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# GROUND RICE HUSK AS FILLER IN RUBBER COMPOUNDING

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**Abstract.** As the greenhouse effects and global warming issues focus their stand in the fast-changing modern world, more efforts are being done towards manufacturing products, which are environment-friendly and recyclable. One of the favourite subjects is the utilization of agricultural waste into useful value-added product. Rice husk which is generated by the rice mills during rice processing composes mainly organo siliceous material. Together with its abundance supply and attractive price, rice husk has the potential to be used as filler in polymeric materials especially rubber. Most of the previous work done was on burnt rice husk ash as filler for rubber or composite. Consequently, this study will concentrate on utilizing ground rice husk, without burning it as filler in rubber compounding. The effectiveness of ground rice husk is determined by evaluating the rheological behaviour and physical properties of rice husk powder (RHP)-filled rubber vulcanisates. Rice husks were ground and sieved to obtain 300 μm and 180 μm of RHP. The RHP-filled rubber vulcanisates for both powder sizes were prepared using a laboratory two roll mill. Using a conventional vulcanization system, the cure behaviour was assessed by Monsanto Rheometer. Filler loading was varied from 0 to 50 pphr at 10 pphr interval. The vulcanisates were then tested. The dispersion of RHP in the vulcanisates was evaluated using scanning electron microscopy (SEM) analysis. For comparison purposes, commercial silica was used at 40 and 50 pphr whilst carbon black was used at 50 pphr. The results showed that the physical properties of RHP-filled vulcanisate are still inferior to carbon black-filled vulcanisate but some of the properties are comparable to silica-filled vulcanisate. The RHP also offers processing advantages over silica.

*Keyword*: Rice husk powder, rubber compounding

**Abstrak.** Seiring dengan kesedaran terhadap kesan rumah hijau dan isu pemanasan global, banyak usaha dilakukan ke arah penghasilan produk yang mesra alam dan boleh dikitar semula. Salah satu subjek yang digemari adalah penggunaan bahan buangan pertanian menjadi produk yang boleh digunapakai. Sekam padi yang dihasilkan daripada pengisar padi semasa proses penghasilan beras, mengandungi tahap silika yang tinggi. Sekam padi mempunyai potensi untuk dijadikan pengisi berdasarkan pada kelebihan daripada segi sumber yang tidak terhad dan kosnya yang murah. Kebanyakan kajian yang terdahulu melibatkan abu sekam padi sebagai pengisi dalam sebatian getah dan komposit. Bagaimanapun, kajian ini lebih menumpukan kepada serbuk sekam padi tanpa membakar sekam tersebut dan digunakan sebagai pengisi dalam sebatian getah. Keberkesanan serbuk sekam padi ini diuji pada sifat reologi dan sifat fizikal dan mekanik bagi sebatian getah. Sekam padi dihancurkan dan ditapis untuk mendapatkan serbuk sekam padi bersaiz 300 μm dan 180 μm. Komposit getah berisi serbuk sekam padi disediakan menggunakan mesin dua rola bersaiz makmal. Dengan menggunakan sistem pemvulkan konvensional, sifat reologi diuji dengan mesin Monsanto Rheometer. Tahap pengisian ditingkatkan daripada 0 kepada 50 pphr pada sela 10 pphr. Vulkanisat kemudiannya diuji. Penyebaran serbuk sekam padi dalam vulkanisat diuji melalui mikrograf SEM. Sebagai

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perbandingan, silika digunakan pada 40 dan 50 pphr manakala karbon hitam digunakan pada 50 pphr. Keputusan menunjukkan bahawa sifat fizikal dan mekanikal vulkanisat berisi serbuk sekam padi masih tidak dapat menandingi vulkanisat berisi karbon hitam. Bagaimanapun, terdapat beberapa sifat yang setanding dengan vulkanisat berisi silika. Serbuk sekam padi juga lebih mudah diadun dengan getah jika dibandingkan dengan silika.

*Kata kunci*: Serbuk sekam padi, sebatian getah

## 1.0 INTRODUCTION

Environmental issues such as the green house effects, climate change and global warming have always been favourite topics of discussion since decades. The increasing awareness of environmental concerns has lead to more and more efforts being done towards utilization of agricultural waste. For rice-producing countries like India, Thailand, China and Indonesia, tonnes and tonnes of rice husks are produced every year by rice mills as agricultural waste. In Malaysia, paddy is the third most widely planted crop after palm oil and rubber. On average, paddy production in Malaysia is about 2 million tonnes yearly [1]. In 2001, about 400 000 tonnes of rice husks are generated. Nowadays, the disposal of rice husk has become a problem especially when open burning is no longer permitted due to environmental concerns. This agricultural problem actually provides wide opportunities for us to utilise rice husk into useful value-added products.

