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Effect of Different Waste Coal Ash (WCA) Loading to Dynamic Load Application of Chloroprene Rubber

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ABSTRACT

This study seeks to address the effect of waste coal ash collected from burnt charcoal after human recreational activities to the chloroprene rubber's dynamic property specifically in cyclic load. Waste coal ash was chosen due to their availability, low cost, and recyclability. The waste coal ash was incorporated into chloroprene rubber via melt compounding process using an internal mixer. The compounding formulation of the chloroprene rubber compound and coal ash were based on 100 phr of the chloroprene rubber. The loading of waste coal ash were varied at 5, 10, 20, 30 and 40 phr. Waste coal ash exhibits the highest reinforcing property at 40 phr loading. The fatigue test was done to determine the peak stress value and stress softening effect and found that at 5 – 30 phr waste coal ash had a detrimental effect on CR compound while at 40phr, the rubber compound exhibit positive result with an increase of 30% of the peak stress. Morphological study using scanning electron microscopy (SEM) showed degrees of agglomeration of the waste coal ash in the chloroprene rubber matrices. The dispersion of waste coal ash agglomerates and morphology of failures were in good agreement with the observed dynamic properties.

Keywords:
Chloroprene rubber, Waste Coal Ash, Fatigue

1 Introduction

Chloroprene rubber (CR) is a very durable synthetic elastomer and easily moulded into different shapes. It exhibits good sealing property and with its elasticity are the reason for its choice as gaskets,

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oil seals and O-rings. Its elasticity also makes it good to be used in dynamic load application that requires loading and unloading of stresses. CR exhibits a good balance of properties and stability overa wide temperature range with good weathering properties. CR can withstand the cold environment up to -25 ° C and still works well below the heat of 93 ° C, but its specially formulated composition allows service at temperatures as low as -55 ° C. It is important among the elastomers due to the mixture of polarity and crystallography.

Everyday human activities has critical influence on the environment and in general, it has reached a significant level. Awareness and public concerns on their daily and recreational activities that affects the environment are now more discussed [1]. This is evident in recent studies where every day wastes from shrimp shells, egg shells and even chicken fats are further explored and investigated for the advanced material syntheses [2-4].

Coal ash contains silica (SiO_2) and previous work reported good interaction of silica with polar rubber with functional groups such as CR [4-9]. The drawback is having poor dispersion with the rubber matrices due to absorption between the curatives and silanol groups. Addition of silane have been reported to improve the dispersion of silica in the matrices [9-10].

Coal ash have been found to improve rubber properties especially mechanical, albeit at a lower magnitude compared to commercial silica. Expected improvement in dynamic load properties would benefit in the long run for chloroprene rubbers. Higher load sustaining would ensure the material could be used in more demanding applications.

Coal ash from electric power plants have long research history. Numerous studies have been conducted to engineer alternative usage of coal ash especially fly ash in order to reduce traditional disposal methods that have detrimental effects to the environment [4]. Another source of coal ash is from recreational activities such as barbecuing and grilling. In 2018, charcoal grills sales in the US amounted to 0.91 billion USD in sales. Since coal ash is hazardous to humans and it is the reason that its obtainment and disposal is strictly controlled by environmental authorities worldwide including Malaysia. Incorporating the waste coal ash (WCA) into the chloroprene rubber seems like a promising effort considering the good interaction between carbon based material with CR which reported by several studies [8]. It is observed to increase the mechanical properties of the rubber composites when incorporated at a suitable level in the matrices.

Mechanical component are not only subjected to static stresses but also mostly experiencing cyclic stresses during service. Considering this, material performance under dynamic loading is crucial. Fatigue life expectations in rubber studies are given by a component of stretch or strain tensor concurring to the number of cycles. Basically, fatigue prediction consists in representing a component of stress (SN curve) or strain (Manson-coffin curve) tensor with respect to the number of cycles. The fatigue experiment used dumbbell test specimen according to ISO 37. So, to characterize the number of cycles necessary to the rupture of the specimen has been used.

This study expects to show effects of different loading of waste coal ash (WCA) collected from human recreational activities towards the dynamic load property of chloroprene rubber composites.

