Static and Dynamic Mechanical Properties of Rice Husk (RH)/Linear Low Density Polyethylene (LLDPE) Composites under Various Loading Rates

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In this study, the static and dynamic properties of LLDPE/RH composites, with different filler contents of 5 wt% RH, 10 wt% RH, 15 wt% RH, 20 wt% RH and, 30 wt% RH were studied at different levels of strain rates (0.001/s, 0.01/s, 0.1/s, 650/s, 900/s and 1100/s) using a conventional Universal Testing Machine and Split Hopkinson Pressure Bar apparatus, respectively. Results show that the strength, stiffness and yield behavior of LLDPE/RH composites were strongly affected by both filler content and strain rate loadings. Apart from that, the rate of sensitivity of LLDPE/RH shows sgreat dependency towards applied strain rate, where it was increased with increasing strain rate. Unfortunately, the thermal activation values show contrary trend. Besides, at dynamic loading, the fracture surface analysis of the composites showed that all specimens experienced massive plastic deformation.

Keywords: Strain rate;Universal testing machine; Split Hopkinson Pressure Bar; Strain rate sensitivity; Thermal activation volume

With growing environmental awareness, ecological concerns and new legislatures, bio-fiber reinforced plastic composites have received remarkable attention during the recent decades. The composites have many advantages over traditional glass fiber or inorganic mineral filled materials, including lower cost, lighter weight, environmental friendliness, and recyclability [1]. Since rice husk and other bio-fibres easily undergo thermal degradation beyond 200°C, thermoplastic matrix used in the composites is mainly limited to low-melting-temperature commodity thermoplastic resins such as polyethylene (PE) and polypropylene (PP) [2]. One of the most promising bio-based polymers that have attracted the interest of many researchers is linear low density polyethylene (LLDPE). LLDPE is an important commercial polymer which is widely used for different applications in modern technology. It has higher tensile strength, impact and punctures resistance when compared to low density polyethylene, and is very flexible, elongates under stress, and can be used to make thinner films, with better environmental stress cracking resistance. It has also good resistance to chemicals. LLDPE has penetrated almost all traditional markets for polyethylene; it is used for plastic bags and sheets (i.e where it allows using lower thickness than comparable LDPE), plastic wrap, stretch wrap, pouches, toys, covers, lids, pipes, buckets and containers, covering of cables, geo-membranes, and mainly flexible tubing. LLDPE can be recycled though into other things like trash can liners, lumber, landscaping ties, floor tiles, compost bins, and shipping envelopes[3].

Currently, various organic fillers such as rice husk, rice straw, saw dust, egg shell, and kenaf fiber are being incorporated into LLDPE. As a result, the use of natural/bio-fibers reinforced composites has been rapidly expanded due to the availability of natural/bio-fibers derived from annually renewable resources, for use as reinforcing fibers in both thermoplastic and thermosetting matrix composites as well as for the positive environmental benefits gained by such materials [4]. Besides, cellulose that content in organic filler will affect the mechanical properties of the composites. In addition, cellulose is one of the strongest and stiffest fibers available and it has a high potential to act as reinforcing agent in biopolymers. Cellulose-based polymer composites are characterized by low cost, desirable fiber aspect ratio, low density, high specific stiffness and strength, biodegradability, flexibility during processing with no harm to the equipment, and good mechanical properties [5].

On the other hand, rice husk (RH) is one of the major food crops in the world that can be used as organic filler due to its availability. Rice husk is a potential material, which is amenable for value addition. Most of the husk from the milling is either burnt or dumped as waste in open fields and a small amount is used as fuel for boilers, electricity generation, bulking agents for composting of animal manure. Disposal of RH is particularly serious problem, which requires special attention due to its large quantities [6]. This problem can be overcome by producing value-added products, such as composite materials from RH fillers. The use of RH in making composite products is

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Identification	LLDPE (wt %)	Rice husk (wt %)
LLDPE	100	0
LLDPE/5% RH	95	5
LLDPE/10% RH	90	10
LLDPE/15% RH	85	15
LLDPE/20% RH	80	20
LLDPE/30% RH	70	30

