

Effect of Rice Husk Powder on Compression Behavior and Thermal Stability of Natural Rubber Latex Foam

Shamala Ramasamy,^a Hanafi Ismail,^{a,*} and Yamuna Munusamy^b

Rice husk constitutes the largest by-product of the rice milling industry. Numerous research studies have been conducted on the utilization of rice husk powder (RHP), but research on incorporating RHP into natural rubber latex foam (NRLF) is lacking. The purpose of this study was to use RHP as a filler for NRLF. A compression test was performed on RHP-filled NRLFs, and they were compared with the control NRLF. The compression set increased, while the recovery percentage decreased, as RHP loading increased. The stress versus strain curve showed increasing stress with increasing RHP loading. The increase in thermal stability and decreased (Q_t/Q_0) value of RHP-filled NRLFs indicated good interaction between RHP and NRLF. RHP incorporation can contribute to NRLF industries with environmentally friendly products such as mattresses and cushioning materials by lowering costs.

Keywords: Natural rubber latex; Foam; Natural fiber; Rice husk powder; Compression test; Compression set behavior; Swelling; Rubber filler interaction; Thermogravimetric analysis

Contact information: a: School of Materials and Mineral Resources Engineering, Universiti Sains Malaysia, Engineering Campus, 14300 NibongTebal, Penang, Malaysia; b: Faculty of Engineering and Green Technology, Universiti Tunku Abdul Rahman 31900 Kampar, Perak, Malaysia;

*Corresponding author: hanafi@eng.usm.my

INTRODUCTION

Recently, environmental considerations and public concern have become increasingly important as the world strives toward environmental quality and preservation through sustainable development and cleaner technology (Li *et al.* 2011). Interest in incorporating bio-fibers has increased in direct relation to the increasing concern about environmental benefits (Rozman *et al.* 1997). The development of biomaterials holds great promise for mitigating many of the world's sustainability problems, offering the potential of renewability, biodegradation, and a path away from harmful additives (Johar and Dufresne 2012). The use of natural fibers as a reinforcing material in polymer composite materials is still attracting much interest at both the academic and industrial levels. Currently, researchers and material scientists all over the world are focusing their attention on the effective utilization of these natural fibers in polymer composites (Premalal *et al.* 2002). Banana, sisal, hemp, jute, pineapple, bamboo, cotton, coconut, rice husk, and kenaf are some of the emerging bio fillers used in polymer composites. The growing interest in bio fillers is primarily because of their renewable origin, relatively high specific strength, light weight, and competitive specific mechanical properties. In addition to their abundance and renewability, the utilization of bio-fillers has advantages for the economy, the environment, and technology (Cao *et al.* 2012; Khalf and Ward 2010).

Rice husk is an agricultural waste material abundantly available in some states of Malaysia and other rice-producing countries. The rice milling industry generates a large quantity of rice husks during the paddy milling process. Societies, especially among the rice millers, often dispose of the rice husk waste using open burning. This situation directly leads to environmental concerns and becomes a great environmental threat, causing damage to the land and the surrounding area in which it was dumped. Rice husks are the natural sheaths that form on rice grains during their growth. Removed during the refining of rice, these husks have had no commercial interest (Johar and Dufresne 2012; Yalcin and Sevinc 2001; Ciannamea and Ruseckaite 2010). Rice husks consist of 35% cellulose, 25% hemicellulose, 20% lignin, and 17% ash (silica 94%), by weight (Premalal *et al.* 2002). Research efforts are in progress to incorporate rice husk powder (RHP) into polymers to enhance their physical, mechanical, and tribological properties (Chand and Prabhat Fahim 2010). RHP has been incorporated into various kinds of polymer matrix such as high-density polyethylene, low-density polyethylene, polypropylene, styrene butadiene rubber, linear low-density polyethylene blends, and polyurethane (Khalf and Ward 2010; Zhao *et al.* 2009; Yang *et al.* 2004; Kim *et al.* 2006).