Rice husk is the outer layer of rice grain which is removed during rice processing. It composes of mainly organo cellulose material. It is reported that rice husk contains 34–44% of cellulose, 23–30% of lignin, 13–39% of ash and 8–15% of moisture [2]. In rubber technology, one area of utilising rice husk is as filler in rubber compounding. Most of the previous works deal extensively on burnt rice husk or rice husk ash (RHA). Not many studies report on the use of ground unburnt rice husk in rubber. Early work by Haxo and Mehta [2] showed that ground rice husk ash prepared via special burning process is moderately reinforcing filler for rubber. They observed that this filler does not adversely affect the vulcanization characteristics or the aging of rubber compound and responds affectively to silane coupling agents in improving properties of the compound. In another study, Nasir and Low [3] demonstrated that different types of RHA can be derived via different incineration processes and conditions. The NR vulcanisates properties are still below the commercial silica and carbon black but the presence of coupling agents improves the performance of vulcanisates. An investigation by Ishak and Bakar [4] revealed that burnt rice husk ash yield two grades of fillers, namely white rice husk ash (WRHA) and black rice husk ash (BRHA). WRHA has been analysed to have about 96% silica content while the BRHA has a lower silica content typically about 54% and a substantially higher carbon content, i.e. about 44%. They observed that WRHA exhibits better physical properties than BRHA and not much inferior compared with commercial carbon black or silica-filled vulcanisates.

Consequently, this study was carried out to utilise rice husk directly without burning the husk. The effectiveness of ground unburnt rice husk in natural rubber compounding was evaluated. This paper discusses the study of the rheological behaviour and physical properties of rice husk powder (RHP)-filled vulcanisates. The Scanning Electron Micrograph (SEM) analyses are also done on the 300 µm-filled vulcanisate to see the dispersion of RHP in the rubber compound. Rice husk powder of 300 µm and 180 µm are used and filler level is varied from 0 to 50 parts per hundred rubber (pphr) at 10 pphr interval. For comparison purposes, commercial silica, Ultrasil VN3 is used at 40 and 50 pphr, whilst carbon black N330, of average size 26 to 30 nm is used at 50 pphr.

## 2.0 EXPERIMENTAL PROCEDURE

### 2.1 Rice Husk Powder Processing

Rice husk was obtained from local mills. Rice husk was ground using flour grinding machine. The size of the metal sieve inside the grinding machine is 0.4 mm. Thus, the ground rice husk obtained from the grinding machine has the size of about 400 µm. The ground rice husk was sieved to get the powder size of 300 µm and 180 µm.

### 2.2 Formulation and Compounding

The level of RHP of 300 µm and 180 µm in gum formulation were varied from 0 to 50 pphr at 10 pphr intervals. Formulations of the compounds were given in Table 1. For comparison purposes, RHP of 180 µm and both silica Ultrasil VN3 and carbon black N330 were used at 50 pphr (see Formulation 4, 5 & 6 in Table 2). Ground rice husk was also compared with RHP of 180 µm and silica, VN3 at 40 pphr (see Formulation 7, 8 & 9 in Table 2). The ground rice husk was used directly from the grinding machine without prior sieving process.

**Table 1** Formulation of mixes by varying RHP level

| Formulation (pphr) | 1 | 2 |
|---|---|---|
| SMR L | 100 | 100 |
| ZnO | 5 | 5 |
| Stearic Acid | 2 | 2 |
| Ralox LC | 2 | 2 |
| CBS | 0.6 | 0.6 |
| Sulphur | 2.5 | 2.5 |
| Rice husk powder 300 µm | Vary | – |
| Rice husk powder 180 µm | – | Vary |

**Table 2** Formulation of mixes

| **Formulation** | **4** | **5** | **6** | **7** | **8** | **9** |
|---|---|---|---|---|---|---|
| RHP 180 μm | 50 | – | – | – | – | 40 |
| Carbon Black, N330 | – | 50 | – | – | – | – |
| Silica, VN3 | – | – | 50 | 40 | – | – |
| Ground Rice Husk | – | – | – | – | 40 | – |

Base stock: SMR L 100, Zn0 5, St. Acid 2, Ralox LC 2, CBS 0.6, Sulphur 2.5, DEG 2*
* Only for formulation 6 & 7

Mixing of the compounds was carried out on the 12" × 6" two-roll mill. The temperature was maintained at 70°C. Mixing of gum mix was done in 10 minutes whilst mixing of filled rubber varied from 12 to 25 minutes depending on the level of RHP incorporated.

