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Performance Evaluation of Green Cushion Gum Composite Formulated Based on Hybrid Filler and Curing System

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Abstract. Cushion gum is a type of rubber composite material used as adhesive in the manufacture of retread tires. Therefore, cushion gum should have good processability and mechanical characteristics, particularly tensile property, and adhesion strength. The effect of hybrid filler and curing system on the performance of green cushion gum composite was investigated to determine the appropriate green cushion gum formula designed at a laboratory scale. The content of CB N330/lignin in hybrid filler was arranged at 40/10 phr and 50/10 phr. Meanwhile, the curing system was performed semi-efficient (CBS/S 1.6/1.6 phr) and conventional (CBS/S 1.6/2.2 phr). Green cushion gum composite was also formulated using pine tar oil as a bio plasticizer. Referred to the curing characteristic and mechanical property test result, it showed that green cushion gum composite formula coded by RF1 which composed of CB N330/lignin as 50/10 phr and applied conventional curing system which ratio of CBS/S as 1.6/2.2 phr was regarded as the acceptable combination in designing green cushion gum composite formula. Higher CB N330 loading in hybrid filler composition and conventional curing system attributed to the relatively high crosslink density indicated by MH-ML value. Consequently, green cushion gum composite was obtained using the RF1 formula that has a better optimum curing time accompanied by good tensile property and adhesion strength. The composite was also comparable to conventional commercial cushion gum.

Keywords: Composite, Curing system, Cushion gum, Hybrid filler, Natural rubber

INTRODUCTION

The development of rubber compound formulation science has grown rapidly. The history of rubber compounding is begun with the invention of vulcanization by Charles Goodyear around the 1830s. Moreover, to increase the rubber vulcanization mechanism, scientists initiated using organic accelerators in 1906 (Morawetz 2000). Nowadays, Rubber compounders worldwide prefer to use eco-friendly or bio-based rubber chemicals to substitute totally or partially petro-based rubber chemicals. Rubber chemicals mainly consist of activators, accelerators, curing agents, processing aid, filler, and anti-degradant. Every type of rubber chemical has a unique effect on the rubber composite

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properties. Some previous studies in the implementation of bio-based rubber chemicals, mainly filler and processing oil, had been conducted by rubber technologists. Sheikh et al. (2017) investigated the effect of a large amount of kaolin (China Clay) on the properties of sulfur-cured natural rubber, polybutadiene rubber, and EPDM composite.

Meanwhile, Hasan et al. (2020) observed the curing characteristic and physical properties of natural rubber by using clay and modified clay as a filler. Another rubber technologist, Petrovic et al. (2013), reported the application of polymerized soybean oil of different molecular weights as a plasticizer in NR/SBR composition. Thus Xu et al. (2020) evaluated the performance of bio-plasticizer from soybean oil modified at the various dosage of sulfur in the characteristic of tire tread rubber.

Retreading tire industry is the second-largest downstream rubber industry in Indonesia. The industry consumes about 25% of total natural rubber domestic production and is driven mostly by small and medium enterprises. Referred to economic reasons, the market share of retread tires is dominated by the logistic and public transportation industries. They should carry out tire replacement frequently, as a consequence, the business runs fluently. Retread tires offer an economic benefit by providing longer service life of a tire. Moreover, it is also more environmentally friendly due to the reduction of the hazardous waste potential that originates from tire replacement (Qi et al. 2011; Thomas & Gupta 2016; Niza et al. 2014; Simic & Dabic-ostojic 2017).

A retread tire is composed of an old used tire (casing layer), cushion gum layer, and new tread layer. Cushion gum is a rubber composite that is applied to facilitate the attachment of a new tread to the surface of the casing layer, either in the hot or cold retreading process (Sasikumar et al. 2010; Abdul-kader & Haque 2011). In accordance with the function, cushion gum requires high adhesive strength on rubber-to-rubber bonding to minimize retread tire damage caused by peeling off the tread layer from the casing layer. The selection of rubber chemicals in cushion gum production plays an important role in determining adhesive strength (Banerjee. 2015). Specifically, Azura et al. (2014) stated that tackifier resin, filler, and plasticizer highly affect the adhesion strength of rubber composite.