Natural rubber latex (NRL), the first latex to be used industrially, comes from the *Hevea brasiliensis* tree. NRL is also defined as the dispersion of natural rubber particles in water (Hossain *et al.* 2010; Schidrowitz and Dawson 1952; Roberts 1988). NRL is a stable colloidal dispersion of polymeric particles in an essentially aqueous medium (Hamza *et al.* 2008; Rahman *et al.* 2009; Varghese and Karger-Kocsis 2003). The colloidal stability of the latex is extremely sensitive to pH and the ionic environment of the dispersing medium (Nawamawat *et al.* 2011). Schidrowitz and Goldsbrough (Blackley 1966) made an attempt in 1914 to produce a porous rubber product from natural latex concentrates, called “latex foam” (Madge 1962). Addition of any kind of foreign material with different pH can easily cause agglomeration of latex. Latex foam rubber is defined as a cellular rubber that has been made directly from liquid latex. The Dunlop process is particularly well adapted to the manufacturing of thick molded latex foam rubber products, such as pillows, cushions, mattresses, and upholstery foam (Blackley 1966). Various applications of latex foam include carpet underlay material, mattresses, pillows, cushioning seats at concert halls, theaters, hotels, houses, vehicles, and factories (Madge 1962; Blackley 1966; Murphy 1952).

General principles can be given for the preparation of aqueous solutions, dispersions, or emulsions for addition to aqueous latices. The particle size of dispersion and colloid stability should be comparable to that of the latex to which they are added. Also, the pH and ionic strength of the aqueous phase of the dispersion should be similar to that of the latex aqueous phase. Few attempts have been made to incorporate fibers into natural rubber latex foam (Blackley 1966). The use of glass fiber to reinforce natural latex foam rubber has been studied in some detail, and such an approach is claimed to reduce shrinkage and increase tensile strength (Madge 1962). Mineral fillers such as kaolinite clays and calcium carbonate have also been added as fillers for latex foam (Blackley 1966). Improvements in properties of high styrene-butadiene lattices have also been claimed by addition of mineral fillers. In general, however, the addition of any normal type of compounding ingredient gives little advantage with respect to the load-weight ratio (Murphy 1951; Blackley 1966). Despite that, to the best of our knowledge, there have been no research studies that have incorporated RHP as filler in natural rubber latex foam. So far, there have been no reports of adding natural fibers to natural rubber latex foam.

Goals of the present study are to understand the effect of RHP on the mechanical and thermal stability of latex foam, as well as to reduce the overall compounding cost of the NRLF. The compression properties, thermal stability, and swelling behavior of controlled NRLF and RHP incorporated NRLF were analyzed. RHP filler is expected to enhance the mechanical and thermal properties and also reduce the material cost of natural rubber latex foam. Usage of RHP is also expected to reduce land fill pollution and contribute as an asset to the rice milling industry rather than being waste material.

EXPERIMENTAL

Materials and Formulation

Table 1 lists the formulations and materials used in this study. NRL (low ammonia (LATZ)-type) and latex chemicals (sulfur, antioxidant, potassium oleate, ZDEC, ZMBT, zinc oxide, DPG, and SSF) supplied by Zarm Scientific & Supplies Sdn. Bhd, Malaysia, were used in this research. Rice husks supplied by Kilang Padi Nazra Sdn. Bhd, Malaysia, were collected, grounded, and sieved into an average particle size of 140 μm .

Table 1. Formulation of Natural Rubber Latex Foam Filled with Rice Husk Powder

Ingredients	Total Solid Content(%)	Formulation(phr) *
LA Latex	60	100
Sulfur	50	2
Antioxidant	50	1
Potassium Oleate	20	4
ZDEC	50	1
ZMBT	50	1
Zinc Oxide	50	3
DPG	40	0.3
SSF	20	1.2
Rice Husk Powder	25	2.5

ZDEC- zinc diethyldithiocarbamate

ZMBT- zinc 2-mercaptobenzthiozolate

DPG- diphenylguanidine

SSF-sodium silicofluoride

* Amounts are given as parts per hundred parts of latex (phr).

Sample Preparation

LATZ-type NRL was sieved, measured, and stirred using a mechanical stirrer for about 30 min. Next, a vulcanizing agent (sulfur), an antioxidant, and potassium oleate soap were added and stirred at 10 rpm. After 2 h, accelerators (ZMBT and ZDEC) were slowly added to the mixture. Then, the NRLF compound was matured for 8 h at room temperature with continuous stirring at 10 rpm. After 8 h of maturation, the NRLF compound was intensively beaten and foamed using a stand mixer (KENWOOD, kMix) until the volume was increased up to three times the initial volume (beating time about 5

min). Once the desired volume was obtained, the foaming speed was lowered to obtain a fine and even foam.