## 2.3 Testing Methods

The single speed rotational Mooney viscometer was used to measure the Mooney viscosity and Mooney scorch of the compound at 100°C. The cure characteristics such as the optimum cure time ($t_{90}$), scorch time ($t_{s2}$), minimum (ML) and maximum torques (MH) were accessed using Monsanto Rheometer, MDR 2000. The compound was cured at 160°C. The following physical testing were carried out, in accordance to ISO test procedure;

| **Test** | **Method** |
|---|---|
| • Tensile strength | ISO 37 : 1994 |
| • Hardness | ISO 48 : 1994 |
| • Compression set | ISO 815 : 1991 |
| • Rebound Resilience | ISO 4662 : 1986 |
| • ISO Abrasion | ISO 4649 : 1985 |
| • Trouser Tear | ISO 34 : 1994 |

The SEM test was carried out by cutting the vulcanisate samples at the cross-section and attached to the specimen stubs with double-sided tape. Specimens were prepared for examination by evaporative coating with an ultra-thin layer of gold under high vacuum. This layer acted as conducting layer to permit SEM examination.

# 3.0 RESULTS AND DISCUSSION

## 3.1 Rheological Study

The Mooney properties and cure characteristics for 300 μm and 180 μm RHP natural

**Table 3** The cure characteristics and Mooney properties of 300 μm RHP natural rubber compound

| Level of 300 μm RHP (pphr) | 0 | 10 | 20 | 30 | 40 | 50 |
|---|---|---|---|---|---|---|
| Rheo Min (ML), lb-in | 5 | 3 | 3 | 4 | 2 | 2 |
| Rheo Max (MH), lb-in | 22 | 24 | 26 | 29 | 30 | 32 |
| Rheo Scorch ($t_{s2}$), min | 5 | 4 | 4 | 4 | 4 | 3 |
| Rheo Cure ($t_{90}$), min | 8 | 8 | 8 | 8 | 7 | 7 |
| Mooney Scorch ($t_5$), min | 48 | 50 | 51 | 50 | 47 | 41 |
| Mooney Viscosity, $M_V$ | 53 | 34 | 34 | 44 | 29 | 29 |

rubber compounds at 160°C are presented in Tables 3 and 4 respectively. For both types of powder, the maximum torque increases with increasing level of RHP. It is observed that RHP level does not have any adverse effect on the scorch time and cure time of the compound.

However, the Mooney scorch for compound with 180 μm RHP has higher value compared with compound with 300 μm RHP since the mixing was done on different days. Compound with 180 μm RHP also gives higher Mooney viscosity because of its finer particle size.

**Table 4** The cure characteristics and Mooney properties of 180 μm RHP natural rubber compound

| Level of 180 μm RHP (pphr) | 0 | 10 | 20 | 30 | 40 | 50 |
|---|---|---|---|---|---|---|
| Rheo Min (ML), lb-in | 5 | 5 | 5 | 5 | 5 | 5 |
| Rheo Max (MH), lb-in | 22 | 25 | 27 | 30 | 32 | 33 |
| Rheo Scorch ($t_{s2}$), min | 6 | 6 | 5 | 4 | 4 | 3 |
| Rheo Cure ($t_{90}$), min | 10 | 9 | 9 | 8 | 8 | 7 |
| Mooney Scorch ($t_5$), min | 62 | 63 | 53 | 43 | 36 | 31 |
| Mooney Viscosity, $M_V$ | 52 | 56 | 52 | 59 | 55 | 52 |

Comparative rheological properties of 180 μm RHP-filled natural rubber compound with silica-filled and carbon black-filled natural rubber compounds at 50 pphr are summarised in Table 5. The maximum torque of compound filled with RHP is about 25 percent lower than with silica or carbon black. It can be seen that RHP-filled compound gives relatively much shorter cure time and scorch time compared with compound filled with silica. Comparing with carbon black, the RHP-filled compound has similar cure time and scorch time. In addition, the Mooney viscosity of the compound filled RHP is obviously lower than silica and carbon black-filled compounds. This indicates the easier processing for RHP. This is probably due to the different nature and chemical composition between RHP and silica. The surface structure of silica is embedded by cellulose and this gives an effect to the cure rate of the compound.

The cure characteristics and Mooney viscosity of 180 μm RHP-filled compound was also compared with ground RH- and silica-filled compounds at 40 pphr. The results are presented in Table 6. There is not much difference for the maximum torque, optimum cure time and Mooney viscosity for 180 μm RHP-filled and ground RH-filled compounds. At 40 pphr, both compounds having ground RH and 180 μm RHP give much shorter cure time, lower maximum torque and lower Mooney viscosity than silica-filled compound.