Table 1
THE FORMULATION USED FOR LLDPE/
RH COMPOSITES

attracting much attention due to its remarkable potential for enormous gains in certain important properties of these products [7]. The wide use of non-renewable raw polymers in many industrial and domestic fields causes ecological problems connected with their utilization. Some synthetic plastics including polyethylene are characterized by relatively high stabilities under both photochemical and environmental conditions. The use of natural or synthetic photo- and biodegradable polymers is promising, but still problematic, for packaging materials [6]. Many studies were done on various material types [7-11]. From previous findings, it has been reported that different filler loadings have given significant effect to the mechanical properties of particulate natural filler reinforced composites. Besides, the performance of these composites under various strain rate conditions is also important, in order to prevent any misfortune during service. From published literatures on mechanical properties of particulate natural filler reinforced polymer composites, it was realized that the effect of strain rate properties are rarely reported. Until now, none of the previous work has specifically reported the dynamic mechanical properties of LLDPE/RH composites. To overcome the lack of information in this kind of area, it is essential to carried out specific experiment to investigate the capability of LLDPE/RH composites under various strain rates loading. To achieve the goal, this experiment is purposefully design to characterize the compressive behavior of LLDPE/RH composites under both static and dynamic loading by using SHPB apparatus and the Universal Testing Machine (UTM), respectively. The effect of the loading rates and RH content, towards the static and dynamic mechanical properties of the composites, were carefully determined. For comparison purpose, the rate sensitivity as well as thermal activation volume of tested specimen were also calculated using an establish parameter based on their recorded flow stresses. The fractographic analysis was also demonstrated in order to investigate the failure mechanism of tested composites under dynamic loading.

Experimental part
Materials and methods
LLDPE was purchased from Titan Petchem (M) Sdn. Bhd. in pellets form. The melting point, the melt flow index and the density of LLDPE are approximately 160 °C, 2 g/10 min and 0.922 g/cm³ respectively. RH used as filler was obtained from Padi Beras Nasional Bhd. (BERNAS), Perlis, Malaysia. RH was ground into powder form using grinder machine. Then, RH powders were sieved to obtain the particle size of approximately 125μm.

Methodology
The particulate fillers (rice husk) and LLDPE granules were prepared to produce composites with different composition as shown in table 1.
LLDPE and RH were compounded using BENCHOP twin screw extruder. The optimum processing temperature used for LLDPE and the composites were 180 °C and the screw speed is 40 rpm. The pure LLDPE was run first and followed by the composites. After the pelletizing, the specimens

were dried at 60 °C for 3 h in oven to remove the moisture contents.
Compression Process
The specimens were compression moulded in hydraulic hot press using a button mould, into 12 mm diameter and 27 mm thick shapes. Firstly, the empty mould was heated to 160 °C before loading the specimen. Then, approximately 2.1 g of the composite was placed for every button mould cavity. Then, pre-heating was carried out for 20 min followed by compression for 10 min and cooling for 10 min. The moulded composite specimens were then cut using a bench saw, into size of 12 mm (diameter) x 18 mm (length) and 12 mm (diameter) x 6 mm (length) for both static and dynamic mechanical testings, respectively.

Mechanical tests
Static compression testing
For static testing, the compression specimen was compressed under a constant crosshead speed of 1.08 mm/min, 10.8 mm/min and 108 mm/min; which corresponds to strain rates of 0.001 s⁻¹, 0.01s⁻¹ and 0.1s⁻¹, using a Universal Testing Machine. As a precaution, a thin film of lubricant was pasted onto both ends of the compression specimens to eliminate the needles effect (i.e. Frictional effect) during the test. Five measurements were taken for each strain rate, in order to quantify the average behaviour of the tested specimens.

Dynamic compression testing
The dynamic compression test was performed using the compression Split Hopkinson Pressure Bar (SHPB) apparatus. Generally, the SHPB apparatus consist of several parts, which are the propelling mechanism; striker bar, incident bar and support stand [12]. The striker, incident and transmitter bars are circular steel rods having the same diameter, and a small disk shaped specimen is placed between the incident and transmitted bars [13]. The schematic diagram of the SHPB apparatus used in this study is depicted in figure 1.
Theoretically, during SHPB test, a striker bar is fired at a certain velocity to collide with the incident bar, creating an incident strain pulse, ε_I, which propagates along the bar until it reaches the specimen. At this point, acoustic impedance mismatch between bar and specimen material results in a portion of the pulse being reflected back along the incident bar, producing a strain ε_R, while some of the pulse is transmitted through the specimen ε_T [14]. The propagation of the elastic wave in slender cylinder bar during the SHPB test can be described by the one-dimensional elastic wave equation. On the other hand, one-dimensional elastic wave equation were also used to obtain strain and stress in the samples [15]. The histories of stress, strain and strain rate during compression SHPB testing were calculated based on the strain measured on the incident

