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MECHANICAL PERFORMANCE OF COMPOSITES MATERIALS PREPARED FROM WASTE PLASTICS AND TUBED TYRE

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Abstract: The management and safe disposal of used tyres have always been a considerable challenge from the economic and processing standpoints. Hence, this study prepared composites by combining recycled high-density-polyethylene (rHDPE) with ground butyl-rubber (bR); which is a material commonly used in the production of tubed-tyres. The bR-rHDPE composites of different bR content with or without the addition of bitumen were produced via melt-mixing in an internal mixer. The impact resistance and mechanical properties of each composite were evaluated using (i) Charpy impact, (ii) drop-weight and (iii) tensile testings. Impact tests show a significant improvement in impact resistance. However, the addition of bR and bitumen adversely affected the tensile properties of the composites. The results indicate that the bR-rHDPE composites performed better with a higher content of bR, and the impact resistance further improved with the addition of bitumen. The obtained property suggests that this composite could be used to manufacture playground tiles, vehicles wheel chocks, door stops and wedges, rubber speed bumpers, etc. The promising impact resistance of bR-rHDPE+bitumen composites coupled with viable processing methods have provided a sustainable solution for the disposal of tubed tyre.

Keywords: Rubber tyre waste, impact behaviour, mechanical properties, drop-weight impact, interfacial adhesion.

Introduction

High-density polyethylene (HDPE) is a commercial grade polyethylene. This commodity is in high demand to produce hard plastic containers for many everyday consumer products such as milk, motor oil, shampoo, conditioner, liquid soap, detergent, bleach and pipes (Spalding & Chattejee, 2017). The prolific use of HDPE has led to an increase in solid municipal wastes. Therefore, reusing or recycling the waste of HDPE or plastic materials into new products (Singh *et al*., 2020; Keskisaari & Kärki, 2018; Seghiri *et al*., 2017) can reduce the negative impact of waste plastic on the environment. Butyl rubber (bR) is a robust synthetic rubber that has excellent tolerance to water, heat, vibrations, ageing, weather and flexing. Therefore, it is an essential material for the production of various applications, such as pharmaceutical stoppers, waterproof and insulating sealants, speakers and audio equipment, sporting equipment, fuel additives and vibration control in the medical, construction and automobile industries. It is also an elastomer that offers outstanding air and gas retention (Philip, 2004); a quality that has yet to be discovered in other types of rubber. It is also ideal to be used as the inner tube of tyres due to its unparalleled vibration dampening characteristics.

In Malaysia, and most ASEAN countries, motorcycle is the preferred mode of transportation for middle-income families in the city or rural areas due to its affordability, and low maintenance cost petrol consumption and road tax. From the manufacturing standpoint, the low production cost of bR makes it ideal for the production of tubed tyres for smaller cubic centimetre (cc) motorcycles. A study published in May 2019 by the Department of

Statistics Malaysia reported an 11.6% increase in motorcycle purchases, which brings the current number of motorcycles on Malaysian roads to 13.7 million (https://www.dosm.gov.my). This number is added to the abundance of used tyres and their inner tubes, which have no means of safe disposal. This is particularly true for the inner tubes because the tubes are classified under thermosetting polymeric materials. Therefore, the improper disposal and recovery of tubed tyres can adversely affect the environment. One of the few environmental friendly disposal methods is incorporating the particles of used tyre rubber, either ground or granulated, into a thermoset (Hejna *et al*., 2020; Aoudia *et al*., 2017) and a thermoplastic matrix (Zhang *et al*., 2009; Montagna & Santana, 2012; Kakroodi & Rodrigue, 2013) to form new polymer composites. The composites can be created using common processing techniques such as extrusion (da Costa *et al*., 2006) or injection moulding (Zainal & Ismail, 2011). The final fabricated composites usually have good properties (Ratnam *et al*., 2013) and are suitable for various applications such as acoustic insulators (Aliabdo *et al*., 2015).