**Table 5** The cure characteristics and Mooney properties of 180 μm RHP, silica and carbon black-filled natural rubber compounds at 50 pphr

| Filler | RHP (180 μm) | Silica, VN3 | CB N330 |
|---|---|---|---|
| Rheo Min (ML), lb-in | 3 | 15 | 7 |
| Rheo Max (MH), lb-in | 32 | 43 | 45 |
| Rheo Scorch ($t_{s2}$), min | 2 | 4 | 4 |
| Rheo Cure ($t_{90}$), min | 7 | 19 | 7 |
| Mooney Scorch ($t_5$), min | 22 | 39 | 19 |
| Mooney Viscosity, $M_V$ | 35 | 107 | 68 |

**Table 6** The cure characteristics and Mooney viscosities of ground RH, 180 μm RHP, silica-filled compounds at 40 pphr

| Filler | Ground RHP | RHP (180 μm) | Silica, VN3 |
|---|---|---|---|
| Rheo Min (ML), lb-in | 1 | 1 | 3 |
| Rheo Max (MH), lb-in | 9 | 10 | 17 |
| Rheo Scorch ($t_{s2}$), min | 2 | 2 | 1 |
| Rheo Cure ($t_{90}$), min | 7 | 7 | 15 |
| Mooney Scorch ($t_5$), min | 37 | 38 | 25 |
| Mooney Viscosity, $M_V$ | 22 | 28 | 72 |

## 3.2 Scanning Electron Micrograph (SEM) Analysis

The Scanning Electron Micrograph (SEM) analysis for rice husk powder and silica are shown in Figures 1 and 2. It can be seen that silica has more uniform round structure compared with rice husk powder that has uneven structure. The SEM analyses for vulcanisates filled with 10 to 50 pphr of 300 μm RHP are shown in Figures 3 to 7. It is observed that at lower level of RHP (10 pphr), the surface is rather smooth due to better dispersion of RHP in rubber whilst at high level of RHP, the vulcanisates have a rough surface and many agglomerates.