The usage of bio-based rubber chemicals resulted in green cushion gum composite. A hybrid filler system composed of carbon black (CB) and sodium lignosulfonate (lignin) is promising to be applied in producing green cushion gum composite. Utilization of CB/lignin in green cushion gum composite exhibits advantages such as lower viscoelastic dissipation. Thus, the presence of the phenolic group in the lignin macromolecular chain makes it a potential substitute for adhesive (Bahl 2014; Thuraisingam et al. 2019). A special type of bio plasticizer that can build auto adhesion is pine tar oil. Pine tar oil is a sticky–tacky dark brown semi-viscous liquid derivate from the destructive distillation of pine wood (Barnes & Greive 2017). Therefore, the combination of CB/lignin and pine tar oil in green cushion gum rubber composite formulation is determined can improve adhesive strength.

In addition to adhesive strength, tensile properties and hardness of cushion gum should also be considered. The performance of the property is associated with the degree of crosslinked density. Crosslink density is defined as the number of sulfur atoms bonded between two carbon atoms of two adjacent chains in a rubber structure. Carbon and sulfur bonds can be in the form of monosulphides, disulfides, or polysulphides types and are predominant according to the curing system. The curing system (conventional, semi-efficient, and efficient) is categorized depending on the ratio of the accelerator to sulfur (Hiranobe et al. 2021).

The objective of the research was to evaluate the effect of a hybrid filler and curing system, which were arranged at various ratios of CB/lignin and CBS/sulfur, on the performance of green cushion gum composite at a laboratory scale. The most appropriate rubber compound formula design, which was determined by selecting the most optimum hybrid filler and curing system, will be further used as a reference at a larger scale of production.

METHODOLOGY

Material

Technically Specified Natural Rubber grade Standard Indonesian Rubber (SIR) 20 provided by INIRO Indonesia was applied as a base elastomer. Hybrid filler is composed of carbon black N330 and sodium lignosulfonate (lignin). CB N330 was a product from OCI Company Ltd, Republic of Korea, while lignin was purchased from LUG Chemical Indonesia, Indonesia. In the research, lignin was used without further purification or chemical modification. Pine tar oil as bio plasticizer was kindly provided by PT Organik Inti Indonesia, Indonesia.

Other ingredients used in the formulation of green cushion gum consisted of zinc oxide (Lanxess Germany), stearic acid (Rhein Chemie Germany), 2,2,4-Trimethyl-1,2-dihydroquinoline (TMQ, Lanxess Germany), N-(1,3-dimethylbutyl) N′ - phenyl - p - phenylene diamine (6PPD, Sinorgchem China), paraffin wax (Rhein Chemie Germany), coumarone resin (Lanxess Germany), N-Cyclohexyl-2-benzothiazole sulfenamide (CBS, Kemai China), and Sulphur (Miwon China) were supplied by PT Multi Citra Chemindonusa Indonesia. All rubber chemicals were in technical grade (purity below 90%).

Research Procedure

The formulation of green cushion gum composite was designed in accordance with Table 1.

Table 1. Green Cushion Gum Compound Formulation Design

Materials	RF0	RF1	RF2	RF3
SIR 20	100	100	100	100
Zinc oxide	5	5	5	5
Stearic acid	2	2	2	2
TMQ	2	2	2	2
6 PPD	2	2	2	2
Paraffin wax	3.5	3.5	3.5	3.5
Coumarone resin	5	5	5	5
Pine Tar Oil	5	5	5	5
CB N330	40	50	40	50
Lignin	10	10	10	10
CBS	1.6	1.6	1.6	1.6
Sulfur	2.2	2.2	1.6	1.6

The formula applied various compositions of CB N330/lignin and CBS/sulfur, which indicated hybrid filler and curing systems, respectively. The ratio of CB N330/lignin was arranged at 40/10 part per hundred rubber (phr) and 50/10 phr. Meanwhile, the CBS/Sulfur content was varied at 1.6/1.6 phr (semi-efficient) and 1.6/2.2 (conventional). In the research, no repetition was made in the production of green cushion gum composite. The green cushion gum composite test data result was analyzed quantitatively.

The formula coded with "Reformulation

(RF) 0" composed of CB N330/lignin as 40/10 phr and CBS/Sulfur as 1.6/2.2; RF1 composed of CB N330/lignin as 50/10 phr and CBS/Sulfur as 1.6/2.2; RF2 composed of CB N330/lignin as 40/10 phr and CBS/Sulfur as 1.6/1.6; and RF3 composed of CB N330/lignin as 50/10 phr and CBS/Sulfur as 1.6/1.6, respectively.