Next, DPG together with zinc oxide (ZnO) was added to the foam as the primary gelling agent, and even beating was continued for another 90 seconds. Immediately following that, sodium silicofluoride (SSF), the secondary gelling agent, was added and the foam was beaten for another 90 seconds. Finally, the un-gelled foam was quickly poured into an aluminum mold and allowed to gel for 3 min at ambient temperature. The gelled foam was then cured in a hot air oven at 100 °C for 2 h.

Once the foam was cured, it was stripped from the mold and washed thoroughly with deionized water to remove the potassium oleate soap and excess non-reacted elements. After washing, the cured NRLF was dried in a hot air oven at 80 °C for 8 h. The well-dried foam appeared off-white in color. The same procedure was used to produce the control sample of NRLF with zero RHP loading.

For the preparation of the NRLF sample with RHP loading, another step was added before the maturation process. After adding the primary (ZDEC) and secondary accelerators (ZMBT), the required parts per hundred (phr) of RHP were added to the compound. Next, the compound was allowed to mature for 8 h while being stirred at a low rpm (10 rpm). The following steps were the same as preparation of the control sample of NRLF with zero RHP loading. The phr of all of the chemicals in the preparation step were as mentioned in Table 1 (Ramasamy *et al.* 2012).

Measurement of Compression Properties

The compression behavior of RHP-incorporated NRLF samples were tested according to ASTM D3574. The samples used in this test method were in the regular shape of 50 by 50 by 25 mm, with parallel top and bottom surfaces and essentially perpendicular sides. Samples of RHP-incorporated NRLF with 0 phr (control), 2.5 phr, 5.0 phr, 7.5 phr, and 10 phr filler loading were analyzed. Three specimens per sample were tested. The specimens were placed in a test apparatus and deflated to 75±1% of their original thickness.

Within 15 min, the deflated specimens and the apparatus were placed in a mechanically convected air oven for 22 h with a test temperature of 70±2 °C. The specimens were immediately removed from the apparatus and measured after 30 min of recovery. The compression properties, such as constant deflection compression set and recovery percentage, were obtained from this testing.

Compression Test

A universal testing machine, Instron 3366, was used to carry out the compression test for the RHP-incorporated NRLF samples (according to ASTM D3574). The samples used in this test method were in the regular shape of 50 by 50 mm, with 25 mm thickness. The test was performed at room temperature, 23 °C. Samples of RHP-incorporated NRLF with 0 phr (control), 2.5 phr, 5.0 phr, 7.5 phr, and 10phr filler loading were analyzed. Three specimens of each sample were tested.

The specimens were compressed up to 50% of their original thickness at a compression speed of 0.83 ± 0.08 mm/s, and the final load was observed after 60±3 s. The initial and final thickness after the compression were measured. The compression deflection values were reported in pascals. The stress versus strain graph was obtained from compression testing.

Thermogravimetric Analyses (TGA)

Thermogravimetric analyses of the RHP-incorporated NRLF samples were performed using a Perkin Elmer Pyris 6 machine. Samples of the RHP-incorporated NRLF were tested at a heating rate of 20 °C/min from 30 °C to 600 °C under nitrogen gas. Samples of RHP-incorporated NRLF with 0 phr (control), 2.5 phr, 5.0 phr, 7.5 phr, and 10 phr filler loading were analyzed. The weight loss was calculated from the initial and final weight of the obtained thermograms.

Swelling Test

Swelling test (rubber-filler interaction) was analyzed benchmarking a method from previous research works (Muniandy *et al.* 2012). RHP incorporated NRLF samples with dimensions of 30 mm × 5 mm × 2mm were accurately weighed, immersed in toluene, and allowed to swell in a closed bottle for 72 h at room temperature (25 °C). The sample was then removed, and the surface of the swollen sample was quickly wiped and weighed. The sample was dried in an oven at 70 °C for 15 min and then weighed. Q , the weight of toluene uptake per gram of rubber hydrocarbon, was determined according to Eq. 1.

$$Q = \frac{\text{Swollen weight} - \text{Dried weight}}{\text{Original weight} \times \frac{100}{\text{Formula weight}}} \quad (1)$$

The rubber-filler interaction was calculated using the Lorenz and Park equation, Eq. 2:

$$\frac{Q_f}{Q_g} = ae^{-z} + b \quad (2)$$

The subscripts f and g in Eq. 2 refer to filled and gum vulcanizates, respectively. Z is the ratio by weight of the filler to the rubber in the vulcanizate, while a and b are constants.