The impact strength of recycled tyres/thermoplastic systems can be assessed using traditional impact testing techniques, such as Charpy impact test or Izod impact test (Kim *et al.,* 2000; da Costa *et al*., 2006). However, little has been done to investigate the impact resistance and corresponding mechanisms of damage via drop-weight impact testing. Compared to Charpy impact testing, drop-weight impact testing can mimic the damage under real-life stress conditions (Navaranjan & Neitzert, 2017). This information might be useful for the applications of industrial flooring and shock absorption.

This study aims to investigate the impact resistance and mechanical properties of rHDPE that were combined with waste bR from the inner tube of tyres and bitumen. The composite samples rHDPE, 50bR-rHDPE, 60bR-rHDPE, 70bR-rHDPE, 50bR-rHDPE+bitumen, 60bR-rHDPE+bitumen and 70bR-rHDPE+bitumen, were prepared. The responses of the composites were determined using Charpy impact testing, drop weight impact testing, and tensile testing. The results obtained from the impact and tensile tests were discussed in correlation with morphological observations from a scanning electron microscope (SEM). This study also discusses the influence of bitumen in improving interfacial adhesion between the bR granules and rHDPE matrix.

Materials and Methods

Different mixtures were prepared using recycled high-density polyethylene (rHDPE), waste butyl rubber (bR) and bitumen. The used inner tubes of motorcycle tyres (which are made of bR) were collected from a motorcycle workshop at the International Islamic University Malaysia (IIUM). The waste bR was washed before crushed into irregular-shaped granules of 3.0 - 5mm (Figure 1) using a crusher machine. rHDPE pellets with 0.950g/cm^3 density were purchased from DCT Plastics Sdn. Bhd. and 80/100-grade bitumen was obtained from Swee Quary Sdn. Bhd.

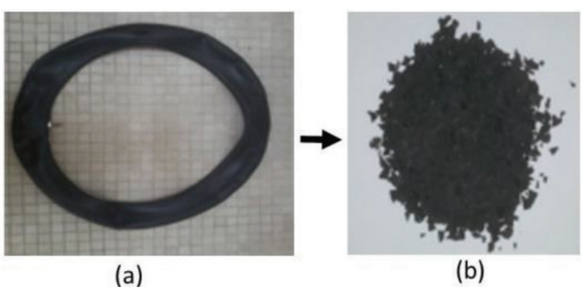

Figure 1: (a) Used inner tubes (b) Irregular-shaped bR granules

Sample Preparation

The compositions of rHDPE, bR and bitumen are shown in Table 1. All materials were compounded via melt mixing in the HAAKE Rheomix 600 Internal Mixer. The temperature and rotor speed were set at 170ºC and 50 rpm, respectively. Then, compounded composites were crushed into smaller sized pellets before compression moulded and hot-pressed into ASTM D638 and ASTM D790 standard specimens using the Xihua XH-406B Tablet Press Machine. The temperature was set at 185^{O}C and five minutes to preheat, and pressed for eight minutes before cooling for five minutes.

Mechanical Characterisation

For the impact and tensile strength tests, all seven samples were tested and the average values were recorded. The Charpy impact test was performed using the Dynisco Polymer Test, Simatic OP7 machine in accordance with the ASTM D6110 standards (applied energy 7.5 J, load 4 kN).

For the drop-weight impact test, the composites (120 x 12 x 3 mm) were impacted using a 60 mm (L) x 12.24 mm (D) hemispherical impactor. All experiments were carried out at room temperature. The specimens were secured to the sample holder using double-sided tape. The impact testers were set to a sensitivity of -4.019, ontrigger mode with a pre- and post-time of 1,000 s. The Dewesoft: Data Acquisition Systems (DAQ) was used to capture the load-time data.

The tensile test was conducted according to the ASTM D368 standards, using the Shimadzu Autograph AGS-X universal testing machine with a crosshead speed of 50 mm/min.

