber bands, footwear, conveyor belts, sports equipment, medical devices, elastic fabrics, and rubber mats and flooring. Natural rubber is prized for its elasticity, flexibility, durability, resistance to wear, biodegradability, thermal and electrical insulation (Shanks and Kong, 2013). These properties give it a distinct advantage over many other materials in numerous industries. Phosphors, materials that glow in the dark through a process known as luminescence, play a crucial role in various applications. They absorb light (such as ultraviolet or visible light) and then release that energy as visible light, allowing them to “glow” in the dark (Baryshnikov *et al.*, 2017). This glow, known as phosphorescence, can last for varying lengths of time, depending on factors like the intensity of light absorbed and the duration of exposure. One widely used phosphor is Strontium aluminate doped with europium (Eu^{2+}) and dysprosium (Dy^{3+}), commonly written as $\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}, \text{Dy}^{3+}$. This particular compound is highly efficient in phosphorescence and can glow brightly and for extended periods after being exposed to light, withstanding temperatures as high

as $1,250^\circ\text{C}$ (Poulose *et al.*, 2021). Therefore, the phosphor is commonly used to make materials, including advertising cards, children’s toys, and safety indicators (Mokhtar *et al.*, 2022).

As the global population ages, one of the most common health issues among the elderly is vision problems, making daily activities more difficult and increasing the risk of accidents, such as falls, which can lead to severe injuries. Innovative solutions have been developed to improve safety and convenience in the home such as handrails and support bars (Kato *et al.*, 2020), automatic lighting systems (Papinutto *et al.*, 2022), glowing doorknob covers (McKenzie *et al.*, 2013), and glowing walkway surfaces (Saleem and Hosoda, 2021).

Therefore, in this research, the researcher aims to study the preparation of glow-in-the-dark rubber flooring products from natural rubber mixed with strontium aluminate phosphors doped with europium ions, and dysprosium ion ($\text{SrAl}_2\text{O}_4:\text{Eu}^{2+}, \text{Dy}^{3+}$) for rubber flooring type tile with a hardness of 50 shore A according to Thai Industrial Standard 2377-2551 (TIS2377-2551). This research could lead to the development of a sustainable, environmentally-friendly product (since it uses natural rubber) that meets industry standards and caters to the growing demand for smart materials in safety for elderly persons.

Table 1. Formulation of phosphorescent natural rubber compounds

Formulation	Rubber and Chemicals Materials (Part per Hundred of Rubber: phr)							
	STR 5L	Wax	CaCO_3	SiO_2	HSR	BHT	DCP	Phosphor
P0	100	1	-	-	-	1	2	-
P1*	100	1	-	-	-	1	2	20
P2*	100	1	-	-	-	1	2	20
P3*	100	1	-	-	-	1	2	20
P4	100	1	-	-	-	1	1	20
P5	100	1	-	-	-	1	3	20
P6	100	1	-	-	-	1	4	20
P7	100	1	-	-	-	1	5	20
P8	100	1	20	-	-	1	2	20
P9	100	1	40	-	-	1	2	20
P10	100	1	60	-	-	1	2	20
P11	100	1	80	-	-	1	2	20
P12	100	1	100	-	-	1	2	20
P13	100	1	-	10	-	1	2	20
P14	100	1	-	20	-	1	2	20
P15	100	1	-	30	-	1	2	20
P16	100	1	-	40	-	1	2	20
P17	100	1	-	20	20	1	2	20
P18	100	1	-	20	40	1	2	20
P19	100	1	-	20	60	1	2	20
P20	100	1	-	20	80	1	2	20
P21	100	1	-	20	100	1	2	20
P22	100	1	-	20	20	1	2	10
P23	100	1	-	20	20	1	2	30
P24	100	1	-	20	20	1	2	40
P25	100	1	-	20	20	1	2	50

* PNR compounds with various phosphor sequences were investigated

Table 2. Mixing step procedure of phosphorescent natural rubber compounds

Step	Time (min)							
	P0	P1	P2	P3	P4-P7	P8-P12	P13-16	P17-P25
STR 5L	0	0	0	0	0	0	0	0
HSR	-	-	-	-	-	-	-	2
Wax	-	-	-	-	2	2	2	4
CaCO ₃	-	-	-	-	-	3	-	-
SiO ₂	-	-	-	-	-	-	4	6
BHT	2	10	2	2	10	10	12	14
DCP	4	12	4	4	12	12	14	16
Phosphor	-	2	14	6	14	14	16	18
Total time	25 min							