RESULTS AND DISCUSSION

Compression Set Properties

There has been speculation that the macroscopic properties of foams, such as air flow, are directly related to foam compression set. However, it has since been recognized that the molecular structure is the relevant size scale for understanding the compression sets, which are simply symptomatic of the underlying polymer structure resulting from the polymerization reactions that occur during foaming (Sonnenschein *et al.* 2007). Researchers have empirically determined that formulation modifications can result in large differences in compression set values (Van der Schuur *et al.* 2004).

The compression set is a measure of the elastic behavior of the material. Figure 1 shows that the control NRLF (with 0 phr RHP) had the lowest compression set. Low compression sets indicate a high elasticity. NRLFs with higher RHP filler loadings had higher compression sets. Figure 2 reveals that the control NRLF (with 0 phr RHP) had the highest and fastest recovery percentage. The recovery percentage of RHP-incorporated NRLF decreased with increased RHP loading. Non-elastic deformation was mainly due to the deformation of the hard phase (Van der Schuur *et al.* 2004). This is probably due to the increased presence of RHP. Upon aging and compression force at 70 °C, RHP filler starts to decompose hence affecting the surrounding NRL matrix and contributing to poor adhesion properties. This non-elastic deformation due to aging of

reinforcing RHP filler leads to slower recovery and may explain the higher compression sets, observed after 30 min relaxation. Our work shows that RHP filler can substantially affect and increase the compression set at 70 °C.

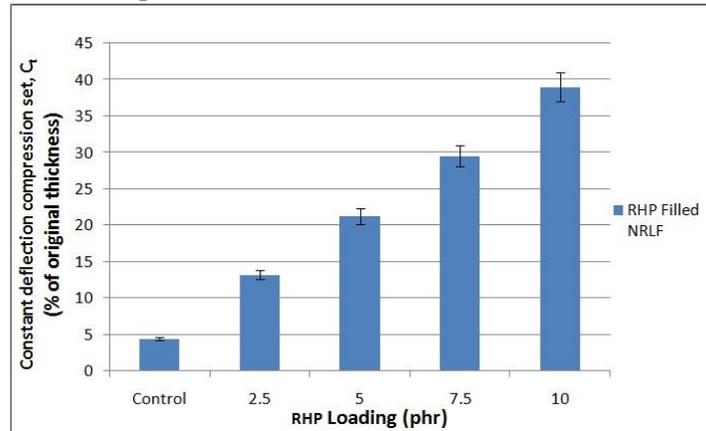


Fig. 1. Constant deflection compression set, C_t , of control and RHP-incorporated NRLFs