2 Methodology

2.1 Materials

The chloroprene rubber trade name Neoprene was provided by Saiko Rubber (M) Sdn Bhd. They also provided the vulcanizing ingredients such as activator, accelerator, antioxidant, curing agent and coupling agent. The waste coal ash (WCA) as in Figure 1 were collected after recreational activities of burning coal and left to dry at ambient temperature for 24 hours. The coal ash was weighed to get final weight after drying. The coal ash was characterizing for their particle size by using a MALVERN

MASTERSIZER 2000 Particle Size Analyzer and also under ZEISS EVO 18 variable pressure scanning electron microscope shown in Figure 2.

Fig. 1. Waste coal ash (WCA) after drying

2.2 Sample Preparation

Compounding was done in Saiko Rubber (M) Sdn Bhd by using an internal mixer. Parameter of mixing at 180°C and rotor speed of 80rpm. Silane (Si 69) was added in order to assist with compatibility of the coal ash with chloroprene rubber based on previous study [10-11]. After compounding, it undergoes hot press using a GoTech GT-7014-H30C hydraulic molding press with a maximum load capacity of 30 tons. Pressing parameter was set at temperature of 170°C within five minutes of preheating, 10 minutes of pressing and five minutes for cooling time to properly cure the compound. Formulation recipe of chloroprene rubber based on Table 1 was provided by Saiko Rubber (M) Sdn. Bhd. based on their available current formulations.

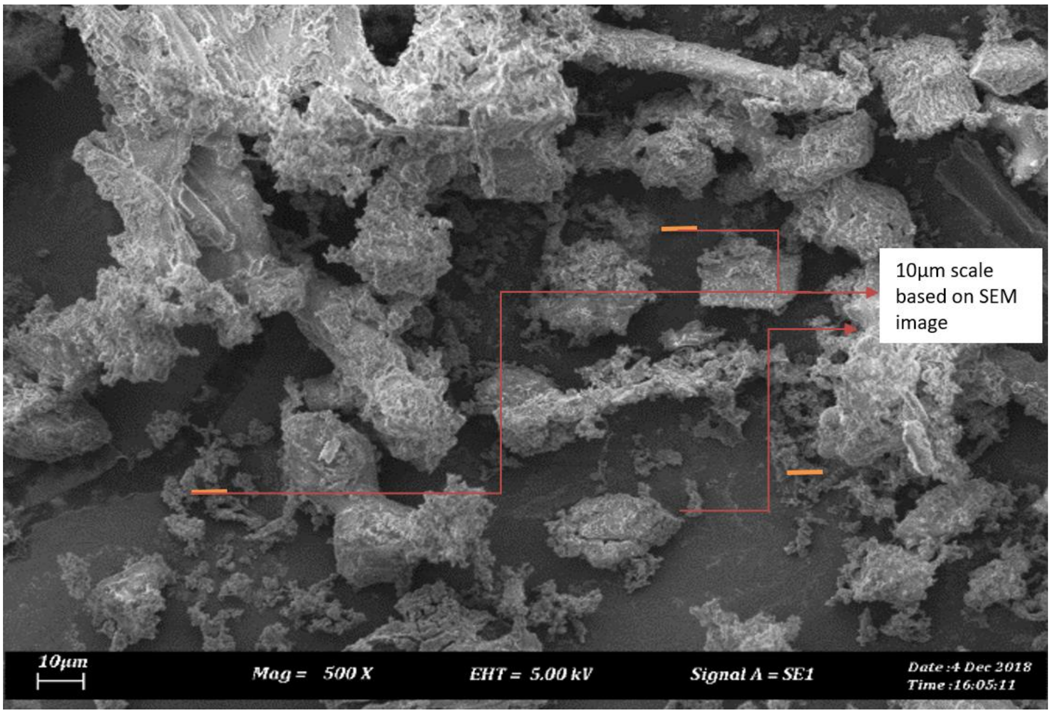

Fig. 2. Scanning Electron Microscope (SEM) image of waste coal ash at 500X magnification

2.3 Fatigue testing

The testing samples used were dumbbell test specimen dimensions which are 100 mm long and have a diameter of 25 mm in the gauge portion according to ISO 37 of 20mm. The testing was performed by using a Shimadzu 10kN compressed air servo controller fatigue testing machine EHF-EM10 with a maximum amplitude of 50 mm. The parameter was to elongate about 40 mm and must return to an original position, 100 cycles at 1Hz frequency and that has repeated for three times for each formulation. The fatigue life test determination for 100 000x no. of cycles was performed until the sample experiences rupture.

