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PERFORMANCE EVALUATION OF NATURAL RUBBER FILLED CURED ETHYLENE PROPYLENE DIENE RUBBER (EPDM) WASTE REINFORCEMENT

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ABSTRACT

Nowadays, a great attention in solving waste disposal material has been a major issue in the world. In this study, a green natural rubber flooring tile was successfully developed from waste cured ethylene propylene diene rubber (EPDM) waste as reinforcement, natural rubber (NR) as binder and was flourishingly prepared by using common internal mixer and conventional curing (CV) system. The waste content was varied from 0, 20, 40, 60, 80 phr and the good compatibility between waste and polymer matrices was depicted by the diversified testing result. The best result come from incorporation up to 60 phr cured EPDM wastes in NR where it effectively improved the hardness up to 53.5 Shore A, 5.208 MPa in tensile strength, 1.252 g/cm^3 for density, 1.1745 g volume loss for abrasion resistance test, at 0.18094 g loss, and 1.777 Mpa fo tensile modulus (M300). This was credited to the reinforcement effect of cured EPDM waste rubber which possess high stiffness and modulus in addition to good dispersion of the constituent components. Whilst other tests illustrate a reasonable value where the reduction in cycle for flex cracking test from 14000 to 3500 cycle and elongation at break from 921.55% diminished to 907.5%. From the conclusive data and observation, cured EPDM waste has great potential to be used as reinforcement filler in rubber tile flooring along with commercializing the waste materials to the next level.

Keywords: Abrasion test; cured rubber waste; flex cracking; hardness test; rubber flooring; Malaysian natural rubber

INTRODUCTION

The demands of Standard Malaysian Rubber grade (SMR) are decreasing from years to years, which is proven statistically by Lembaga Getah Malaysia (LGM) in their report on Natural Rubber Statistics 2015 [1]. Malaysian natural rubber (NR) demands are decreasing because other countries had started to develop and sell their synthetic rubbers, which impart almost equal properties to the NR [2]. However, NR has better properties over synthetic polymer due to a small proportion of protein and fatty acids which help to increase the cure rate. It also has better toughness, green strength, plasticity retention index and renewable material [3, 4].

Polymer materials have been filled with several inorganic synthetic or/and natural compounds in order to increase several properties like heat resistance, mechanical strength and impact resistance to decrease other properties like electrical conductivity or permeability for gases like oxygen or water vapor [5]. Most of the research illustrate addition of reinforcement will increase tensile strength and decrease elongation of polymer compunding. The key factors for the improvement of physical and mechanical performance are homogeneous distribution of reinforcement within the polymer matrix [6], and a strong interfacial adhesion between matrix and reinforcement. 7% of 39 million tons of waste rubber come from cured EPDM waste polymer, which is approximately 1.2 million tons generated worldwide in the year of 2005 meanwhile 4 millions of waste rubber product generated during manufacturing process of rubber [7]. Compounding waste cured EPDM with NR is a method which has not been carried out yet in rubber flooring industries and this is the opportunity to show that the Standard Malaysian Rubber, SMR 10 has their role to play in rubber flooring sector. This effort will hopefully increase the demand on Malaysian rubber once again. Furthermore, this is a solution to overcome environmental problems caused by the disposing of waste cured EPDM rubber in Malaysia.

EXPERIMENTAL

Materials and Chemicals
Natural rubber SMR 10 grade was supplied by Hock Soon Rubber Sdn Bhd. Free waste cured EPDM rubber was supplied by ZM industry and was crush using crusher machine (Dongguan Motor model YI32M-4) before used. Other chemicals including activator (Zinc Oxide), processing additives (Stearic Acid), Antioxidant (2,2,4-Trimethyl-1,2-Dihydroquinoline polymer,TmQ), filler (Titanium Dioxide, TiO_2), accelerator (Mercaptabenxothiazoles, MBTS) and crosslinker (sulphur), green color pigments was supplied by Uniworldwide Resources Sdn Bhd. All materials were commercially obtained from local market.