$$\sigma_S(t) = E\frac{A_b}{A_s}\varepsilon_r(t) \tag{1}$$

$$\varepsilon_S = -\frac{2C_0}{L}\int_0^t \varepsilon_r(t)dt \tag{2}$$

$$\dot{\varepsilon}_S = \frac{d\varepsilon(t)}{dt} = \frac{-2c}{L}\varepsilon_R(t) \tag{3}$$

and transmitter bars. The equations involved are shown as follows:
where A, E and c (= E/$\rho^{1/2}$, ρ is mass density of the bar) are cross-sectional area, Young's modulus and wave velocity of the bars, respectively. L and A_s refer to the length and cross-sectional area of the sample. $\varepsilon_r(t)$ and $\varepsilon_t(t)$ are the recorded axial strains of the reflected pulse and transmitted pulse, respectively, measured in reflected and transmitted bar as function of time.

The derivation of equation (1)-(3) is closely related with the following assumptions and idea [16] :
- the propagation of wave in the Hopkinson bars is well approximated by one-dimensional theory where the wave dispersion is totally negligible;
- the stress and strain states in the specimen are homogeneous;
- the friction and radial inertia effect are negligible.

Results and discussions
Strength properties
In this study, the relationship between the compressive strength and filler loading of the composites at several of strain rate is portrayed in figure 2. The bar graph in figure 2 clearly shows that the LLDPE/RH composites react differently under both static and dynamic loading. For static loading, the ultimate compressive strength (UCS) increases from LLDPE up to 15wt% of RH. However, it started to decrease when the filler content is above15 wt% of RH. However, for dynamic loading, the ultimate compressive strength (UCS) increases from LLDPE up to 20 wt% of RH

and started to decrease when the filler content is above 20 wt% of RH. However, after 20% of RH, it showed the decrement in UCS values. Theoretically, it is contributed by the agglomeration of filler that occurs within the LLDPE/RH body. According to Fu et al. [17] , it is believe that the increment of strength values occurs up to certain optimal filler content, before it starts to drop due to weaker particle dispersion and the agglomeration problems. As Rabiatul et al. [18] pointed out; the increase in the filler content may increases the micro-space between the filler and the matrix that weakens the LLDPE/RH composites interfacial adhesion.

Besides, the LLDPE/20% RH composite recorded approximately 8 MPa and 14.8 MPa of ultimate compression strength, under the static region (0.001 s^{-1} to 0.1 s^{-1}) and under the dynamic region (650 s^{-1} to 1100 s^{-1}), respectively. The strength is higher at dynamic loading, because at higher strain rates, the strength increases linearly with the strain in the initial elastic region. After the yield point, the specimen deforms plastically before it fractures. According to Dung et al. [19], the increment of the strength can be attributed to the additional of filler particles into the viscoelastic LLDPE matrix. The filler particles may react as load-bearing elements in the tested composites, thus reduce the overall elasticity of the material.

Stiffness properties
The compression modulus of neat LLDPE and LLDPE/RH composites were obtained under various levels of strain rates, as shown in figure 3. From figure 3, it is clearly seen that the relationship between strain rate and compression modulus are almost identical under both static and dynamic loadings, where the compression modulus increases steadily with increasing strain rate. The highest
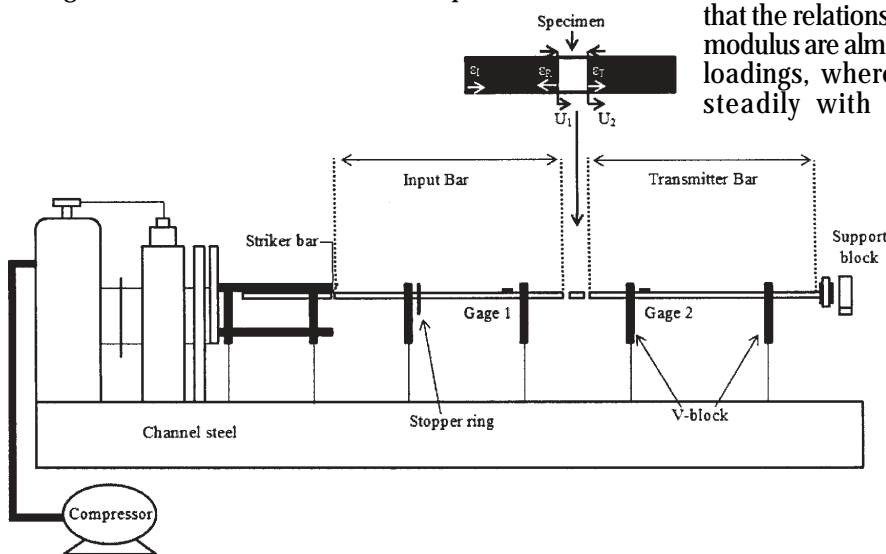