Table 1
Formulation of sample compounding

SAMPLE	Composition (phr)					
	Control Sample	1	2	3	4	5
Chloroprene	100	100	100	100	100	100
Coal Ash	0	5	10	20	30	40
ZnO	5	5	5	5	5	5
Stearic Acid	2	2	2	2	2	2
6PPD	2	2	2	2	2	2
TMTD	0.3	0.3	0.3	0.3	0.3	0.3
CBS	1	1	1	1	1	1
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5
Paraffin Wax	1	1	1	1	1	1
Si-69 (Silane)	0	1	1	1	1	1

Fig. 3. Sample for fatigue testing

3 Results

3.1 Particle size

Figure 4 shows the particle size distribution of the waste coal ash (WCA). It was found that 50% of the coal ash had a diameter of below 25.063 µm based on volume weighted particle size distributions method. The WCA was collected without further grinding shows potential of acquiring some portion of lower diameter particle sizes of less than 10 µm. However, there are higher diameter range of more than 80 to 600 µm presence in the collected WCA. The high order particles sizes could remained as agglomerates within the CR matrices and influenced the final properties of the composites. Further refining step was not introduced during the study to really investigate the as-produced loose WCA from the burning activities.

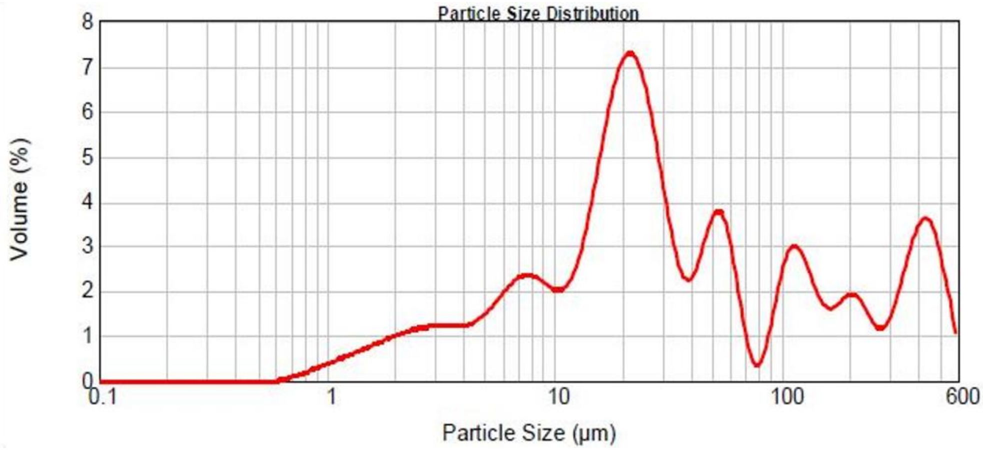

Fig. 4. Particle size distribution analysis

3.2 Vulcanization – cure time

Cure time testing was carried out at the temperature of 160 °C according to ASTM D5289 (Standard Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters). It was found that the addition of coal ash increases both scorch time T_{S2} and cure time T_{C90}. This means that with addition of coal ash, the rubber compound experiences delay when curing starts and further delayed its final curing time. Kongvasana had reported the same effect of fly ash particles on neat CR compounds, which they reported that the silica content has retarded the cure of non-polar NBR vulcanizates in their previous work [12].