The laboratory scaled two rolled open mill (Berstorf) was used in the preparation of green cushion gum compound. The compounding procedure referred to the ASTM D 3182, which began with the mastication of SIR 20 into softened mass in order to facilitate the mixing of the high amount of rubber chemical into the natural rubber matrix. Continuous addition of rubber chemicals through the following order coumarone resin – ZnO – stearic acid – pine tar oil – hybrid filler (CB/lignin) – TMQ – 6PPD – paraffin wax – CBS and sulfur. The rubber compound mixture was then milled and homogenized before being kept at room temperature for 20 hours.

Instrument and Data Analysis

Every green cushion gum obtained from the compounding process was sampled at 50 g for curing characteristic analysis. Procedure for curing characteristic analysis was developed by IRRI, and was carried out at 150 °C for 30 minutes by using MDR Alfa 2000 (Alfa Technologies, Akron, USA). The residual green cushion gum compound was then press-cured into a vulcanized rubber plate at a temperature of 150 °C for the optimum cure time (tc90) obtained from the curing characteristic analysis using a hydraulic press machine.

Green cushion gum vulcanizates were used as the sample for physical and mechanical properties evaluation which included the following parameters hardness (ISO 48-4 "Rubber, vulcanized or thermoplastic – determination of hardness," Frank Durometer Shore A), tensile strength, elongation at breaks, and modulus 300% (ISO 37 type 2 "Rubber, vulcanized or thermoplastic – determination of tensile stress-strain properties," UTM MTS), tear strength (ISO 34 "Rubber, vulcanized or thermoplastic – determination of tear strength," UTM MTS), specific gravity (ISO 2781 "Rubber, vulcanized or thermoplastic – determination of density"), and adhesion strength (ISO 814 "Rubber, vulcanized or thermoplastic – determination of adhesion strength," UTM Instron).

The physical and mechanical test results of the green cushion gum composite were analyzed by applying standard deviation statistical analysis. The green cushion gum composite characteristic was further evaluated by comparing it with commercial conventional cushion gum rubber composite (CC-CG) obtained from one retread tire industry located in Bogor, West Java, Indonesia.

RESULTS AND DISCUSSION

Curing Characteristic

The influence of hybrid filler and curing system on the curing process of green cushion gum compound is summarized in Table 2. At the same hybrid filler ratio, the curing system had a significant effect on the changes of modulus torque maximum (MH) and minimum (ML). Formela et al. (2015) stated that MH indicates stiffness and shear modulus of the vulcanized sample, while ML corresponds with compound viscosity and processability. Independently from the hybrid filler ratio, the higher value of MH and ML were observed for green cushion gum cured with a conventional curing system. Their higher stiffness and lower processability were caused by the decreased mobility of the macromolecular rubber chains, which was also confirmed by the level of hardness value (Fig. 1). The increase of MH and ML, as shown by the application of the conventional curing system, was strongly correlated with the formation of cross-linkage between the rubber chains bridged with sulfur atoms. The crosslink density of the rubber vulcanized sample is represented by MH-ML (ΔM).

Table 2. Curing Characteristic of Green Cushion Gum Compound at 150 °C

Parameter	RF0	RF1	RF2	RF3
MH	10.19	10.28	7.47	9.55
ML	0.70	0.81	0.60	0.68
MH-ML	9.49	9.47	6.87	8.87
t90	7.39	5.02	5.28	5.07
ts2	2.46	2.16	3.00	2.31
CRI	20.28	34.97	43.86	36.23

The ratio of hybrid filler influenced the scorch time (ts2) and optimum curing time (t90), but on the contrary, the semi-efficient curing system had no essential impact on this parameter. The higher value of ts2 and t90 was shown by the addition of CB N330/lignin as 40/10 phr. This phenomenon was due to the chemical nature of lignin used. Lignin molecular surface presents phenolic groups which inhibited curing reaction. According to Liu et al. (2013), the phenolic group contained in the lignin molecular chain had played the role of a radical scavenger for curing reactions. Furthermore, the surface of the lignin molecule was also potentially adsorption the activator (ZnO), which contributes to the disruption of sulfur curing efficiency (Aini et al. 2019).