Results and Discussion

Charpy Impact Properties

Figure 2 shows the variations of impact strength for bR-rHDPE composites with 50, 60 and 70 wt% bR loadings. The impact strength of bR-rHDPE composites increased substantially with the incorporation of bR. There was a linear correlation between bR-rHDPE impact strength and bR loading up to 60 wt%-bR; the improvements were more than 130% compared to the neat rHDPE sample. However, a slight decline in impact strength was observed in the loadings above 60 wt%-bR. Nevertheless, it remained well above the performance of the neat rHDPE. As seen in Figure 2, the introduction of bR phase has greatly improved the impact strength of bR-rHDPE composites. This phenomenon occurred due to the damping properties of bR. It is noted that bR has higher damping capability than plastics, whereby this feature promotes greater energy absorption (Geethamma *et al*., 2014). Khodadadi *et al*. (2019) compared to the ballistic performance between Kevlar/rubber and Kevlar/epoxy composites, and found that the energy absorption was superior in the Kevlar/rubber composite. They agreed that this result was contributed by the high damping property of rubber.

Table 1: Designation and compositions of composites

Sample	rHDPE (wt%)	Butyl Rubber (wt%)	Bitumen (wt%)
50bR-HDPE	50	50	-
50bR-HDPE+bitumen	50	50	30
60bR-HDPE	40	60	-
60bR-HDPE+bitumen	40	60	30
70bR-HDPE	30	70	-
70bR-HDPE+bitumen	30	70	30

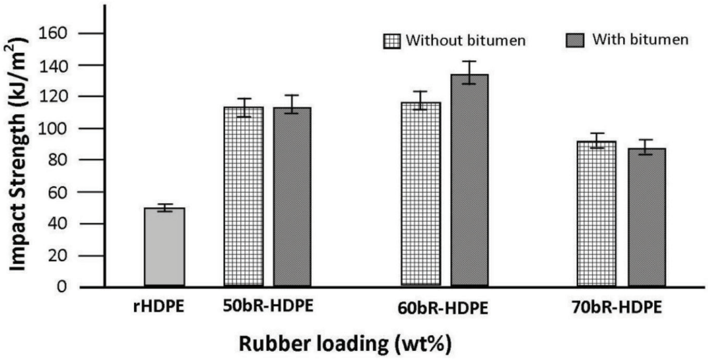

Figure 2: The impact strength of rHDPE vs bR-rHDPE composites of varying rubber loadings

The SEM micrograph observations also reveal good adhesion between bR and rHDPE can be obtained to a certain extent. Lievana and Karger-Kocsisl (2004), and Tao *et al*. (2013) reported that the mechanical stress of the extrusion process breaks the sulphur crosslinks in the rubber, which is known as devulcanisation. Fewer crosslinks allow stronger molecular entanglement with the polymer matrix and improved interfacial adhesion that enhance the stress transferring mechanism. The current study used an internal mixer which is believed to impart mechanical stresses comparable to the extrusion process.

The impact strength of bR-rHDPE composites was further increased by the addition of bitumen, primarily in sample 60bR-rHDPE. The presence of bitumen has benefitted the impact strength of the composites. These results are consistent with previous works which used compatibilizer in the rubber/thermoplastic composites (Kim *et al*., 2000; Sonnier *et al*., 2008). According to Sengoz and Isikyakar (2008), bitumen is viscoelastic at ambient temperature, but it transforms into a highly fluid state when heated. This transformation enhances the softening of composites (Grigoryeva *et al*., 2005). Previous studies also suggested that bitumen could act as a devulcanising agent (Lievana & Karger-Kocsis, 2004) and provide plasticising effects (Zhang *et al*., 2009). All these characteristics assisted in improving the adhesion of bR to rHDPE phase in the composites. Apart from inherent damping properties of bR, the increased impact strength of bR-rHDPE+bitumen composites was due to more efficient stress transfer between rHDPE and bR. This situation caused higher energy absorption under impact loading.

Drop-weight Impact Properties

In addition to the conventional impact testing methods; Charpy and Izod (Anderson, 2017; Lampman, 2003); drop-weight impact testing is important in determining the impact resistance of a material. The results for the drop-weight impact test of bR-rHDPE composite samples are presented in Figure 3. Most of the samples exhibit a nonsymmetrical shape in the load-time history. In general, the collision of the impactor with the sample has resulted in a marginal load increase, before increasing linearly and reaching the maximum limit. Then the load is decreased. Besides that, there was a common feature of force-time history in drop-weight impact tests such as oscillation lines (Sevkat *et al*., 2009; Balaganesan *et al*., 2017).