Table 2
Rheometer test result

Sample	T_{S2} (min)	T_{C90} (min)
Control	2.10	5.46
1	3.24	7.11
2	5.08	7.57
3	6.11	8.31
4	6.42	8.47
5	5.55	8.53

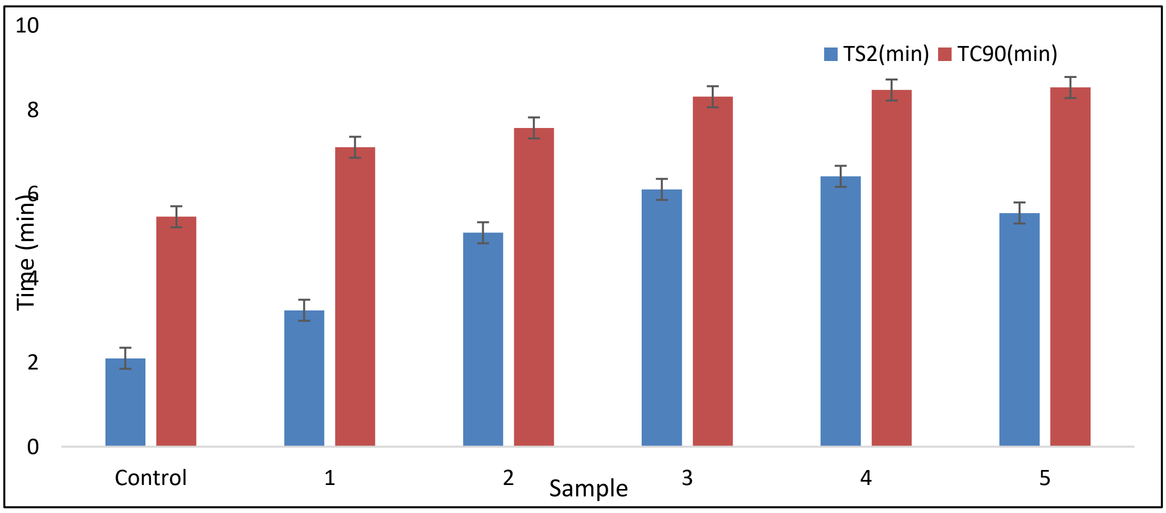

Fig. 5. The cure time and scorch time for the samples

3.3 Fatigue test analysis in stress vs strain (Mullins Effect)

The Mullins effect is a particular aspect of the mechanical response in filled rubbers in which the stress–strain curve depends on the loading condition. It is when filled rubbers exhibit significant inelastic responses such as hysteresis and stress softening under cyclic loading. To observe this phenomenon, in this study the data of stress vs strain for the CR composites was taken at 1 cycle until 100 cycles under the uniaxial tensile stresses. The stress vs strain graphs for control sample and sample loaded with 40 phr waste coal ash are depicted in Figure 6 (a) and (b).