Materials and Methods

Materials

STR5L-graded Standard Thai Rubber was purchased from Northeast Rubber Co., Ltd., Buriram, Thailand. Butylated hydroxytoluene (BHT), Dicumyl peroxide (DCP), Paraffin wax, and Silica (SiO₂) were purchased from the Chemical and Materials Co., Ltd. Calcium carbonate (CaCO₃) was obtained from P.S. Science Chemical Co., Ltd. High styrene resin (HSR) was purchased from Kij Paiboon Chemical Ltd. Part. Strontium aluminate (SrAl₂O₄:Eu²⁺, Dy³⁺) was purchased from Am Here Intertrade Co., Ltd. All the chemicals were commercial commodities and used as received without further purification.

Preparation of Phosphorescent Natural Rubber (PNR) Compound

The PNR compound formulations and compounding steps are shown in Tables 1 and 2. The PNR compounds were carried out with an internal mixer (MX500-TQ, Chareon Tut Co., Ltd, Thailand) at initial temperature of 50°C, the rotor speed of 70 rpm. PNR compounded rubbers were left overnight at ambient temperature in a desiccator before compression molding, and then were vulcanized by using compression molding (Qingdao Yadong Rubber Machinery Co., Ltd, China) at 160°C. The vulcanization time of each PNR compound was determined by using a Moving Die Rheometer (MDR) (Industrial Estate Co., Ltd, India) at 160°C for 18 min. The obtained PNR compound sheets were stored at ambient temperature for at least 24 h prior to characterization.

Characterizations

Phosphorescence properties were analyzed by using the fluorescence spectrophotometer model PerkinElmer-LS55. Tensile and tear properties were analyzed using a Dynisco universal testing machine (CG Engineering Co., Ltd, Thailand) in accordance with ASTM D412 and ASTM D624, respectively.

Dumbbell specimens for tensile tests were cut using die C from PNR compound sheets with a thickness of approximately 2 mm. Angle-shaped rubber specimens for tear tests were cut using die C from PNR compound sheets with a thickness of approximately 2 mm. The specimens were tested with a 1 kN load cell and a 500 mm/min crosshead speed. The hardness of PNR compounds was measured following ASTM D2240, using a Durometer Type A (Asker Model X-A series). In addition, the morphologies of PNR compounds were investigated using a scanning electron microscope (JSM-6010LV, Jeol Ltd., Tokyo, Japan) at an acceleration voltage of 15 kV. Tested dumbbell samples were immersed in liquid nitrogen for 4 min to create cryogenic fracture surfaces. After that, the samples were shattered in liquid nitrogen, sputtered with platinum, and imaged.

Results and Discussion

Phosphorescence properties of PNR compounds with various phosphor doping sequences as shown in Figure 1. The final-sequence phosphor (P3) in PNR compounds can increase the light intensity to its maximum due to the phosphor being scattered

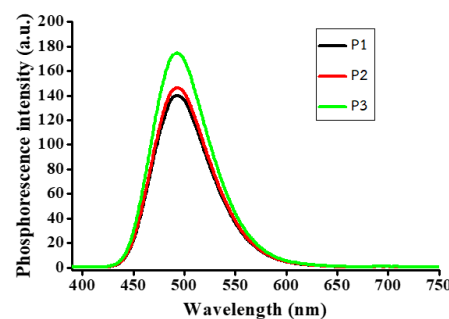


Figure 1. Phosphorescence intensity of the PNR compounds at different phosphor doping sequences

around on the surface of PNR compounds. The PNR compounds were discovered to be able to emit light at a wavelength of 490 nm.

Effect of DCP Vulcanizing Agent

As the DCP amount increases (from 1 to 5 phr), the phosphorescence intensity decreases, as shown in Figure 2. Increasing DCP content strengthens the bonding between rubber molecules, which leads to more encapsulation and masking of the phosphorescent particles due to DCP acts as a vulcanizing agent in the rubber matrix. In addition, DCP is used as a vulcanize to form acetophenone, which has a phenyl ring structure that can absorb light from light emission. As a result, the light intensity decreases (Snyder and Testa, 1979).

The mechanical properties of PNR compounds at varying content of DCP, shown in Figure 3. Increasing DCP content of more than 2 phr leads to a brittle and harder rubber, as excessive crosslinking reduces flexibility and elasticity of the rubber matrix

(Peng *et al.*, 2002), leading to lower tensile strength and elongation at break. Thus, 2 phr of DCP is the optimal content for the best performance for the PNR compound.