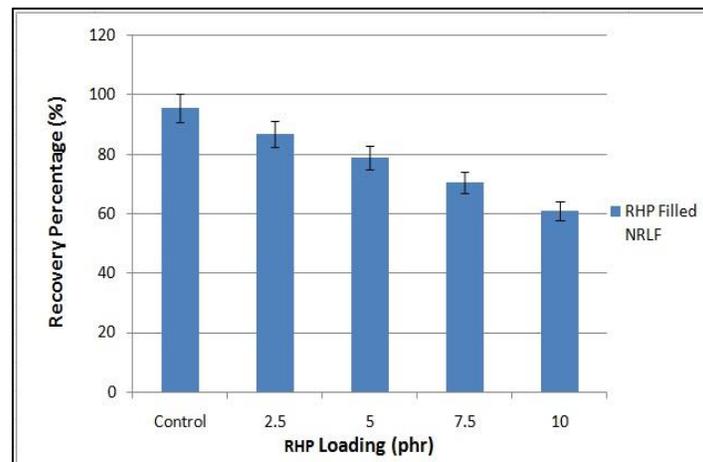


Fig. 2. Recovery percentage of control and RHP-incorporated NRLFs

Compression Test Properties

Figure 3 represents the effect of RHP filler loading on the stress-strain behavior of the RHP-incorporated NRLF. The stress-strain curves show that a higher amount of force was required to compress the RHP-incorporated NRLF with higher RHP loading. Figure 3 shows a clear trend of increasing stress with increasing RHP loading at 10, 20, 30, 40, and 50% strain. At 50% compression, the NRLF with 10 phr RHP loading showed the highest value of stress, while the control NRLF showed the lowest stress value. Addition of reinforcing RHP filler improves the interfacial RHP and NRL compound. RHP, being a harder material, indirectly increased the hardness of NRLF upon its addition. This effect can be attributed to the enhanced RHP-matrix interaction, which was further supported by the results of morphological studies (Ramasamy *et al.* 2012). Hence, it is clearly shown that RHP filler loading contributes to an increase in hardness but a reduction in the elasticity of RHP-incorporated NRLF.

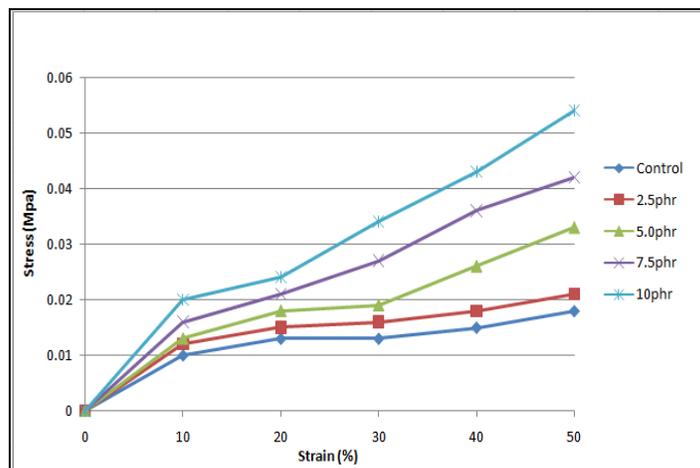


Fig. 3. Compressive stress-strain relationships of control and RHP-incorporated NRLFs

Thermogravimetric Analyses (TGA)

Thermal stability is the ability of a material to maintain the required properties, such as strength, toughness, or elasticity, when the temperature is changed. A detailed understanding of how polymers break down upon heating is important in the design of materials with improved properties for particular applications (Varkey *et al.* 2000).

The results of the thermogravimetric analysis (TGA) of control NRLF (with 0 phr RHP loading) and RHP-incorporated NRLFs with 2.5, 5, 7.5, and 10 phr RHP loadings are shown in Table 2, Figs. 4 and 5. The weight change of RHP-filled NRLFs with increasing temperature under a nitrogen environment was measured, and a graph of weight loss as a function of the temperature was plotted. A derivative thermogravimetric graph was also plotted. Table 2 shows the temperature at 5% weight loss, the maximum weight loss (%), the temperature at the maximum weight loss rate(%), and the final char residue (%). The temperature at 5% weight loss decreased with increasing RHP loading. Studies have reported that the decomposition of hemicelluloses in RHP occurred at low temperatures (Sharma and Rao 1999). Increasing the amount of RHP caused the decomposition of hemicelluloses in the RHP-incorporated NRLFs at lower temperatures. The presence of hemicelluloses in RHP apparently causes decomposition of RHP-filled NRLF at lower temperatures as compared to controlled NRLF.

As shown in Figs. 4 and 5, the control NRLFs and the RHP-incorporated NRLFs had weight loss corresponding to the volatilization of the degraded samples. The control NRLF and RHP-incorporated weight loss from 350 °C to 590 °C was related to the decomposition of the natural rubber and cellulose content in RHP. Lignin in RHP, which decomposed at a high temperature, also contributed to the weight loss of the RHP-incorporated NRLFs (Sharma and Rao 1999). Thermal stability of a polymer is enhanced by increasing the char residue, as the formation of char hinders the diffusion of the volatile decomposition products (Alexandre and Dubois 2000). Similarly, as shown in Table 2, the final char residue increased with increasing RHP filler loading, thus contributing to a higher thermal stability. The improvement of the thermal stability of the RHP-incorporated NRLFs can also be attributed to the good dispersion of RHP in the NRLF,

which led to a better filler-matrix interaction (Ramasamy *et al.* 2012). Therefore, it can be concluded that the thermal stability of the NRLFs was improved with increasing amounts of RHP.