The combination of CB N330/lignin as 40/10 phr and CBS/S as 1.6/2.2 phr (formula coded by RF0) showed the lowest CRI. A similar observation was recently presented by Formela et al. (2015), where the application of a conventional curing system resulted in the lowest CRI values. Meanwhile, the formula coded by RF2, which applied CB N330/lignin as 40/10 phr and CBS/S as 1.6/1.6 phr, offered the highest ts2 value, which enabled safer processing of rubber compound.

Hardness

The average hardness of green cushion gum composite with different ratios of hybrid filler and curing system is revealed in Figure 1. In line with the MH-ML value, the hardness drops dramatically for the compound of hybrid filler with CB N330/Lignin 40/10 phr by applying semi efficient curing system (RF2). Similar research findings were also confirmed by Kruzelak et al. (2017), which said that the hardness of rubber compounds depends on the crosslink density resulting from the type of curing system.

However, as seen in Figure 1, in a conventional curing system, the hardness of the green cushion gum composite of CB N330/lignin hybrid filler with 50/10 phr (RF1) is close to that of the green cushion gum composite of CB N330/lignin 40/10 phr (RF0). This closeness in hardness values may be attributed to the good dispersion of the carbon black aggregates in the rubber matrix facilitated by the presence of lignin. Bahl et al. (2014) reported that some lignin particles entered the space between the rubber and carbon black molecular linkage and prevented carbon black particles from forming a network of their own. This hybrid filler reinforcement mechanism was also shown by the RF3 green cushion gum composite.

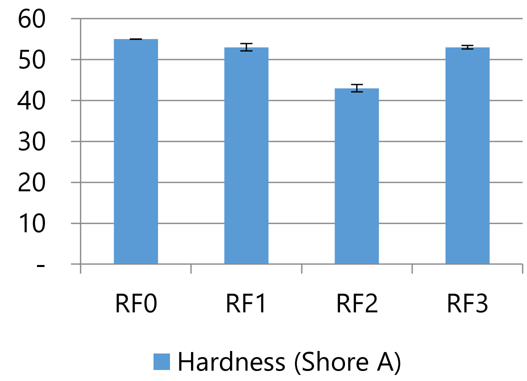

Fig. 1: Hardness of Green Cushion Gum

Tensile Properties

The tensile strength, modulus of 300%, and elongation at break for all the green cushion gum composite are presented in Figures 2 and 3, respectively. The results show that RF1 has the highest tensile strength and modulus of 300% compared to another green cushion gum sample. From Figure 2, it becomes clear evidence that the tensile strength and modulus of 300% of green cushion gum composite were improved with the increasing amount of carbon black and sulfur loading in the applied rubber compound formula. By contrast, the reduction of tensile strength and modulus of 300% was observed in the case of lower sulfur loading in an applied semi-efficient curing system. The modulus of 300% of the RF3 green cushion gum composite is not detected in Figure 2 since the RF3 green cushion gum reached 230% elongation.

Nie et al. (2017) stated that natural rubber has the ability to crystallize due to the exposure of external stretching. The rubber crystallization phenomenon is known as strain-induced crystallization (SIC). Referred to Flory Models, SIC is the function of crosslink density since the ability to crystallize is performed by the high regularity of rubber macromolecule (Huneau 2011; Candau et al. 2011). During vulcanization, the structure of the molecular rubber chain transformed from amorphous into crystal phase. Therefore, increasing the crosslink density of the vulcanized green cushion gum-based natural rubber resulted in higher SIC and elasticity, indicated by higher tensile strength and modulus of 300%.

The degree of crosslink density also plays a role in determining elongation at breaks of the vulcanized rubber. As shown in Figure 3, the rise in crosslink density apparently resulted in a linear decrease of elongation at breaks. The significant reduction of the elongation at breaks was attributed to the addition of the higher amount of carbon black and reduction of sulfur content. The presence of more carbon black particles dispersed among the rubber inter crosslink chains caused the increasing restriction of the crosslink joints (Zhao et al. 2011). A similar finding was also confirmed by Rattanasom et al. (2005), who confirmed that an increase in the carbon black content and crosslink density account for the reduction of elongation at breaks.

Tear Strength

Tear strength is important to evaluate the cushion gum performance since it measures the ability of vulcanized green cushion gum to resist the growth of any cuts under tension. The superior tear strength of a vulcanized green cushion gum means it has better resistance to the expansion of a tear (tear propagation). A tear in the cushion gum surface is regarded as triggering the detachment of a new tread on a retread tire.