Fig. 1. The schematic diagram of the split Hopkinson pressure bar apparatus
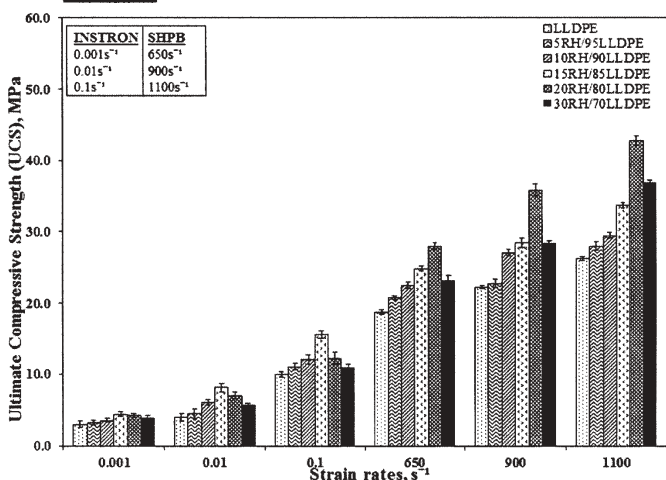

Fig. 2. The ultimate compressive strength (UCS) of the pure LLDPE and the LLDPE/RH composites under various levels of strain rates investigated
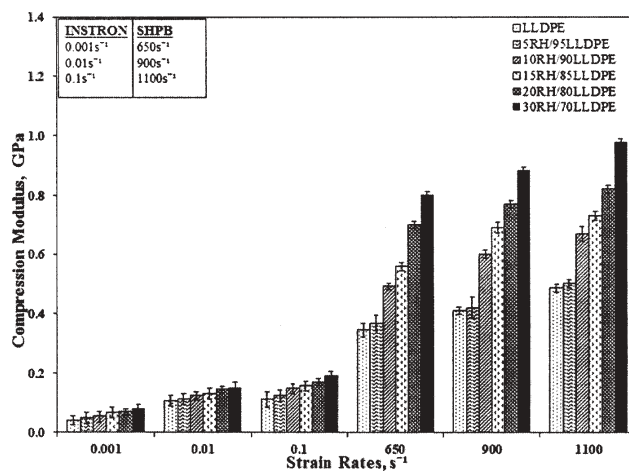

Fig. 3. The compression modulus of the pure LLDPE and the LLDPE/RH composites under various levels of strain rates investigated
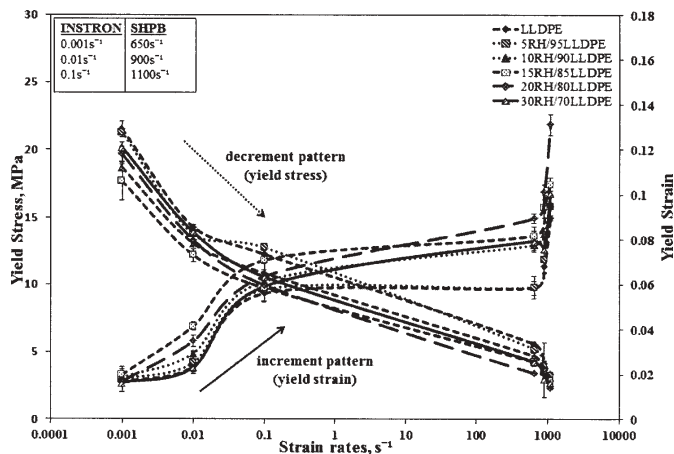
Fig. 4. The yield stress and the yield strain of the pure LLDPE and the LLDPE/RH composites under a wide range of strain rates investigated

compression modulus was recorded by 30 wt% of RH compare to LLDPE for both static and dynamic testing. For example, the LLDPE/30% RH composite recorded a relative increment of about 0.72 GPa and 0.178 GPa between 0.001 s^{-1} to 650 s^{-1} and 650 s^{-1} to 1100 s^{-1}, strain rates respectively. This finding is consistent with the work reported by Chen et al. [20] , where it believe that this increase in stress has a close relationship to the secondary molecular processes. Increasing strain-rate will decrease the molecular mobility of the polymer chains and thus make the material stiffen [20].