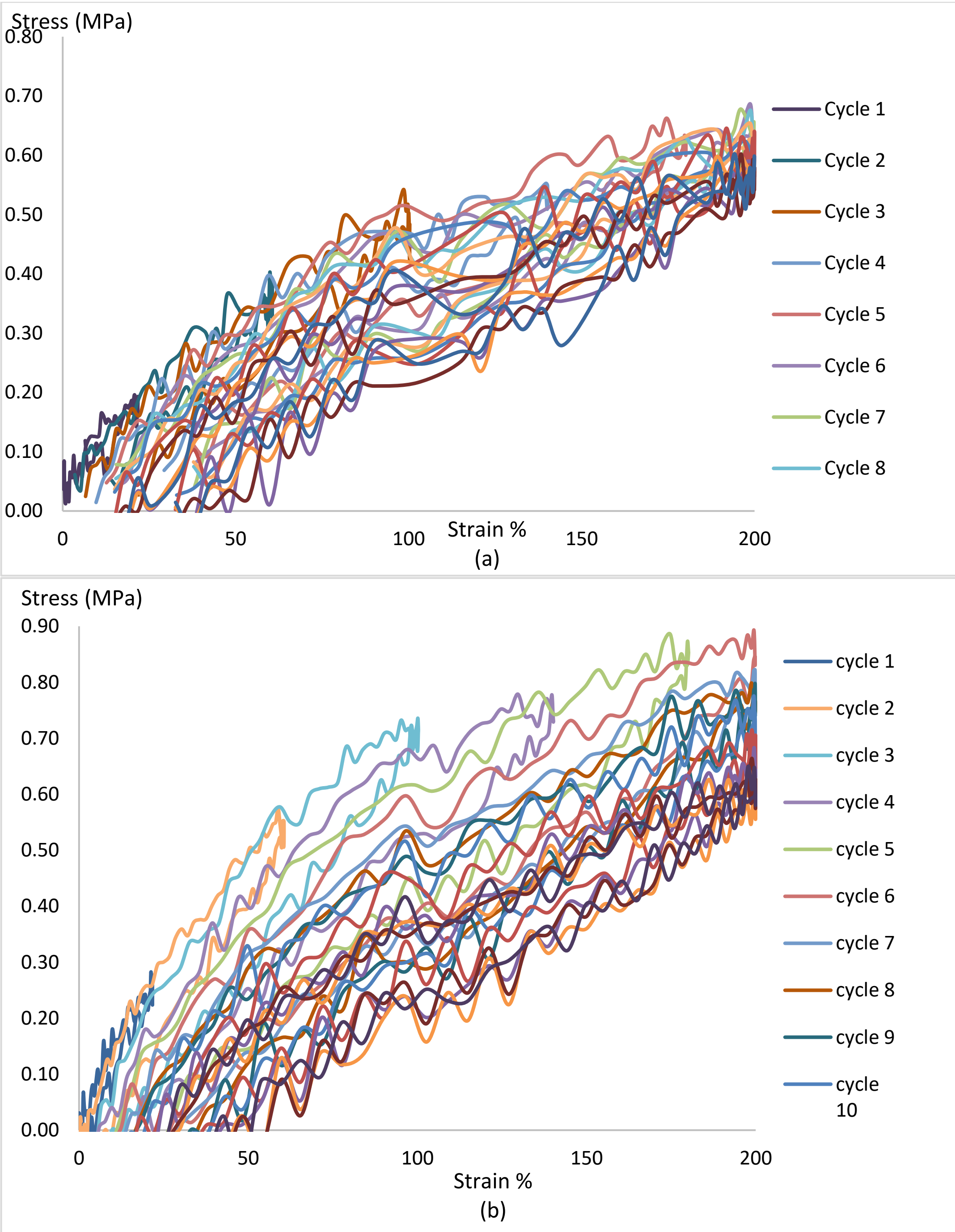

Fig. 6. The stress vs strain curve for (a) control sample and (b) 40 phr WCA sample

It is clearly observed that both control sample and sample 5 (40 phr WCA) exhibit the Mullins effect which disappears after around five loading cycles. After the 5th cycle, the loading and unloading paths reached the maximum set strain of 200% with gradual reduction in maximum stresses after every cycles completed. Hysteresis is characterized by various pathways of loading and unloading and may be correlated with either viscoelasticity, viscoplasticity or strain-induced crystallization. Stress-softening refers to a significant reduction in stress between two successive cycles, particularly between the first and second loadings.

From Figure 6, it is observed that the Sample 5 with 40 phr WCA reinforcement (b) had shown higher maximum stresses at every cycles if compared to the control sample (a). This is a clear indication of strengthening effect introduced by the presence of the WCA in the CR matrices.

In this study, the fatigue test was set to the maximum 100 000 cycles in order to determine the maximum no. of cycles to failure for each samples; the fatigue life. Figure 7 depicts the post failure samples after fatigue testing. The control sample manifested the lowest fatigue life since it failed at the 50 000 cycles during the cyclic loading. The CR chains easily slipped on top of one another, rearranged themselves under tensile stresses and experienced rupture once the localized stress value exceeded the tensile strength of the material. Meanwhile, no rupture evident for sample 1 and sample 2 with 5 phr & 10 phr WCA loading even at the 100 000 cycles. At this level, the WCA might serve as the lubricants to ease the movement of the chains under deformation with certain degree of reinforcing effect and increase the toughness of the material.

The reinforcing effect increases with the increase of WCA loading and reached maximum at 40 phr. However, as the WCA loading increased beyond 10 phr, WCA agglomerations become apparent and might act as stress concentrator once the chains fully strengthened under the tensile loading. It initiates crack which further propagates as the stress increases. It is observed clearly when sample 3 until sample 5 with 20, 30 and 40 phr loadings experienced crack and eventually rupture at the maximum cyclic loading of 100 000 cycles. Therefore, it clearly shows that the variation in the coal ash as a filler material affects the fatigue behavior of the CR composites.