Plots of tear strength as a function of hybrid filler loading and curing system are compared in Figure 4. Figure 4 reveals that the greater loading of carbon black added into the green cushion gum formulation exhibit greater tear strength in the same curing system. However, it appears that the tear strength of the vulcanized green cushion gum at the same hybrid filler composition becomes comparable in conventional or semi-efficient curing systems. This result infers that the tear strength of the vulcanized green cushion gum-based NR is independent of the curing system, primarily when a high amount of carbon black is added to the rubber compound formulation.

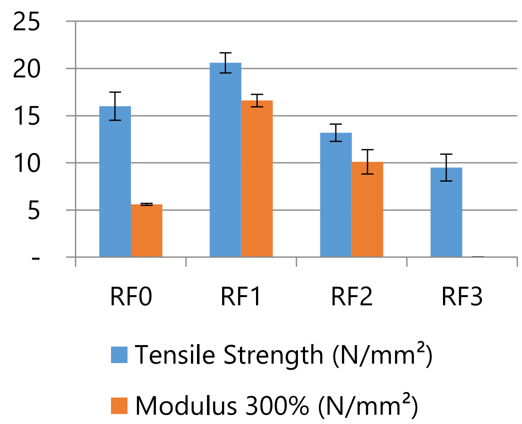

Fig. 2: Tensile Strength and Modulus 300% of Vulcanized Green Cushion Gum

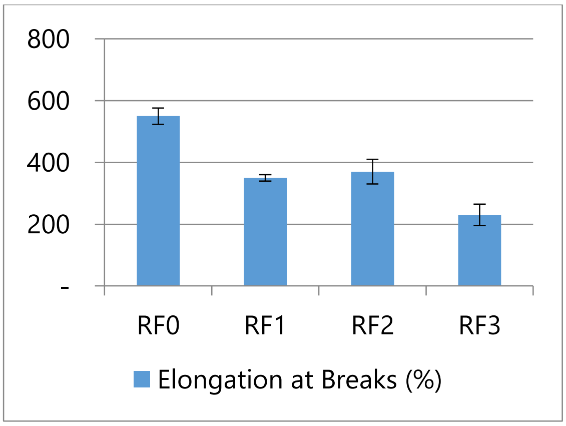

Fig. 3: Elongation at Breaks of Vulcanized Green Cushion Gum

Specific Gravity

Commercial cushion gum product requires soft and light rubber sheet with relatively low specific gravity. Therefore, the hardness of the vulcanized green cushion gum is closely related to the specific gravity. In general, the addition of filler into rubber composite with various dosages has an effect on the difference in specific gravity and hardness value.

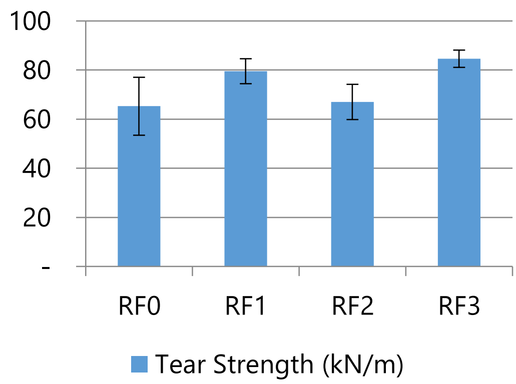

Fig. 4: Tear Strength of Vulcanized Green Cushion Gum

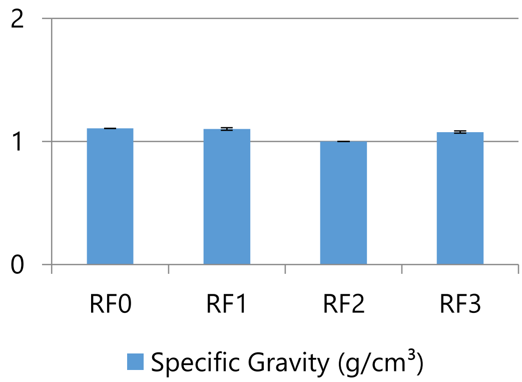

Fig. 5: Specific Gravity of Vulcanized Green Cushion Gum

As displayed in Figure 5, the specific gravity of the vulcanized green cushion gum is linear with the hardness value (Fig. 1). Vulcanized green cushion gum composed of hybrid filler N330/Lignin 40/10 phr cured by a semi-efficient system (RF 2) showed a sharp drop of specific gravity. It is likely confirmed that the carbon black loading and curing system, which affects the hardness, is also responsible for the specific gravity value of vulcanized green cushion gum. The low level of crosslink density of RF2 proposed by MH-ML value, as present in Table 2, evidently exhibits the lowest specific gravity level rather than the other green cushion gum formula.