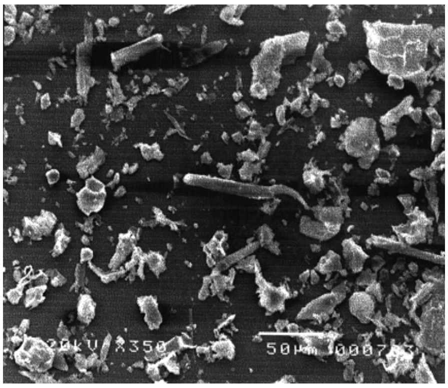

**Figure 1** SEM of Rice husk

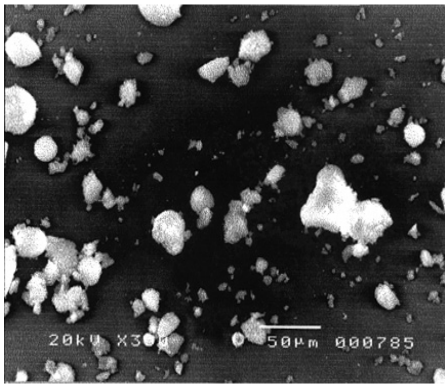


**Figure 2** SEM of Silica

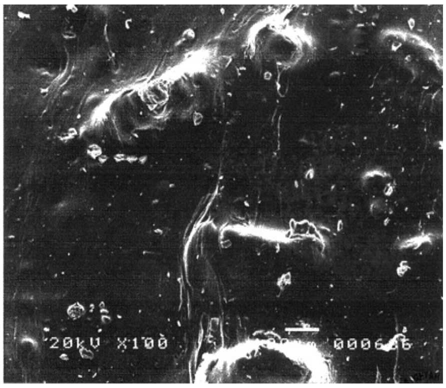


**Figure 3** SEM of 10 pphr RHP

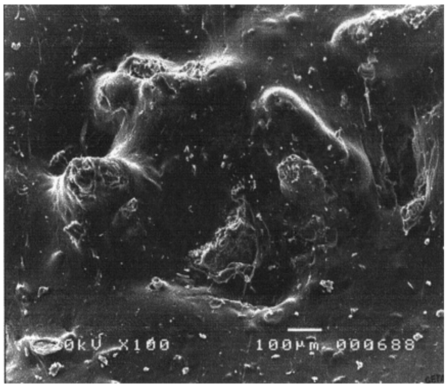


**Figure 4** SEM of 20 pphr RHP


**Figure 5** SEM of 30 pphr RHP

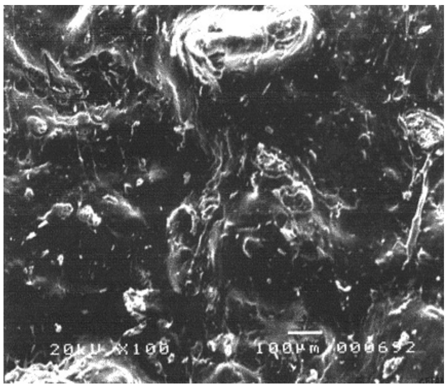

**Figure 6** SEM of 40 pphr RHP

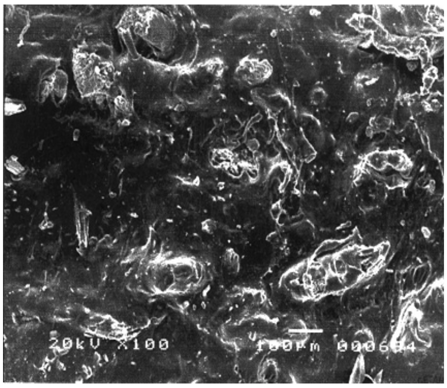


**Figure 7** SEM of 50 pphr RHP

## 3.3 Physical Properties

The effect of RHP level on tensile strength of vulcanisates is shown in Figure 8. For both 300 µm and 180 µm RHP, the tensile strength of vulcanisates have the optimum value at 10 pphr but further increments of RHP level cause a reduction in the tensile strength of vulcanisates. This corresponds well to the results of SEM analysis that after 10 pphr, there is a poor dispersion of RHP in the rubber resulted in agglomerations of RHP to occur on the surface of the vulcanisates. These agglomerates might cause the tensile strength to reduce and poor filler to rubber interaction. Ismail and Chung [5] reported the similar observation for RHA in natural rubber compounding, where the

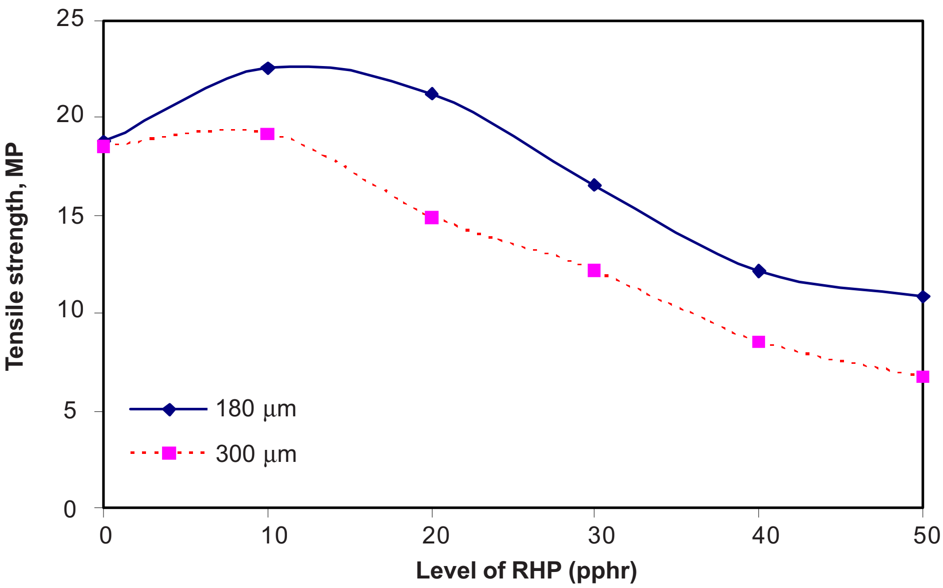


**Figure 8** The effect of RHP level on tensile strength

addition of RHA increases the tensile strength up to an optimum value (i.e. 10 pphr of filler loading) but after this level, the property starts to decrease due to the weaker rubber-filler interaction. It is also noted in Figure 8 that 180 µm RHP-filled vulcanisate exhibits better tensile strength compared with 300 µm RHP-filled vulcanisate. In other words, finer particle size would result in better filler dispersion and subsequently tensile strength.

For elongation at break, the same trend is observed for both types of powder. In Figure 9, elongation at break increases up to 10 pphr and then it tends to decrease as the level of RHP increases. This may be due to poor dispersion of RHP at higher level. The results obtained also show that 180 µm RHP-filled vulcanisates give higher elongation at break than 300 µm RHP-filled vulcanisate.