Effect of CaCO_3 Filler

CaCO_3 is used as a filler to reduce the overall cost while slightly reinforcing the rubber (Ismail *et al.*, 2002). The study investigates the use of varying amounts of CaCO_3 , with loadings at 0, 20, 40, 60, 80, and 100 phr. The phosphorescence properties of the PNR compound with different amounts of CaCO_3 filler, as shown in Figure 4, indicate that adding CaCO_3 in amounts between 20 and 60 phr leads to an increase in phosphorescence intensity. However, the phosphorescence intensity decreases when the CaCO_3 amounts more than 60 phr, as the excess CaCO_3 may cause the particles to cluster together, which can create a less uniform distribution of filler in the material. This agglomeration can lead to

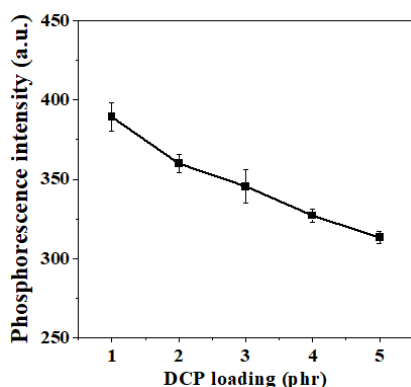


Figure 2. Phosphorescence intensity of PNR compounds at different DCP amounts
Please mention Figure 2 in text.

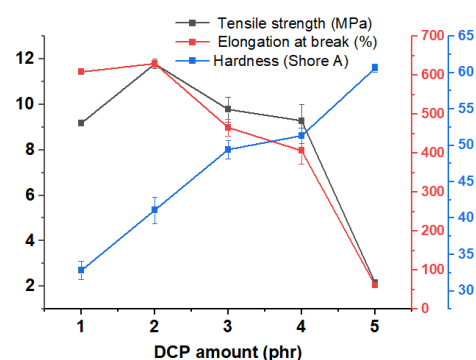


Figure 3. Mechanical properties of PNR compounds at different DCP amounts
Please mention Figure 3 in text.

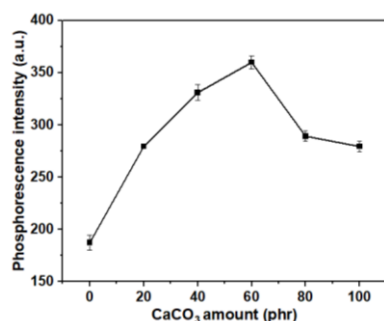


Figure 4. Phosphorescence intensity of PNR compounds at different CaCO_3 amounts

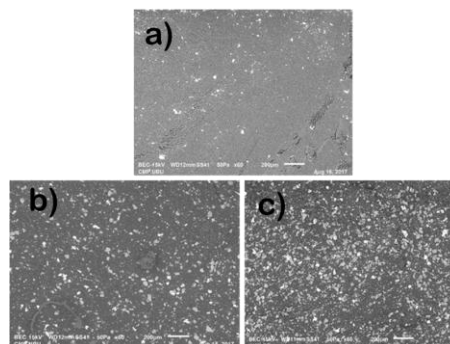


Figure 5. SEM image of PNR compounds at different CaCO_3 amounts at (a) 0, (b) 60 and (c) 80 phr

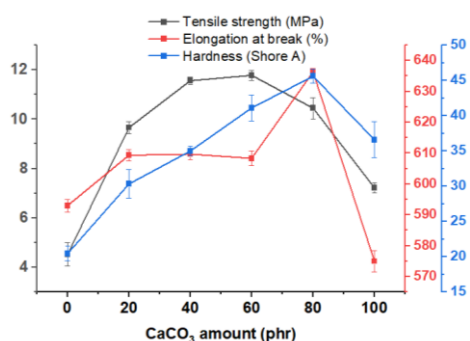


Figure 6. Mechanical properties of PNR compounds at different CaCO₃ amounts

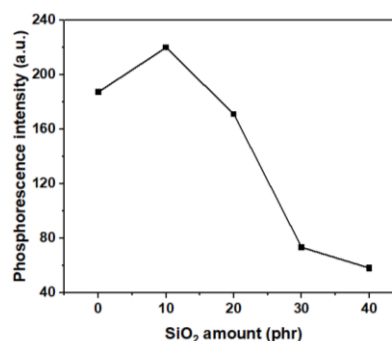


Figure 7. Phosphorescence intensity of PNR compounds at different SiO₂ amounts

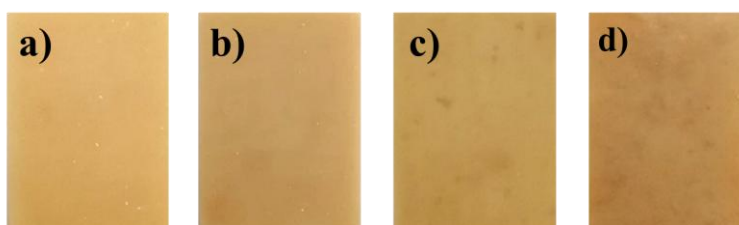


Figure 8. Physical image of PNR compounds at different SiO₂ amounts (a) 10 phr, (b) 20 phr, (c) 30 phr, and (d) 40 phr