Close examination of the impacted samples found that damage occurred via deflections at varying angles as shown in Figure 4. The peak-load of all samples is summarised in Table 2. The tests show that the bR-rHDPE composites (without bitumen) have higher peak-load values than the bR-rHDPE+bitumen samples. This result could be attributed to the stiffness (Isa *et al.,* 2014) commonly found in HDPE due to

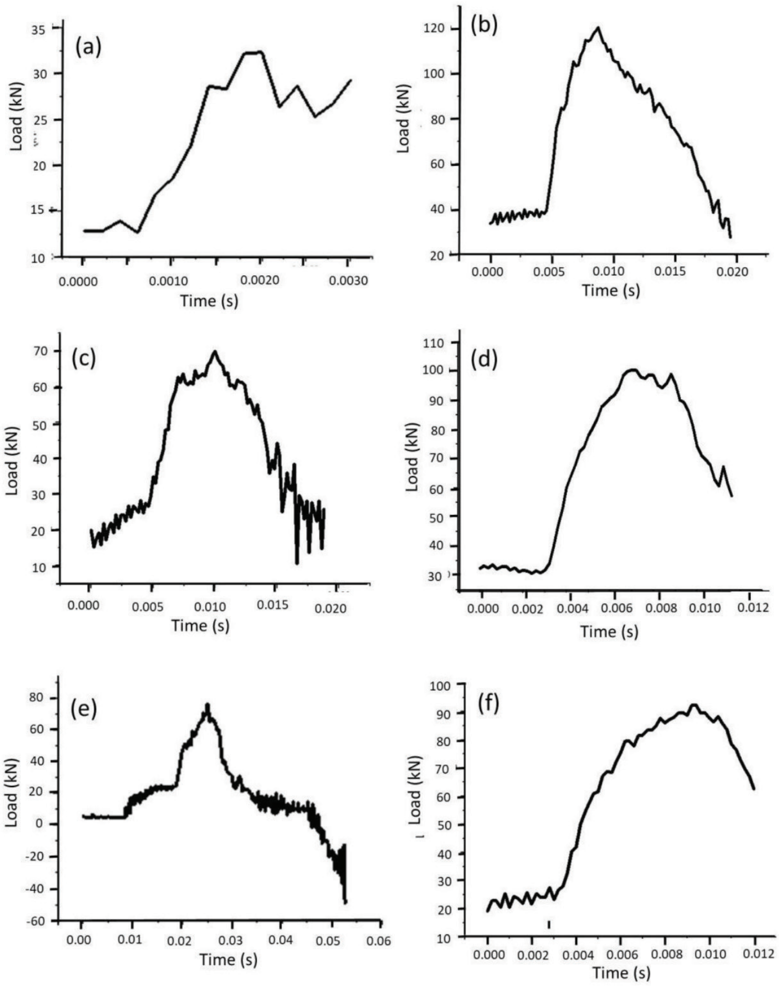

Figure 3: Impact load-time responses of bR-rHDPE composites of (a) 50bR-HDPE+bitumen, (b) 50bR-rHDPE, (c) 60bR-rHDPE+bitumen, (d) 60bR-rHDPE, (e) 70bR-rHDPE+bitumen and (f) 70bR-rHDPE

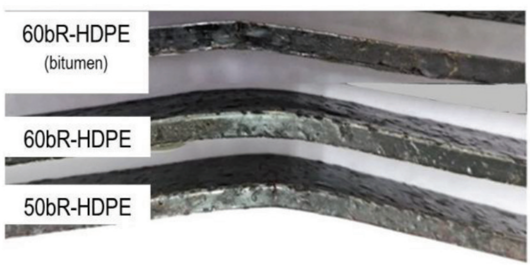

Figure 4: Deformed samples after drop-weight impact testing

Table 2: Peak-load values of bR-rHDPE composites

Sample	With bitumen (kN)	Without bitumen (kN)
50bR-HDPE	40.99 ± 11.77	123.09 ± 11.77
60bR-HDPE	69.13 ± 5.90	108.45 ± 8.50
70bR-HDPE	64.06 ± 10.84	86.39 ± 5.18

its high modulus (1,380 MPa) (Figure 6). The highest stiffness was observed in the 50bR-rHDPE sample because it contains the highest amount of rHDPE; 50 wt%. In contrast, the load - time history of the 50bR-rHDPE+bitumen sample shows a sudden drop with minimal oscillation after it reached its peak-load in a shorter period (Figure 3a). The sample fractured into two, whereby corroborating the data. This situation occurred because the sample had lost its impact resistance, which enabled the impactor to perforate it.