Table 2. Thermal Stability Parameters of Control NRLF (0 phr RHP) and RHP-Filled NRLFs

Sample Code	Temperature at 5% weight loss	Maximum weight loss (%)	Temperature at Maximum weight loss (%)	Final char residue (%)
Control	328	95.799	582	4.201
2.5 phr	305	95.264	584	4.736
5.0 phr	302	95.159	585	4.841
7.5 phr	296	92.946	588	7.054
10 phr	295	92.675	589	7.325

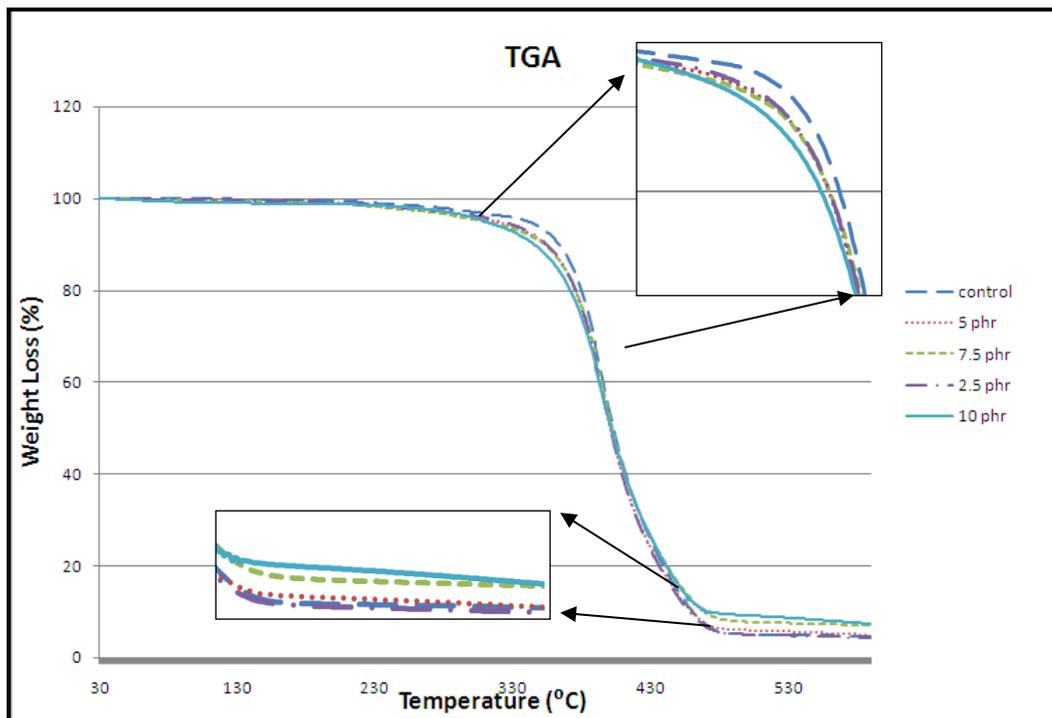


Fig. 4. Thermogravimetric analysis of RHP-incorporated NRLFs with 0 phr (control NRLF), 2.5 phr, 5.0 phr, 7.5 phr, and 10 phr