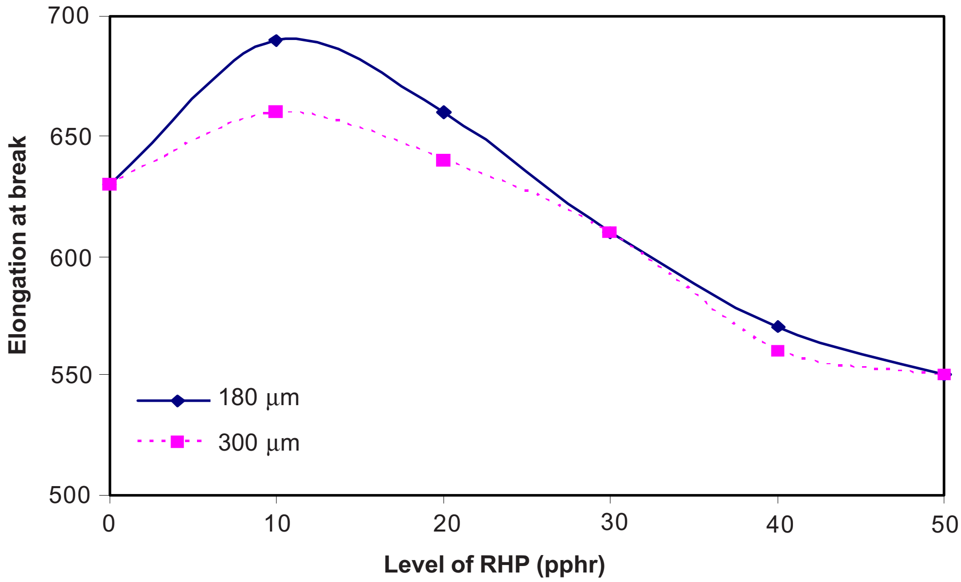


**Figure 9** The effect of RHP level on elongation at break

The trouser tear strength results are presented in Figure 10. The tear strength of 300 µm RHP filled vulcanisate is enhanced up to 30 pphr. No further improvement is observed after that level and tear strength is maintained at 9 N/mm. On the other hand, the tear strength of the 180 µm RHP-filled vulcanisate tends to decrease and plateau after a loading of 20 pphr of RHP. Unlike the tensile strength, the tear strength for RHP-filled vulcanisate shows different behaviour as 300 µm RHP gives higher strength compared with 180 µm RHP. This is mainly attributed to not only the particle size, but also the dispersion and orientation of the RHP in the rubber matrix. In addition, the property is controlled by the rate and method of tearing.

Jacques [10] reported that the incorporation of fillers increase the hardness and reduce the resilience of vulcanisates, particularly with the more reinforcing types of filler, and

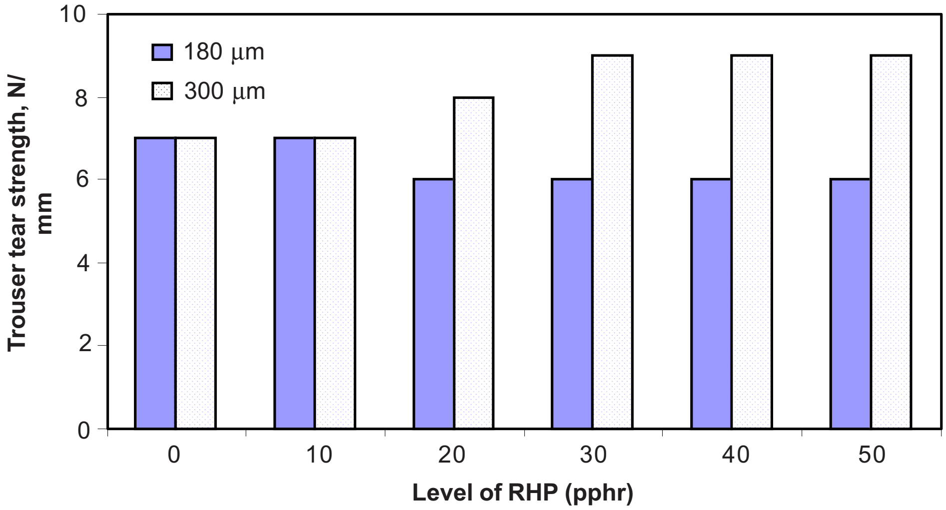


**Figure 10** The effect of RHP level on tear strength

the effect being more marked at a given filler level as the particle size is reduced. As expected in Figure 11, the hardness of vulcanisate increases gradually with increasing level of RHP. There is no noticeable difference in the hardness level between 180 µm and 300 µm RHP-filled vulcanisates. This suggests that RHP has less significant effects on hardness property due to the non-reinforcing nature of RHP.

The effect of RHP level on resilience of vulcanisate is shown in Figure 12. For both types of powder, resilience slowly deteriorates with increasing level of RHP. Resilience