Besides, filler loading has become an important factor that influences the compression properties of LLDPE/RH composites, which can be observed from figure 3. Visually, at a strain rate of 0.001 s^{-1} and 1100 s^{-1}, the compression modulus for pure LLDPE is 0.04 GPa and 0.485 GPa. Meanwhile, at 30% RH content, the compression modulus was found increase to 0.079 GPa and 0.977 GPa. The increments in compression modulus were strongly affected by the addition of RH filler, thus making the composites stiffen. As Supri et al. [21] pointed out; the addition of particulate natural fillers (water hyacinth) in the matrix will increase the rigidity of the composites. According to

Omar et al. [12], the increase in rigidity is associated with the lack of chain mobility and deformability of the matrix, due to the present of additional rigid particle (rice husk), which disturb the deformation of the crystalline region in the LLDPE matrix. Both reasons can be used to explain the increment of the compression modulus under various loading rates.

Yield behaviour

Figure 4 shows the yield stress and strain values at static and dynamic loading rates. For static loading, the yield stress increases from LLDPE up to 15wt% of RH. However, it started to decrease when the filler content is above 15 wt% of RH. It is believe that after 15 wt%, the RH filler tend to get contact to each other and form agglomerate. Theoretically, it was believed that this agglomeration will react as stress concentration and weaken the composite body. Similar finding has been reported by Medupin et al.[22],where they speculated that the filler particles may not interact with polymer matrix as the particles agglomerate. Another phenomenon was reported by Robert et al. (1993), state that particle agglomeration tends to reduce the strength of a material even though agglomeration may be strong enough to increase the initial modulus. In addition, agglomerates are weak points in the material and break fairly easily when stress is applied [23].

Besides, it is interesting note that, the increment of yield stress is different between static and dynamic loadings. Increment in yield stress, at static loading conditions, are observed up to 15 % filler content, before the value started to reduce at 20% filler content. In comparison, at dynamic loading, increase can only be seen at 20% of filler content and the yield stress started to decrease at 30 % of filler content. According to the Shergold et al. [24] , it is believe that at dynamic loading, the polymer chain is restricted due to insufficient time to re-oriented themselves thus increase the rigidity. Besides, restriction of the polymer chains mobility at a high strain rate loading may also enhance the formation of additional intermolecular force between the structures. These accumulated inter-molecular forces would give a strengthening effect to the

LLDPE/RH with different filler content (%)	Range of strain rates (s^{-1})	Classification	$\beta = \dfrac{\sigma^2 - \sigma^1}{\ln\left(\frac{\dot{\varepsilon}^2}{\dot{\varepsilon}^1}\right)}$ (MPa) $\dot{\varepsilon}^2 > \dot{\varepsilon}^1$ $\varepsilon = 0.025$	$V^* = kT\left[\dfrac{\ln\left[\frac{\dot{\varepsilon}^2}{\dot{\varepsilon}^1}\right]}{\sigma^2 - \sigma^1}\right]$ $\dfrac{kT}{\beta}$ (m^3) $\dot{\varepsilon}^2 > \dot{\varepsilon}^1$ $\varepsilon = 0.025$
LLDPE	0.001 to 0.1	Static	0.0869	4.3398×10^{-26}
	0.1 to 650	Static to dynamic	1.2043	3.1315×10^{-27}
	650 to 1100	Dynamic	15.3966	2.4494×10^{-28}
5	0.001 to 0.1	Static	0.2736	1.3784×10^{-26}
	0.1 to 650	Static to dynamic	1.3047	2.8905×10^{-27}
	650 to 1100	Dynamic	13.3057	2.8343×10^{-28}
10	0.001 to 0.1	Static	0.2823	1.3359×10^{-26}
	0.1 to 650	Static to dynamic	1.8682	2.0187×10^{-27}
	650 to 1100	Dynamic	7.6033	4.9600×10^{-28}
15	0.001 to 0.1	Static	0.2845	1.3256×10^{-26}
	0.1 to 650	Static to dynamic	2.1152	1.7830×10^{-27}
	650 to 1100	Dynamic	7.4132	5.0870×10^{-28}
20	0.001 to 0.1	Static	0.2888	1.3058×10^{-26}
	0.1 to 650	Static to dynamic	1.2691	2.9716×10^{-27}
	650 to 1100	Dynamic	34.9751	1.0783×10^{-28}
30	0.001 to 0.1	Static	0.2779	1.3570×10^{-26}
	0.1 to 650	Static to dynamic	1.3201	2.8568×10^{-27}
	650 to 1100	Dynamic	18.6280	2.0245×10^{-28}