Regardless of whether the composites contain bitumen, higher bR loading (60 and 70 wt%) had reduced stiffness. The samples were more flexible, which translated to better impact damping capabilities. The improved flexibility of the samples was corroborated by the Charpy impact test results (Figure 2). A comparison of the load-time curve of samples for 60bR-rHDPE and 70bR-rHDPE composites with (Figure 3c,e) and without bitumen (Figure 3d,f) reveal that samples with bitumen have the following characteristics: i) lower onset-load (< 20kN), ii) intense oscillation after attaining peak-force, and iii) longer impact duration. The characteristics of the load-time curve also confirm the greater flexibility of samples for 60bR-HDPE+bitumen and 70bR-HDPE+bitumen. This was further evidenced by the prompt impact damage response when the impactor struck the surfaces of the samples. The higher impact damping capabilities of both samples with bitumen (60 and 70 wt%) managed to prevent the impactor from perforating the samples. This was achieved by decelerating the impactor that stabilised the crack. These mechanisms enhanced the impact energy absorption, which was reflected by a lesser degree of bending deflection of both samples compared to the stiffer 50bR-rHDPE sample (Figure 4). From the results, it can be concluded that the incorporation of bR and bitumen had improved the damping properties via the absorption of more impact energy.

Morphological Properties

The results of fracture surface from the Charpy impact testing on rHDPE and bR-rHDPE composites, with and without bitumen, are shown in Figure 5. An rHDPE (Figure 5a) exhibits many small deformed fragments, which consist of drawn micro-fibrils, indicating plastic deformation (Samat *et al.*, 2010). However, the fracture surface of the 50bR-rHDPE sample was different from that observed of the rHDPE sample. A distinct bR granule and HDPE matrix were spotted in the bR-rHDPE composite without bitumen (Figure 5b). An examination of this fracture surface found strong and weak adhesion strengths between the constituents. Weak bR granules and HDPE matrix adhesion in some parts of the composite have led to interfacial debonding and the formation of voids, whereas good bR granules and HDPE matrix adhesion were observed in other parts (Figure 5b and 5d). These observations concluded that the choice of mechanical processing can induce good surface interaction but at different adhesion strengths. The presence of voids causes a slightly lower impact toughness of samples without bitumen compared to the samples with bitumen. Despite the presence of voids, the impact toughness of bR-rHDPE composites without bitumen was better than that of the rHDPE samples. As previously mentioned, the major energy absorbing mechanism is attributed to the damping properties of bR.

The addition of bitumen had resulted in the characteristic of wavy fracture surfaces which signifies plastic deformation (Figure 5c). Wavy fracture surfaces were consistently found in tougher materials (Lazim & Samat, 2019), indicating that a higher amount of energy is absorbed. Unlike the 50bR-rHDPE sample (Figure 5b), bR granules and HDPE matrices were virtually indistinguishable and seamless in the 50bR-rHDPE+bitumen sample (Figure 5c). Bitumen encapsulated both phases and filled any voids between the bR granules and HDPE matrices. Furthermore, the fracture surface had fewer and smaller voids. These micrographic findings supported the initial assumption that bitumen facilitates the interfacial adhesion of composites and induces the composites to flow plastically.