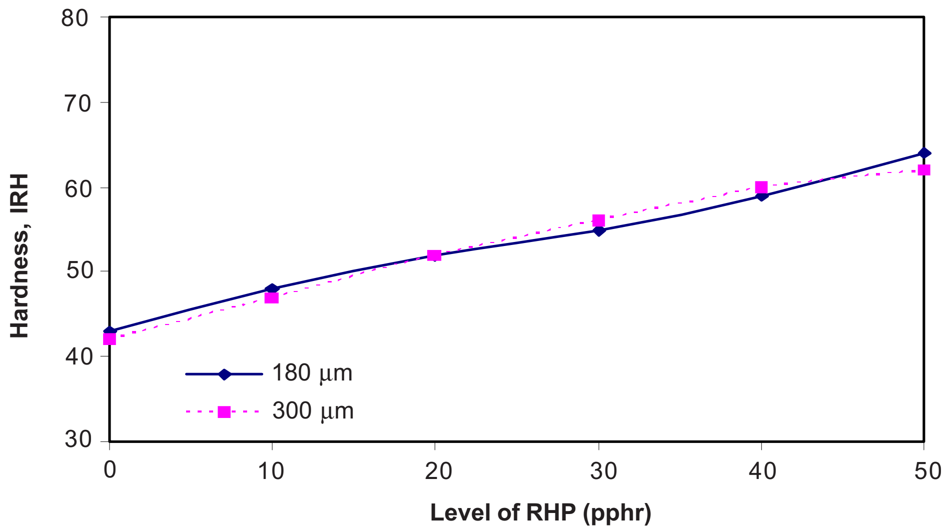


**Figure 11** The effect of RHP level on hardness

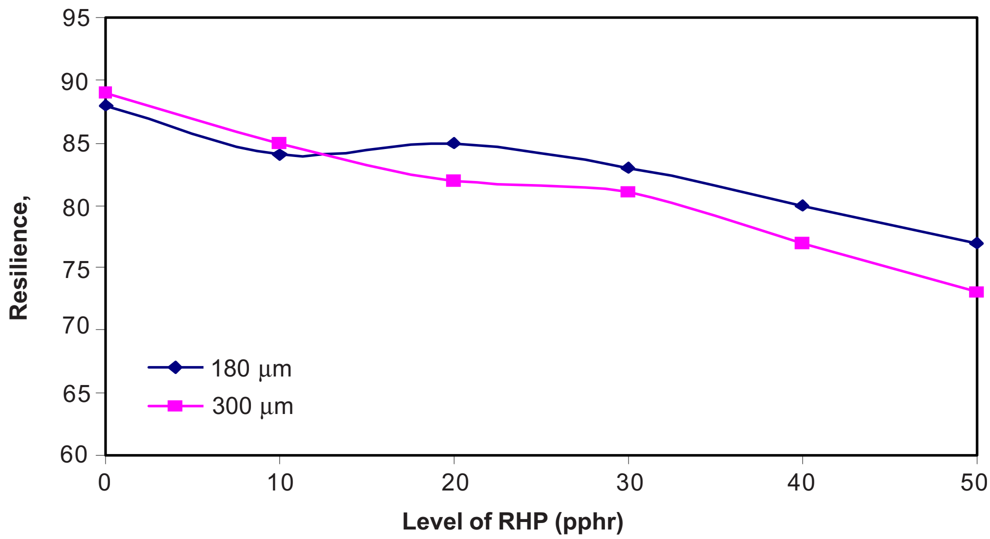


**Figure 12** The effect of RHP level on resilience

of 180 µm RHP-filled vulcanisate is slightly higher than 300 µm RHP-filled vulcanisate. This is also expected as mentioned earlier [10]. Resilience is a measure of elasticity of vulcanisate. The incorporation of RHP resulted in lowering the elasticity of vulcanisate.

According to Jacques [10], the resistance of filled vulcanisate to abrasion increases progressively with smaller particle size of filler. As shown in Figure 13, the 180 µm RHP give better abrasion resistance than 300 µm RHP. Increasing the RHP level, decreases the abrasion resistance of the vulcanisate. Noticeably, 300 µm RHP-filled

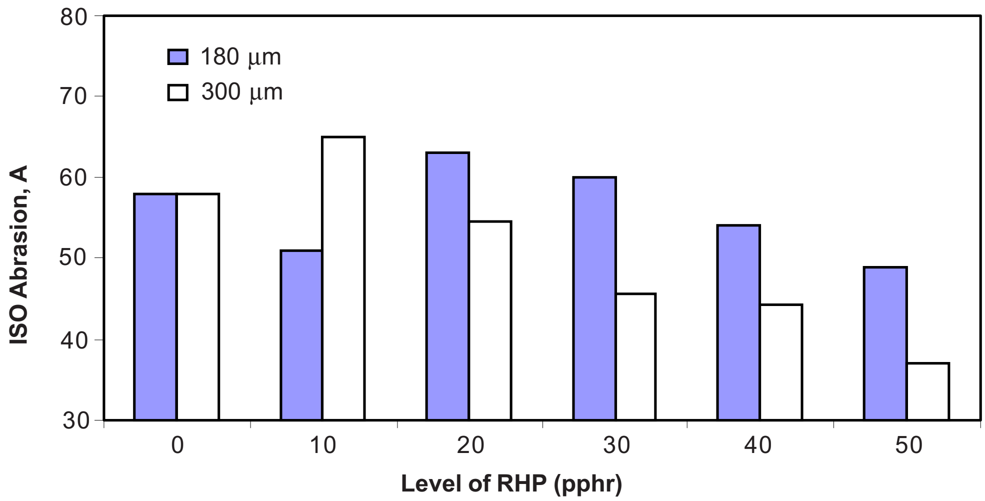


**Figure 13** The effect of RHP level on abrasion of vulcanisates

vulcanisate has the optimum abrasion at 10 pphr whilst 180 μm RHP-filled vulcanisate has the optimum resistance at 20 pphr. Decreasing the particle size of filler indicates a larger surface area and hence, greater interface between the rubber matrix and the filler which resulted in higher abrasion resistance. Hence, it is envisaged that abrasion resistance can be further optimised by using finer filler size.