inefficiencies in the phosphorescence process such as obscuring the phosphor of CaCO₃ filler. The SEM images can further explain this, as shown in Figure 5.

The mechanical properties of PNR compounds with different amounts of CaCO₃ filler, as shown in Figure 6. It was found that increasing the content of CaCO₃ filler resulted in increasing the tensile strength of the PNR compounds and adding CaCO₃ filler at 60 phr gave the PNR compounds a high tensile strength. While adding more than 60 phr of CaCO₃ causes too much filler to form lumps. As a result, the tensile strength tends to decrease. This is consistent with other experiments showing that rubber's tensile strength steadily diminishes after reaching a certain point regardless of the amount of filler (Leblanc, 2002). As shown in Figure 5. The hardness of PNR compounds supplemented with 20-100 phr CaCO₃ filler revealed that 80 phr CaCO₃ filler produced the maximum hardness of PNR compound at 45 shore A. Because of the CaCO₃ filler particles being placed between the rubber's molecular chains. For increasing the blazing rubber sheet's toughness, a result. However, the PNR compound's hardness value does not meet the Thai Industrial Standard 2377-2551 for rubber flooring.

Effect of SiO₂ Filler

Silica (SiO₂) as a reinforcing filler to enhance the mechanical properties of rubber. As the SiO₂ content increases (from 10 to 40 phr) the phosphorescence intensity decreases, as shown in Figure 7. This reduction in phosphorescence is attributed to the increasing SiO₂ amount, the PNR compound became darker and more opaque, as shown in Figure 8. This opacity can block light from reaching the phosphorescent material, reducing its ability to absorb light and subsequently emit phosphorescence.

The mechanical properties of the effect of SiO₂ filler amount in PNR compounds are shown in Figure 9. It was found that the SiO₂ filler amount greater than 20 phr the tensile strength and elongation at break of the PNR compounds will tend to decrease. Due to increasing the SiO₂ amount can be agglomerated in the PNR compounds could result in poor dispersion and agglomeration since silica (polar and hydrophilic surface) is incompatible with non-polar rubber (Neethirajan *et al.*, 2002). In addition, the higher temperature of PNR compound mixing causes the bond in the structure of rubber to be destroyed (Arguello and Santos, 2016). Moreover, the hardness was trend increasing because the SiO₂ filler was