Mechanical Mixing
The compounds with the ingredients according to Table 1 formulation were used and mixed using Banbury internal mixer machine (Model: DW5310A) for ten minutes and sheet out using 2 roll mill machines (Sinan XX 160 A). The ingredients were prepared according to Table 1 as per formulation sequence and mixed at temperature 100°C. The compounding was sheet out by using two roll mill and rheometer machine (Hung Ta, model HT-M2000) to determine the curing time. Cure time (t90), scorch time (ts2), and maximum torque (MH) were obtained from this test. The rubber compounds were moulded by using compression moulding (Model:CM 6-0T) and hot press (Vistec Technology, Model: HP-50T) at 150°C according to the respective cure time resulting

from the curing results.

Table 1: The experimental formulation for Conventional (CV) curing system

Ingredients Content (phr)	Content (phr)
SMR 10	100
Activator	5
Processing additives	2
Anti oxidant	2
EPDM waste	0/20/40/60/80
Filler	5
Color	5
Accelerator	0.6
Crosslinker	3

Tensile properties

The sheets with 2 mm in thickness obtained from compression molded were cut into dumbbell shaped by using a Wallace die cutter. By referring to ASTM: D 412-06, the tensile test was carried out by using Instron tensile tester machine (model instron 5567) at room temperature with a crosshead speed of 500 mm/min and load cell 30 kN. Tensile strenght is given in megapascals (Mpa) by the formula F/A where F is the breaking force in newtons and A is the initial cross sectional area, in square millimetres.

Abrasion Resistance Test

Abrasion test was successfully carried out by using Akron Abrasion Tester (Hung Ta, Model: HT- 8022) and referred to a standard rubber (ASTM D394). Abrasion resistance can be expressed as abrasion resistance index (ARI) from the equation:

$$ARI= \frac{V_s}{V_t} \quad X\ 100$$

V_t : Volume loss (in mm^3) of the test rubber
V_s : Volume loss (in mm^3) of the standard rubber determined under the same test conditions

Density Test

The machine used is Densometer (EW-300SG) and densimeter was obtained in accordance to International Organization of Standardization (ISO) 2781. Density measurement was measured directly by immersing the sample in the container by using densimeter.

Flex Cracking Test

ISO 132 is the preferred method to be used for flex cracking testing of rubber. For this study, flex cracking test is used to determine the fatigue of the rubber and its resistivity towards crack growth after being exerted over repeating action of bending. Cracking can be initiated by applying a small cut on the surface of the rubber product. The cracking may vary in size, rapidly increasing and proceed to failure although it has reached the limit of flexing-fatigue resistance. The test was done by using Demattia Flex Cracking Machine (Vistec Technology- Ke Jian,Model: KJ-3029). The test was done with frequent inspection until the first minute sign of cracking was detected. The result can be expressed as the number of flexing cycles at this point and the grade of cracking reached by each test piece on each test from the machine is stopped.

Cracking was graded according to following scale:

Grade 1	The cracks at this stage look like pin pricks to the naked eye. Grade 1 if the ‘pin pricks’ are 10 or less in number.
Grade 2	-The ‘pin bricks’ exceed 10 in number -The number of cracks is less than 10, but one or more cracks have developed beyond the ‘pin prick ‘stage, i.e. have perceptible lenght without much depth but their lenght is not more than 0.5 mm
Grade 3	One or more of the ‘pin bricks’ have become obvious cracks, i.e. have perceptible lenght and little depth and their lenght is greater than 0.5 mm but not greater than1 mm
Grade 4	The lenght of the largest crack is greater than 1mmbut not greater than 1.5 mm
Grade 5	The lenght of the largest crack is greater than 1.5mm but not greater than 3 mm
Grade 6	The lenght of the largest crack is greater than 3 mm

Durometer Hardness Test (Shore A)

For hardness test, 6 mm thick of round specimen was measured with Shore A durometer which it utilizes a spring to provide intending forces. Shore A is more appropriate to be used compared to Shore D as Shore D is used to investigate the hardness of plastic material and Shore A is for rubber. For this test, five parts were tested for each of the samples at different points to get the appropriate results by using the average hardness of those parts as the actual results by reffering to ASTM D2240.