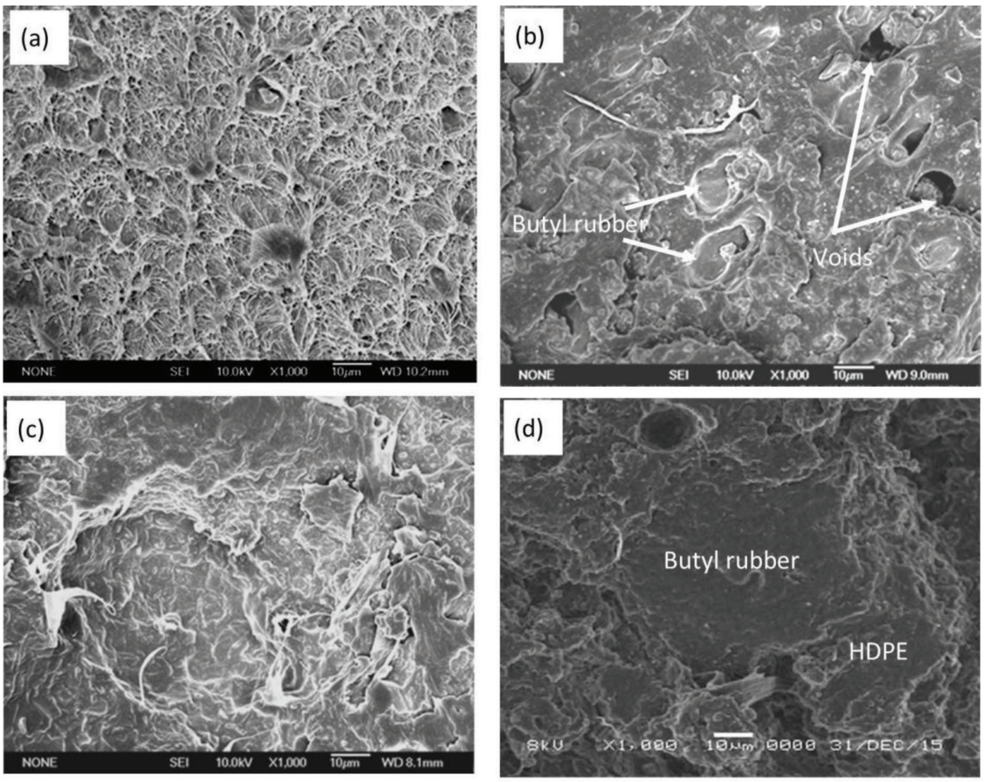

Figure 5: SEM micrographs of impact-fracture surface of (a) rHDPE, (b) 50bR-rHDPE and (c) 50bR-rHDPE+bitumen (d) 60bR-rHDPE composite without bitumen

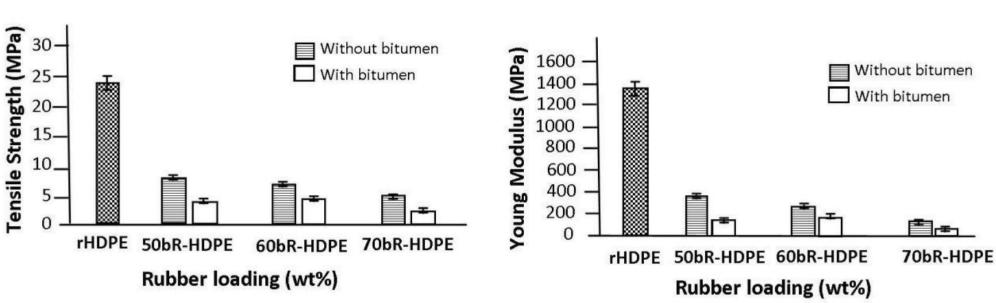

Figure 6: (a) Tensile strength and (b) Young's modulus of bR-rHDPE composites

Tensile Properties

Figure 6 shows the tensile test results of rHDPE and bR-rHDPE composites in correlation to the bR loading. Higher bR loading has led to a significant decrease in tensile strength. The decrease was much greater in composites containing of bitumen with more than 70% decrease when compared to rHDPE. In addition, Young's modulus (Figure 6b) decreased linearly with every addition of bR. This finding shows that the stiffness of rHDPE was reduced by the presence of bR. The results in this work are consistent with the studies by Mujal-Rosas *et al.* (2011), and Kakroodi and Rodrigue (2013) on HDPE/ground tyre rubber (GTR) composites; and Xu *et al.* (2020) on rubber/wood fibre composites. These studies found that higher GTR or rubber concentrations show a considerable drop in the tensile strength and Young's modulus of composites. The addition of rubber particles with different size (500and 1000 μm) exhibits similar results (Montagna & Santana, 2012) when the concentration of GTR was high. According to Tao *et al*. (2013) the devulcanisation level of GTR which was obtained from the mechanical shearing process had affected the tensile properties. A relatively low or excessive devulcanisation level had deteriorated the tensile property (Aoudia *et al*., 2017). Hence, it is suggested that the appropriate devulcanisation level might had not been achieved in this work.