Adhesion Strength

Concerning the cushion gum quality evaluation, the adhesion strength parameter should be determined. The parameter indicates the ability of cushion gum to prevent the retread tire defect caused by peeling off the new tread from the surface of the used old casing tire. In addition, natural rubber-based adhesive generates a vital role in the adhesives used in cushion compounds for bonding procured tread to the casing (rubber to rubber bond) (Job and Joseph 1995). A study conducted by Bhowmick et al. (1989) found that the adhesion strength of rubber joints could be improved by using a filler or specific crosslink agent in the formulation of rubber compounds.

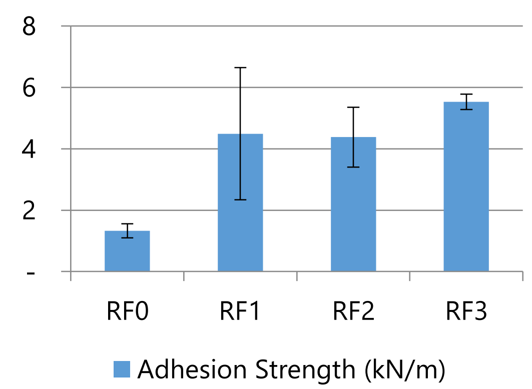

Fig. 6: Adhesion Strength of Vulcanized Green Cushion Gum

In the experiment, the adhesion strength of the vulcanized green cushion gum is illustrated in Figure 6. In Figure 6, we show that increasing carbon black content in the hybrid filler produces a better adhesion strength value in the same curing system. Positive change in adhesion strength also resulted from the reduction of sulfur loading at the same hybrid filler system. Semi-efficient curing system resulted in cohesive bond failure, which allows for greater adhesion strength. In correlation with tear strength, vulcanized green cushion gum formulated with higher carbon black content possesses larger adhesion strength.

Table 3. Comparison of cushion gum composite characteristic

Parameter	RF1	CC-CG
MH	10.28	12.20
ML	0.81	0.82
MH-ML	9.47	11.38
t90	5.02	9.18
ts2	2.16	1.59
CRI	34.97	13.18
Hardness	53	55
Tensile strength	20.6	19.4
Elongation at breaks	350	370
Modulus 300%	16.6	15.2
Tear strength	79.5	65
Specific gravity	1.101	1.126
Adhesion strength	4.49	0.69

Evaluation of the Green Cushion Gum

Conventional commercial cushion gum (CC-CG) was used as a reference to evaluate the green cushion gum composite performance. The characteristic comparison between CC-CG and green cushion gum composite is summarized in Table 3. In accordance with the result of physical and mechanical properties of green cushion gum composite as previously mentioned above, it is determined that green cushion gum composite coded RF1 has the closest quality to the CC-CG.

The curing characteristic result shows that CC-CG has slightly higher MH-ML, longer t90, but faster ts2 than RF1. The value of ML is the same between CC-CG and RF1, which indicates that both composites have similar processing behavior. The extreme difference in mechanical property is shown by adhesion strength. RF1 resulted in about 6.5 times of adhesion strength compared to CC-CG.

CONCLUSIONS

In this work, we evaluated the green cushion gum composite performance in accordance with the processability and physical-mechanical properties, using four different compound formulations in two curing systems: semi-efficient (CBS/S 1.6/1.6 phr) and conventional (CBS/S 1.6/2.2 phr) and in the presence of two ratios of hybrid filler systems CB N330 /lignin: 40/10 phr and 50/10 phr.

Green cushion gum rubber compound formula consisting of CB N330/lignin 50/10 phr and CBS/S 1.6/2.2 phr which was coded by the RF1, is regarded as the most appropriate formula design to be developed at a larger scale of production. Green cushion gum composite produced based on this formula has better processability accompanied by better mechanical properties, which are primarily indicated by short optimum curing time, proper hardness values, higher tensile property, and adhesion strength. RF1 green cushion gum composite also indicated better characteristics compared to conventional commercial rubber cushion gum composite (CC-CG), foremost on adhesion strength property.

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