Table 2
THE RATE SENSITIVITY AND THERMAL ACTIVATION VOLUME OF LLDPE/RH COMPOSITES WITH DIFFERENT STRAIN RATES
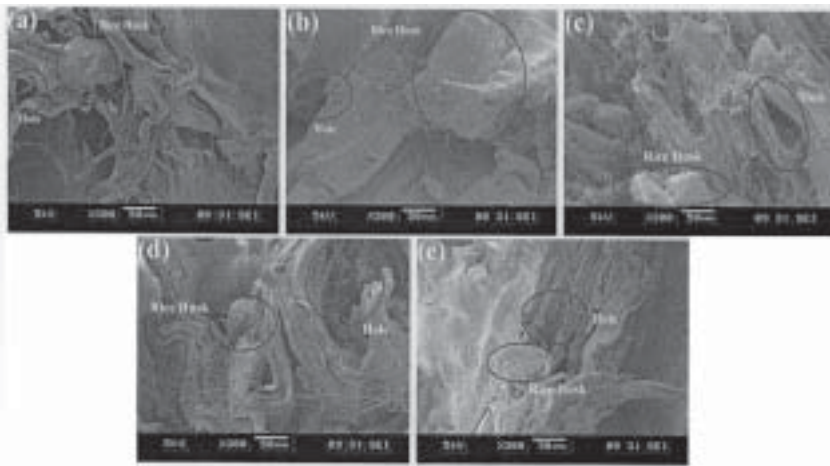
Fig. 5. The fracture structure of LLDPE/RH composites, (A) 5% RH, (B) 10% RH, (C) 15% RH, (D) 20% RH and (E) 30% RH at dynamic loadings
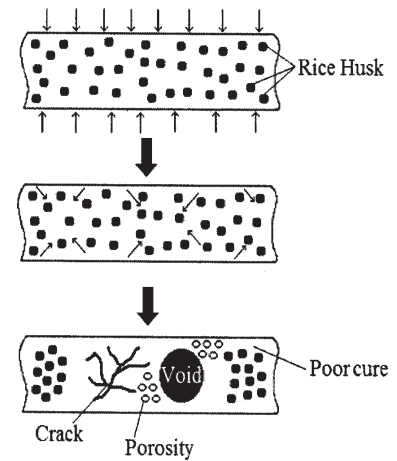

Fig. 6. The schematic diagram of LLDPE/RH composites fracture structure at dynamic loadings

composite as the strain rate increase [25]. On the other hand, the yield strain decrease with increasing strain rates for all tested specimen. Higher yield strain values indicate that the composites are more ductile and easily deformable [25]. For static loading, the yield strain for 15% RH contents contributes the highest value while for the dynamic loading; the yield strain for 20% RH contents contributes the highest value.

Strain Rate Sensitivity and Thermal Activation Volume

In summary, all calculated strain rate sensitivities and thermal activation value of each LLDPE/RH composites specimen have been grouped together and illustrated in table 2. From the results shown in table 2, it can be seen that all tested specimens showed positive increment, in terms of their rate sensitivity of flow stress, with increasing strain rates, from static to dynamic regions. However, thermal activation volume shows a contrary pattern, which decreases with increasing strain rate. Thus, we believe that this phenomenon attributes to the mobility of polymer chain during applied strain rates. At dynamic loading, a higher flow stress is required to perform deformation up to 0.025 of strain, as the mobility of the polymer chain is restricted. Therefore, the rapid increasing flow stress will increase proportionally toward increasing rate sensitivity. However, the rapid transfer of mass along the polymer chain was also affected by the restriction of the chain's mobility at a high strain rate, that decrease the thermal activation volume [12].