Comparison of the physical and mechanical properties of RHP-filled vulcanisate with silica-filled and carbon black-filled vulcanisate are presented in Table 7. At 50 pphr, vulcanisate having RHP of 180 μm gives better resilience and compression set values compared with silica. This may be attributed to the different structure of RHP and silica as well as the surface activity. Filler structure is always associated with the modulus and stiffness of the vulcanisate. RHP-filled vulcanisate is less stiffer than silica-filled vulcanisate, and consequently more elastic and give higher rebounce resilience. On the other hand, the elongation at break as well as the modulus at 100 and 300% are also higher than silica. However, the tensile strength, hardness and abrasion value are still inferior to silica. When compared with carbon black, 180 μm RHP-filled vulcanisate gives superior resilience and comparable compression set values. The other properties are still inferior to carbon black.

**Table 7** The physical and mechanical properties of 180 μm RHP, silica and carbon black-filled vulcanisates at 50 pphr

| **Properties** | **RHP (180 μm)** | **Silica, VN3** | **CB N330** |
|---|---|---|---|
| Tensile strength, (MPa) | 10.1 | 12.9 | 24.8 |
| Elongation at break, (%) | 650 | 560 | 460 |
| Modulus at 100%, (MPa) | 2.1 | 1.2 | 4.2 |
| Modulus at 300%, (MPa) | 3.7 | 2.5 | 16.3 |
| Resilience, (%) at 23°C | 74 | 55 | 56 |
| Hardness, IRHD | 64 | 79 | 76 |
| ISO Abrasion, ARI (%) | 35 | 45 | 104 |
| Akron Abrasion, ARI % | 5 | 16 | 96 |
| Comp, set at 24hr, 70°C (%) | 47 | 80 | 47 |
| Trouser Tear strength, N/mm | 6 | 7 | 11 |

The physical and mechanical properties for ground RH, RHP of 180 μm and silica at 40 pphr are summarised in Table 8. It can be seen that the tensile strength for ground RH-filled vulcanisate is slightly lower than 180 μm RHP-filled vulcanisate, but other properties are quite comparable. Even though the ground RH has a larger particle size than 180 μm RHP, its resilience and compression set value are better than silica-filled vulcanisate. Again, this is due to the nature and structure of RHP as mentioned earlier.

**Table 8** The physical and mechanical properties of ground RH, 180 μm RHP and silica-filled vulcanisates at 40 pphr

| Properties | Ground RH | RHP (180 μm) | Silica, VN3 |
|---|---|---|---|
| Tensile strength, (MPa) | 8.5 | 12.5 | 14.1 |
| Elongation at break, (%) | 600 | 620 | 670 |
| Modulus at 100%, (MPa) | 1.5 | 1.6 | 1.1 |
| Modulus at 300%, (MPa) | 2.2 | 3.0 | 2.3 |
| Resilience (%) | 77 | 72 | 59 |
| Hardness, IRHD | 55 | 58 | 62 |
| ISO Abrasion, ARI (%) | 57 | 56 | 60 |
| Akron Abrasion, ARI % | 5 | 16 | 96 |
| Comp, set at 24hr, 70°C (%) | 57 | 60 | 85 |
| Tear strength, N/mm | 9 | 6 | 8 |

## 4.0 CONCLUSIONS

The level of RHP has some effects on the rheological behaviour of rubber compound especially on the maximum and minimum torque, but the scorch and cure time remain unchanged. RHP 180 μm-filled vulcanisates give better tensile strength, elongation at break and abrasion compared with RHP 300 μm filled vulcanisates. This is expected because finer and smaller particle size provides better dispersion in the rubber compound, and hence improvements in these properties. However, other properties, such as hardness and resilience, the differences between the two types of powder are not that significant. The same observation can be seen when comparing ground RH with RHP of 180 μm. The vulcanisate filled with ground RH gives comparable resilience, hardness, compression set and abrasion despite the lower tensile strength and elongation at break compared to RHP of 180 μm - filled vulcanisate. Interestingly, RHP-filled vulcanisate gives superior resilience than both silica and carbon black. In addition, a good compression set is also determined for RH-filled vulcanisate, where the value is comparable to carbon black-filled vulcanisate and better than silica-filled vulcanisate. Overall, RHP is a satisfactory filler for natural rubber and its use could resolve the agricultural problems and environmental issues effectively.

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