Fig. 7. Sample testing after fatigue test

3.4 Effect of peak stress with different loading of coal ash

Peak stress is defined as the maximum value of stress that was reached their corresponding cycle. The subsequent value of peak stress reduces with increasing number of cycles due to rubber softening. Figure 8 shows the peak stress values versus number of cycles for all samples. The control sample highest peak stress value was at 0.6868MPa. The addition of coal ash from 5 to 20 phr has reduced the peak stress values. The values decreasing with the lowest at 0.5462 MPa at 10 phr. After 30 and 40 phr the sample increased its peak stress at the highest value of 0.8929 MPa for 40 phr. An increase of 30% of peak stress value. This value is comparable to fatigue testing of a polychloroprene rubber reinforced with carbon black reported by Berton et al. [13] and Poisson et al. [14]. Loo et al had also experienced a gradual drop of peak stress progressively which relates to the loading effect seen Figure [15].

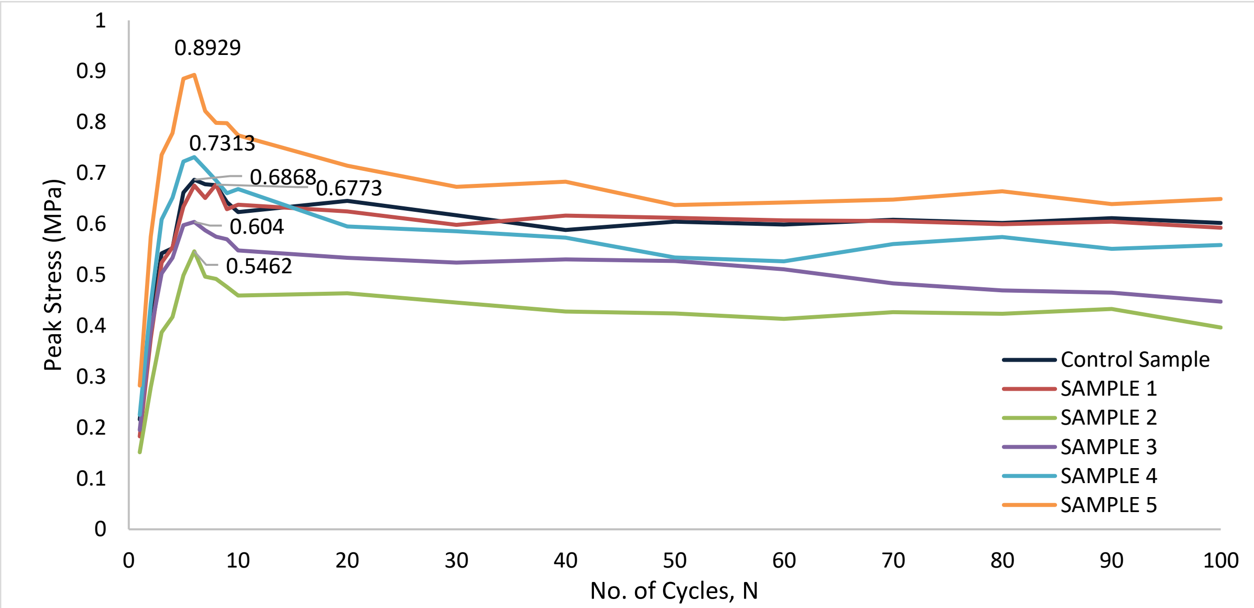

Fig. 8. Peak stress values versus number of cycles

3.5 Scanning Electron Microscopy

Morphological study was performed by using a Scanning Electron Microscope (SEM) on the 40 phr sample since it exhibited the most increase in dynamic load effect with the addition of WCA. Comparison of the morphology was performed with the control sample. Figure 9 (a) shows the morphology image of control sample at 500x of magnification while Figure 9 (b) shows the 40 phr of WCA sample.

The 40 phr WCA sample showed a rougher surface morphology and increased the tearing lines as compared to control sample. These tearing lines correlates with the increased in mechanical property especially the resistance to crack propagation which explains the increase in peak stress value and dynamic property [16-17]. Surface defects are more evidenced with the addition of WCA as more voids exits from the agglomeration of WCA.