Furthermore, it can be seen that the particle content does not give any significant trend on the rate of sensitivity and thermal activation volume of LLDPE/RH composites. This typically shows that LLDPE/RH composites recorded high rate sensitivities than that pure LLDPE. However, the variation is not consistent with particle content and applied strain rate. This trend is in the line with the one that been reported by Omar et al. [12], with different reinforcement type which is particulate mineral filler. However, the magnitudes of change in terms of strain rate sensitivity as well as thermal activation volume were slightly different. Statistically, particulate mineral filler reinforced composite recorded higher strain rate sensitivity than that of our specimen especially under dynamic loading. Based on the recorded result, we believe that the nature ability of both types of reinforcement play an important roles in determining the sensitivity of tested specimen. Natural filler (RH) with higher moisture content and highly porous structure may result in lower rate sensitivity then its counter-part (mineral filler).

Fracture Surface Analysis

From fractographic analysis, it was observed that the fracture surface of all tested composites at dynamic loading is relatively rough, due to the enhancement of the applied stress during a high strain rate loading. SEM micrographs for all tested specimen under 1100 s^{-1} of strain is illustrated in figure 5. According to Hajlaoui et al. [26], at high strain rate, the rate of shear band nucleation not sufficient enough to accommodate the applied strain rate. Thus, additional shear bands must be formed in order to accommodate the large amounts of imposed strain in a smaller time frame. With the shear bands, a step-like relief forms in a large portion of sample in figure 5 (a-e). The presence of large number of shear bands is then associated with increase in ductility [26]. At filler loading of 5 wt% and 10 wt%, a few filler particles are seen, the composites mainly representing plastic deformation. At 15 wt% filler loading, slightly increased numbers of holes where filler particles have pulled out traces are seen and at 20 wt% filler loading, more traces are appeared. At 30% filler loading, more filler particles are seen rather than polymer matrix. From this fractographic analysis, it was observed that the composite tend to be pulled-out under a dynamic loading that caused the presence of holes and voids, clearly indicating the poor adhesion interaction between the filler particles and the polymer matrix. Similar observation was reported by Yang et al. [27], state that poor interfacial bonding induces micro-spaces between the filler and matrix polymer, and these cause numerous micro-cracks when the testing occurs. This phenomenon can be pictured in the schematic diagram of LLDPE/RH fracture that illustrated in figure 6. When the LLDPE/RH composite are applied to dynamic loading, the stress transfer at the matrix/filler interface inefficient will enhance the formation of void, porosity and crack. Interestingly, the micrograph in figure 5 showed a good correlation with the strength properties discussed in figure 2.

Conclusions

In this study, static and dynamic compression tests were successfully performed on different filler loadings (5, 10, 15, 20 and 30 wt %) of RH under different strain rate loadings (0.001 s^{-1}, 0.01 s^{-1}, 0.1 s^{-1}, 650 s^{-1}, 900 s^{-1} and 1100 s^{-1}) using the conventional Universal Testing Machine and SHPB apparatus. From the overall results, the following conclusions can be drawn:
- the mechanical properties of all the tested LLDPE/RH composites showed a great dependency on the strain rate

applied. The ultimate compression strength, compression modulus and yield stress were proportionally increased as the strain rate increase. However, the yield strain showed a contradictory pattern where it was gradually decreased with applied strain rates;

- generally, the compression modulus of theLLDPE/RH composites increased with the addition of rice husk contents, under both static and dynamic loadings. Besides, increments in compressive strength, at static loading condition, are observed up 15 wt% of filler loading, before the value starts to decrease at 20 wt% of filler loading. In comparison, at dynamic loading, the increased can be seen at 20 wt% of filler loading and the compressive strength started to reduce at 30 wt% of filler loading;

- the strain rate sensitivity and thermal activation volume ofLLDPE/RH composites were successfully measured. It was found that the strain rate sensitivity of the LLDPE/RH composites increased with increasing strain rates, while the thermal activation values show contrary trend. In addition, it can be seen that the filler loading does not give any significant trend on the rate sensitivity and thermal activation volume of the LLDPE/RH composites;

- furthermore, from fracture surface analysis, it was observed that pulled-out traces of fillers can be seen at dynamic loading, enhanced the formation of holes and voids.

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