RESULT AND DISCUSSION

Tensile Properties

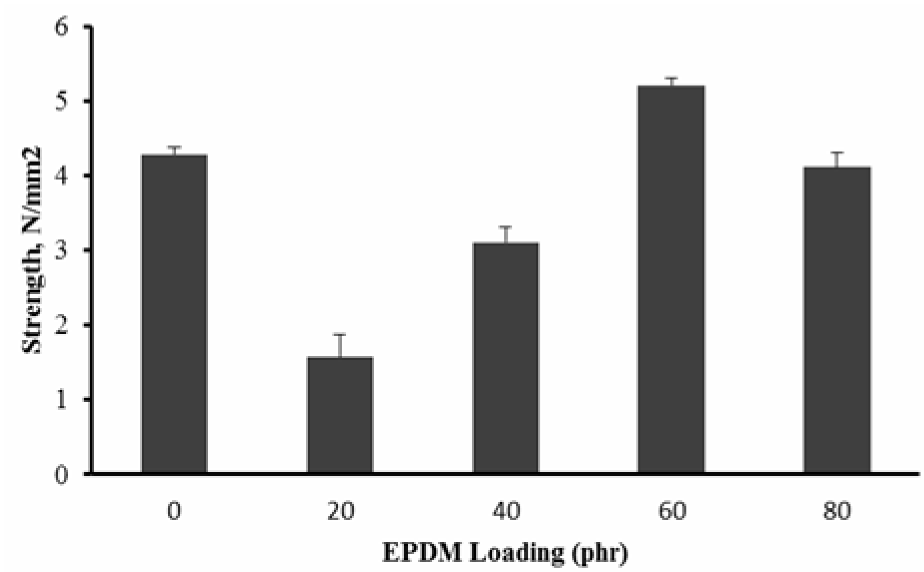

Figure 1: Tensile strenght vs loading (phr)

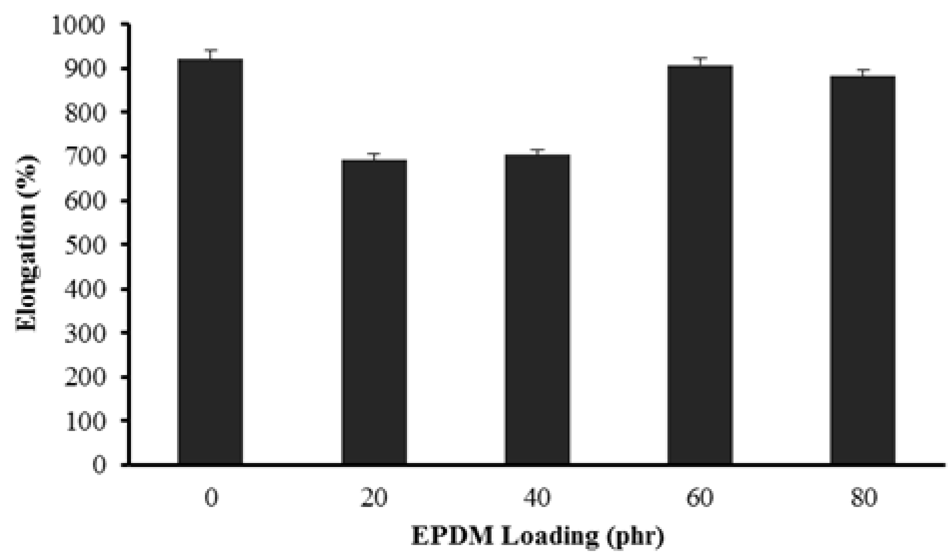

Figure 2: Elongation at break vs loading (phr)

The effect of various phr of cured EPDM waste loading on tensile strength, elongation at break and tensile modulus are shown in Figure 1, 2 and Table 3. Tensile strenght is done to ensure uniformity and consistency of the products produced. The results of tensile trenght reflects the capacity of the body to withstand a pulling force without rupture. At 80 phr, poor dispersion and adhesion towards the waste has reduced the tensile trenght results. Increasing EPDM waste loading shows enhancement in tensile strength. Results depict that at 0 phr loading, the tensile strength is lower (4.284 N/mm^2) compared to 60 phr (5.208 N/mm^2). The high strength at 60 phr is attained due to homogeneous distribution and dispersion of EPDM waste in the rubber, as a result the stress transference between EPDM waste and rubber is more effective, affecting positively the performance. Muataz et al. [8] mentioned that uniform filler dispersion is important to let matrix distributes the force to the fillers which carry most of the applied load hence able to stand the stress. The other future such as tackiness characteristic in NR also help the interaction between the waste and binder [9]. Tensile modulus (Table 3) represent the relationship between stress (force per unit area) and strain (proportional deformation) and used to measure the stiffness of the samples. The increament in reinforcement loading depicted an enhancement in tensile modulus value (mpa) at M100, M200 and M300 continuously. Reduction of elongation at break with addition of cured EPDM waste was obtained for all formulations. Addition of more cured EPDM waste in rubber matrix continuously increased the stiffness of green rubber tile which was responsible for the decrement in elongation at break.