The stress-strain behaviour of bR-rHDPE composites under tensile loading is presented in Figure 7. The incorporation of higher bR loading has resulted in a decrease in yield strength. However, the strain at the failure level increased concurrently. The presence of bitumen has caused a further decrease in yield strength. A comparison with samples without bitumen found a two-fold decrease, but these samples were fractured or broken at higher strain. Composites with bitumen have better strain than those without bitumen, which occurred due to the plasticising effects of bitumen (Zhang *et al.*, 2009) and better interfacial adhesion (Lievana &

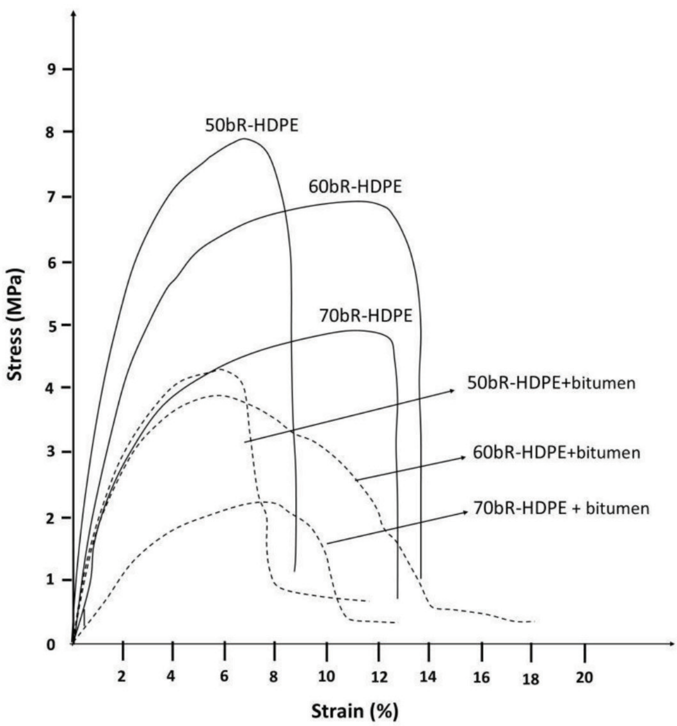

Figure 7: Stress-strain curve of bR-rHDPE composites

Karger-Kocsis, 2004). Fracturing or breaking at higher strain was only observed in 50bR-rHDPE and 60bR-rHDPE samples. On the other hand, 70bR-rHDPE samples were fractured or broken under lower strain, which explained the slight drop in the Charpy impact test results of 70bR-rHDPE composites.

Conclusion

This study had investigated the influence of waste butyl rubber (bR), from the used inner tubes along with the addition of bitumen on the behaviour of recycled HDPE (rHDPE) composites. The investigation was conducted using Charpy impact testing, drop-weight impact testing, and tensile testing. Comparisons were drawn between rHDPE, bR-rHDPE of varying bR loadings (50, 60 and 70 wt%) as well as with and without the addition of bitumen. The impact resistance of rHDPE composites had substantially improved regardless of bR loading and without the addition of bitumen. A major energy absorbing mechanism was inherited from the damping properties of bR. However, higher loading of bR (70 wt%) has led to a slight decrease in impact resistance, but these results were better than the tests on rHDPE. The addition of bitumen also led to positive impact resistance results. This was further corroborated by the findings of the instrumented drop-weight impact test. The deformation by the impactor was delayed by the presence of bR and further delayed in samples that contain bitumen. Micrographs from a scanning electron microscope (SEM) discovered voids and varying degrees of interfacial adhesion in bR-rHDPE composites without bitumen. The plasticising effect and devulcanising potential of bitumen had induced plastic deformation or flow plasticity. However, the results from tensile strength and Young's modulus tests indicate the opposite of the impact resistance test results; the good interfacial adhesion did not improve the tensile strength.

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