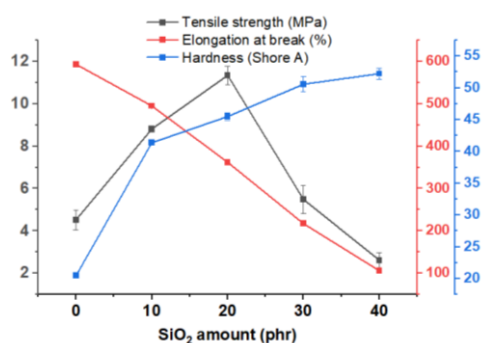


Figure 9. The mechanical properties of PNR compounds at different SiO₂ amounts

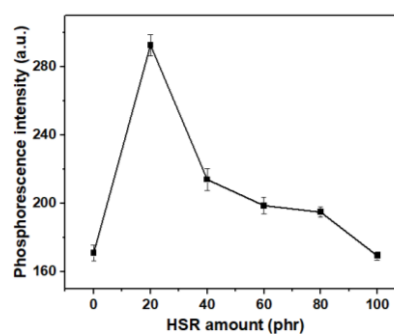


Figure 10. Phosphorescence intensity of PNR compounds at different HSR amounts

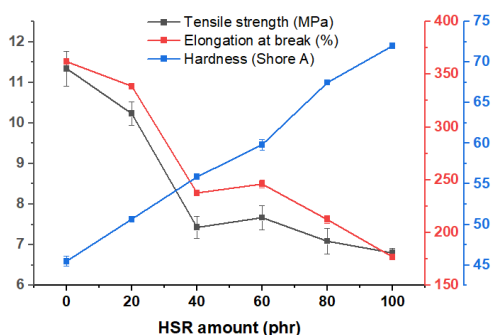


Figure 11. Mechanical properties of PNR compounds at different HSR amounts

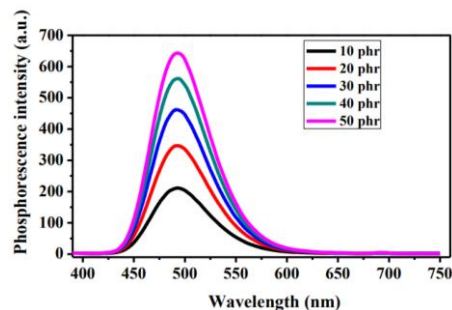


Figure 12. Phosphorescence intensity of PNR compounds by adding phosphor at the amount of 10, 20, 30, 40, and 50 phr

as a reinforcing filler. However, the PNR compound mechanical properties at different SiO₂ amounts do not meet the TIS2377-2551 for rubber flooring.

Effect of SiO₂ Combined with HSR Filler

High styrene rubber (HSR) as a reinforcing filler to enhance the hardness properties of rubber (Pal *et al.*, 2010). HSR can increase the phosphorescence intensity of PNR compound more than SiO₂ filler, as shown in Figure 10. Due to HSR is a white resin. However, as the HSR amount increases (from 20 to 100 phr) the phosphorescence intensity decreases. This reduction in phosphorescence is attributed to the increasing HSR amount, the aromatic ring in HSR structure that can absorb light (Kawanishi and Hagiwara, 1986) from light emission from phosphor in rubber matrix of PNR compound.

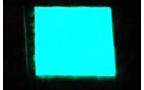
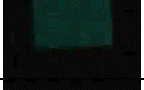
The mechanical properties of the effect of HSR filler content in PNR compounds are shown in Figure 11. It was found that the HSR filler amount greater than 20 phr the tensile strength and elongation at break of the PNR compounds will tend to decrease. Due to increasing the HSR amount

can be agglomerated in the PNR compounds. The aggregation leads to the formation of weak points in the natural rubber matrix, accordingly reducing the elastomeric strength (Sharif *et al.*, 2005) of PNR compound. Moreover, the hardness was trend increasing because the increase of HSR content results in more plastic properties of natural rubber matrix (Arguello and Santos, 2016).

The comparison of the Phosphor doping sequence, DCP vulcanizing agent content, CaCO₃, SiO₂, and HSR filler amount. It discovered that the PNR compound, which is the final addition of phosphor 20 phr, SiO₂ 20 phr, HSR 20 phr, and DCP 2 phr gives a tensile strength of more than 4 MPa, elongation at break more than 150%, and hardness more than 50 Shore A, which has good mechanical properties for rubber flooring type tile according to Thai Industrial Standard 2377-2551 (TIS2377-2551).

The light persistence time test by digital camera imaging. The distance between the camera and the PNR compounds (P17) of 1 meter, as shown in Table 3. It was found that the best formulation PNR compounds can be able to glow for 4 h, but

Table 3. Shows the persistence time of the best PNR compounds (P17) by digital camera imaging

Time (min)	Light Persistence
0	
30	
60	
120	
180	
240	

from the observation with the naked eye, the PNR compounds can glow for up to 6 h in the dark.

The phosphorescence properties of the PNR compounds also increase with an increase in the amount of phosphor, as shown in Figure 12. It was also found that increasing the amount of phosphor did not affect the mechanical properties of the PNR compounds. However, To save costs of PNR compounds in the next studies on different topics, we still chose a phosphor amount of 20 phr.

Conclusions

In this study, we successfully developed phosphorescent natural rubber (PNR) compounds using natural rubber (STR 5L) blended with various chemicals, phosphors, fillers, and vulcanizing agents. The results showed that phosphorescence intensity was directly proportional to the phosphor content, with the optimal filler mass ratio of SiO₂ to HSR (20:20 phr), BHT (1 phr), DCP (2 phr), and phosphor (20 phr) producing the best results. These properties align with the rubber flooring tile standards (TIS2377-2551), making the PNR compounds a viable candidate for applications such as flooring, especially in safety-related contexts for

elderly persons. This research opens the door to various potential applications of natural rubber. Additionally, testing PNR compounds in a broader array of applications, such as in automotive safety components, emergency exit signs, or even wearable technologies, could provide deeper insights into their versatility and performance.

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