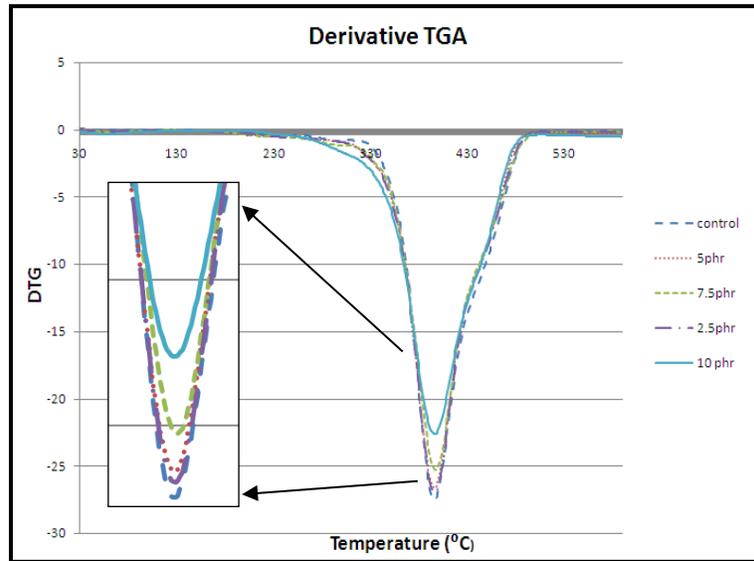


Fig. 5. DTG of RHP-incorporated NRLFs with 0 phr (control NRLF), 2.5 phr, 5.0 phr, 7.5 phr, and 10 phr

Swelling Test

Figure 6 displays the trend of rubber-filler interactions (Q_f/Q_g) of RHP incorporated-NRLFs. It is known that the lower the Q_f/Q_g value is, the higher the extent of interaction between the filler and matrix is (Ismail *et al.* 1999). From the results, it can be concluded that the addition of RHP caused a reduction in the Q_f/Q_g value, meaning that the value of Q_f/Q_g continuously decreased with increasing amounts of RHP filler. This explains why the RHP filler provided a good rubber-filler interaction in the RHP-incorporated NRLFs. A similar study with rattan powder as the filler in NR compounds has been reported (Muniandy *et al.* 2012). Increasing the amount of RHP filler contributed to the reduction of filler-filler interactions and the enhancement of filler-matrix interactions. This increased the possibility of crosslinks formation in the rubber chain, thereby causing better interactions between the RHP and NRL matrix. Results of this study were further supported by morphological evidence (Ramasamy *et al.* 2012).

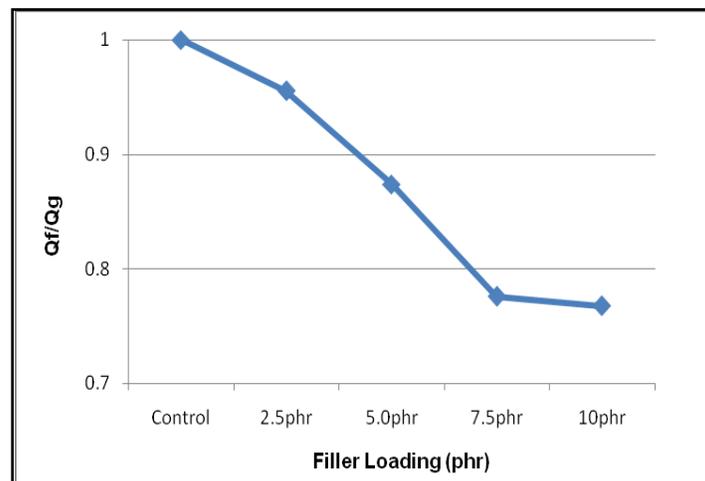


Fig. 6. The rubber-filler interaction of RHP-incorporated NRLFs

CONCLUSIONS

1. The compression set of rice husk powder (RHP)-incorporated natural rubber latex foams (NRLFs) increased with increasing RHP loading. The control NRLF (with 0 phr RHP) with high elasticity had the lowest compression set. The control NRLF had the highest recovery percentage, while the RHP-incorporated NRLFs showed decreasing recovery with increasing filler loading. The RHP filler reduced the elasticity of the NRLF, resulting in a slower recovery prior to the compression set.
2. The stress-strain curves showed an increasing stress value with increasing RHP loading, which demonstrates that filler loading contributes to an increase in hardness but a reduction in elasticity of the RHP-incorporated NRLF.
3. The thermal stability of NRLF was improved by incorporating RHP and was enhanced with increased RHP loading from 0 phr to 10 phr.
4. Increasing the amount of filler loading in the RHP-incorporated NRLF gave a lower Q_f/Q_g value and thus a higher extent of interaction between the filler and the matrix.
5. RHP filler acted as a reinforcing filler, making it suitable to produce low cost RHP-filled natural rubber latex foam (Ramasamy *et al.* 2012).

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