Abrasion Test

Abrasion is the loss (wear) of material resulting from frictional action upon a surface. It is determined by comparing the wear of rubber under test with a standard rubber under the same conditions. The results were measured in terms of volumetric loss of a specimen exposed to the mechanical action of an abradant. From Table 2, eventhough the 20 phr loading density and hardness data depict higher value than the blank rubber, it shows the lowest weight (0.1325 g) and volume loss (0.1201 cm^3). At 20 phr loading, cured EPDM waste was dispersed uniformly in matrix which caused a reduction in volume loss. It also indicates good miscibility and compatibility between the waste with the rubber where the strong interaction allows rubber to hold the waste particle strongly. However, at 80 phr loading, poor dispersion is observed as proven in the increament in volume loss value at 1.2425 cm^3. Low ARI value and inflation of volume loss in increasing of EPDM waste content indicates low resistance towards abrasion. It figures out that the addition of waste will enhance the abrasion resistance towards certain extent.

Table 2: Weight loss, density, volume loss and abrasion resistance index results for rubber tile at different EPDM waste amount (phr).

EPDM (phr)	**Weight Loss (g)**	**Density, g/cm^3**	**Volume Loss (cm^3)**	**Abrasion Resistance Index (ARI)**
0	0.1640	1.146	0.1486	1142.7
20	0.1325	1.212	0.1201	1413.8
40	0.1800	1.233	0.1630	1040.5
60	0.1925	1.252	1.1745	972.93
80	0.2675	1.274	1.2425	700.15

Flex Cracking Test

Repeated flexing of a rubber vulcanizate causes cracks to develop in that part of the surface where tension stress is set up during flexing. Frequent inspections are made until the first small sign of cracking is detected on each test piece. If this part of the surface contains a crack, it can cause the crack to extend in a direction perpendicular to the stress. Table 3 indicates the continuous reduction in cycle which void formation is visible, cracking grade and hardness data. Even with good dispersion of cured EPDM waste, 0 phr loading represents the top cylic loading with 14000 instead of 20, 40, 60, 80 phr. Besides the dispersion of the reinforcement within the NR matrix, the interface between the reinforcement and NR matrix is also important [10]. The irregular shape of cured EPDM waste decreases the interfacial adhesion thus make it not able to support stress from the NR matrix. For the weak interface, it would have stress concentration, then the generation of cracks. Thus the reduction in flex cracking cycle figured out the weak interface interaction which lead to reduction of the cycle resistency and bigger cracking deformation (grade 4). This is also due to the increment in stiffness as supported in other test data like in density (Table 2), tensile modulus, shore A hardness test (Table 3) and curtailed in elongation at break for all samples. Reduction in flexibililty leads to bigger voids, rupture and deformation on the samples. Thus, it will lead to weak interface and resulted to a lower flexing strength and also a lower tension stress.

Table 3: Cracking grade, flexing test cycle number, tensile modulus, elongation at break, tensile strenght and hardness (shore A) results at different EPDM waste loading (phr)

EPDM (phr)	Crack grade	Flex Cycle	Tensile Modulus (N/mm^2)			Elongation at break (%)	Tensile Strength (N/mm^2)	Hardness (Shore A)
			M100	M300	M500			
0	3	14000	0.79	1.767	2.730	921.55	4.284	49
20	3	5000	0.83	1.797	2.713	691.65	1.567	50.6
40	3	3800	0.84	1.818	2.729	703.25	3.098	52.3
60	3	3500	0.85	1.777	2.647	907.5	5.208	53.5
80	4	2200	0.91	1.854	2.773	883.35	4.113	54

CONCLUSION

The result presents complaisant dispersion and surface interaction of the waste cured EPDM in the NR matrix by simple mixing tehnique. The best formulation at 60 phr exhibits the paramount tensile strength and elongation at break and medium hardness, modulus, density, cracking grade and cycle which void formation is visible. Thus, the waste is suitable to be used as reinforcement in the development of green natural rubber flooring tile and solving the waste disposal problem and widen up the utilization and application of malaysia NR.

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