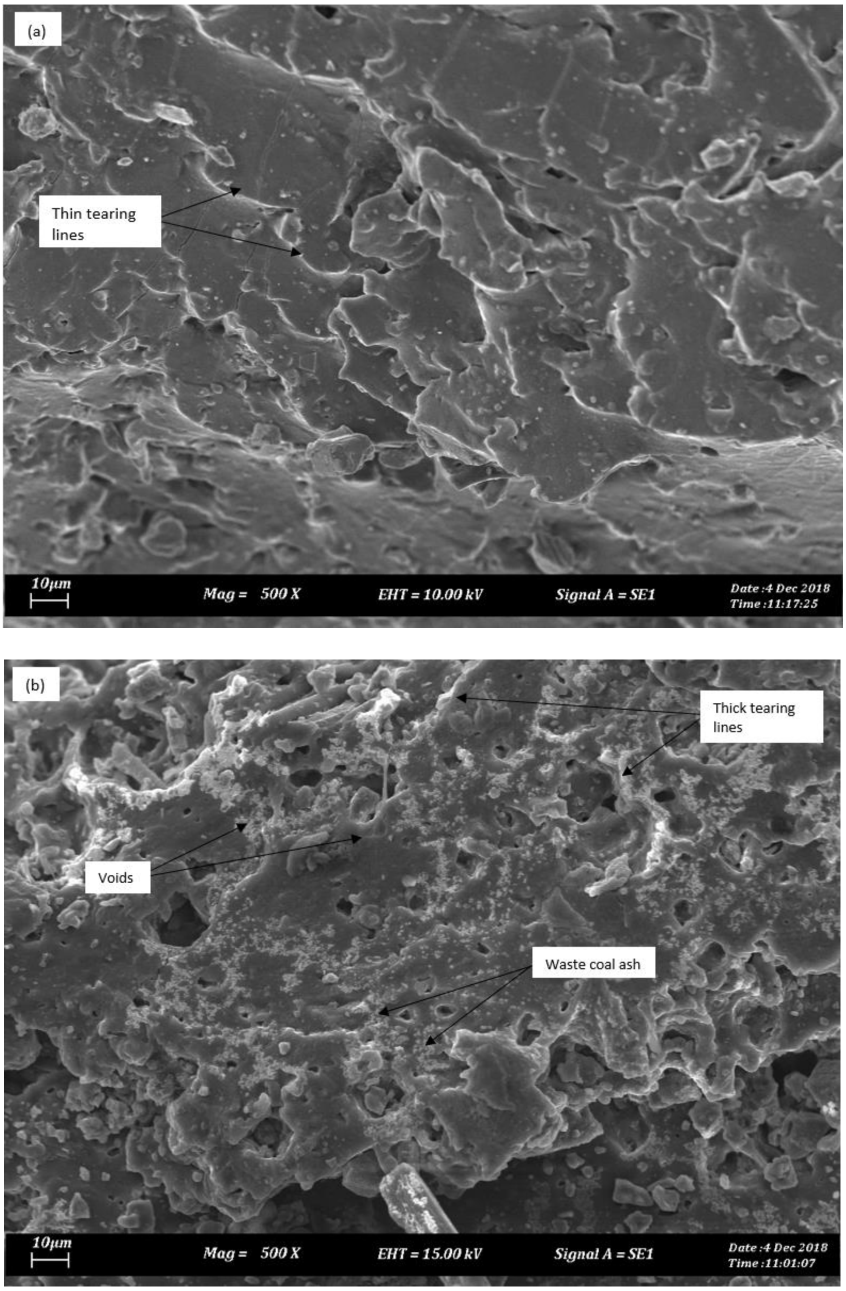

Fig. 9. SEM image of (a) control sample and (b) 40 phr WCA sample

4 Conclusions

The study on the effect of different loading of waste coal ash (WCA) on the dynamic load application for chloroprene rubber has been conducted. It was found that the 40 phr of WCA has contributed to the highest increase in peak stress value of chloroprene rubber. Lower than 40 phr loading of WCA has resulted in a detrimental effect to the stress peak value. WCA also retards the scorch and curing time for chloroprene rubber. Morphological study shows that with the addition of WCA resulting in tearing line and rougher surface characteristic when compared to control sample.

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