PHYSICAL AND THERMAL PROPERTIES OF RUBBER GLOVE WASTE AS POTENTIAL FILLER FOR POLYMER COMPOSITES

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ABSTRACT: A serious threat of the rising rubber waste globally has made researchers extensively look for an alternative to recycle it. Rubber waste is difficult to degrade due to its highly cross-linked structure that can lead to pollution and spreading of diseases. Rubber waste is very elastic and possess high durability behaviour which potentially can provide enhancement towards the composite properties. In this work, rubber waste was obtained from discarded rubber gloves and went through cryogenic grinding to produce crumbs. The glove waste was soaked in liquid nitrogen until frozen and grounded using crusher. The crumbs were later analysed by using physical and thermal characterisation methods. The sizes of the rubber crumbs were determined by using sieving method and ranging from 300 μm to 2 mm. The shape of the crumbs and their surface details were observed by using a scanning electron microscope (SEM) and thermal analysis was conducted by using thermogravimetric analysis (TGA). The microscopy showed the rubber crumbs has regular shapes and smooth surfaces. The thermogravimetric results revealed both waste and fresh rubber crumbs degraded in the same manner. They degrade in three stages and obvious mass loss occurred between 250ºC to 400ºC due to degradation of the main natural rubber element; poly-cis-1,4-isoprene. In comparison to fresh rubber crumb, thermal behaviour of waste rubber crumbs remains the same. Thus, the properties of waste rubber are still similar to fresh rubber crumbs. Consequently, on the basis of properties obtained from this work, the rubber waste crumbs acquired from rubber waste glove shows great potential to be used as a filler for the polymer composites.

KEYWORDS: *Rubber Crumb; Physical Properties; Thermal Properties; Rubber Waste; Cryogenic Grinding*

1.0 INTRODUCTION

Environmental concern has been a never-ending discussion among environmentalist, researchers and scientists. In order to ensure survivability of mankind and life being in the future, numerous, and extensive efforts by all relevant parties have been conducted in order to materialise this greener environment campaign [1]. Increasing volume of rubber waste has become a major threat to the environment globally [1–3]. Rubber waste are discarded rubber products from various sources such as used tyres, manufacturing scraps, rubber pipes, rubber shoe soles and gloves [5–7]. Researchers have conducted numerous studies that involved rubber waste in the composites manufacturing, such as polymer-based composites and ceramic-based composites. From the literatures, it was reported that rubber waste went through crushing, grinding, macerating or pulverising processes in order to obtained smaller sizes of rubber prior added into the composite matrices such as concrete, thermoset, thermoplastic or elastomer matrix. Rubber waste went through size reduction process in order to obtain smaller sizes rubber to be efficiently incorporated into the matrices. The smaller size rubber can be in the form of crumbs, particles, granulates, powder or dust [8-9]. In recent years, rubber crumbs has gained popularity in the market as a filler or reinforcing elements in many applications such as polymer composites, pavement industries and construction materials because of it has many excellent properties such as high elasticity and high absorption capacity [9].

The characteristic of rubber crumbs, particles, granulates, powder or dust can be analysed by using physical and thermal methods [10]. Physical characteristics of these rubbers include the size distribution, shape and surface characteristics. It is important to study the shape and surface morphology as its reflects the surface activity of the crumbs [9]. Various sizes of rubber crumbs ranging from <100 μm to 16 mm has been reported being used in widespread applications such as in corps growth field, asphalt binder modifiers, aggregates in concrete, also being used for oil spill recovery, incorporated in sport turf and replacement for railway ballast [4, 12]. In some literatures has mentioned that, the size of rubber crumbs being incorporated into the composite is in the range from 75 μm to 500 mm [8, 13].

Many methods has been used to produce rubber crumbs from the rubber waste such as ultra-high pressure water jet process, solid state shear extrusion, ambient or cryogenic grinding [10-11]. The cryogenic grinding process is a method of using liquid nitrogen or other materials or techniques that are able to freeze the rubber articles below its glass transition temperature (T_g) like the natural rubber T_g is -70 ºC. Rubber changes from elastic to brittle behave "glass-like" property thus it is able to be ground or crushed to reduce size [13]. Rubber will return to its original properties as its temperature above the T_g. The cryogenic grinding also preserves rubber from heat degradation and maintains its unique properties [9, 15]. The shape of the crumbs can be varied from irregular, regular to spherical shape and their surface can be rough or smooth [9, 11, 14]. Shapes and surface characteristics of the crumbs plays a significant roles for the filler–matrix adhesion that will determine the physical and mechanical properties of the composites [11, 16].

Thermal analysis is one of the techniques that study the behaviour of materials as a function of temperature. Thermogravimetric analysis (TGA) is the common method used for this purpose [16]. It is important to conduct thermal analysis study as the oxidative and thermal degradation of the rubber crumbs are very much influenced by its suitability for the desired application, especially its impact on the products' performance and shelf life [17]. TGA is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. It is due to the effect of degradation and oxidation of the samples [16].

2.0 METHODOLOGY

2.1 Materials

Rubber waste used for this work was the discarded rubber gloves collected from laboratories in Universiti Putra Malaysia. These gloves were made from natural rubber latex with few additives such as curatives, pigments and surfactants. They were washed, rinsed, and dried in a dry and airy environment with the non-direct sunlight. The rubber gloves went through grinding process under the cryogenic condition to produce rubber crumbs. The gloves were cut into chunks and soaked in liquid nitrogen until frozen, after that the frozen gloves were crushed by using an electrical powered rotating sharp metal blade. Figure 1 shows the main processes of producing rubber crumbs.

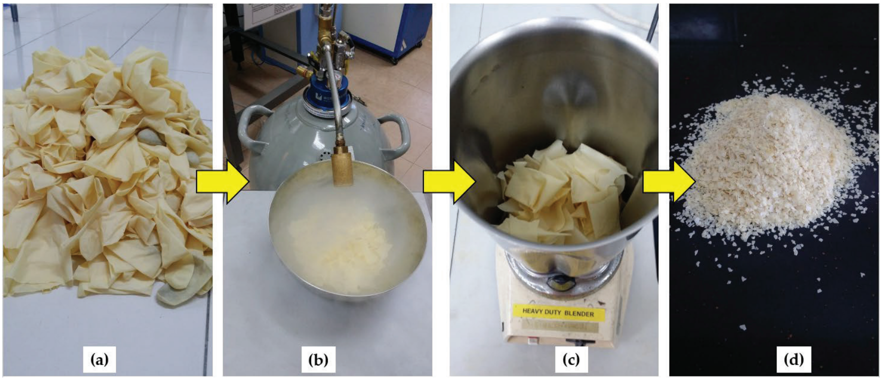

Figure 1: Rubber crumbs preparation: (a) waste rubber gloves were washed and dried, (b) gloves were soaked with liquid nitrogen until frozen, (c) frozen gloves were crushed immediately and (d) rubber crumbs

2.2 Characterisations

The rubber crumbs produced from the cryogenic grinding process were then prepared for several characterisations tests to study its potential as reinforcement material in composites.

2.2.1 Size of Rubber Crumbs

The size of rubber crumbs was determined by an auto sieve shaker that operated mechanically (Figure 2). The crumbs were sieved by using mesh sizes of 300 microns, 450 microns, 600 microns, 850 microns and 2 mm.

Figure 2: Auto sieve shaker

2.2.2 Morphology of Rubber Crumbs

The size measurement, shape and surface morphology of the rubber crumbs were analysed by using JSM-6010Plus/LV Scanning Electron Microscope (SEM) from JOEL (Figure 3). The relevant amounts of crumbs were attached to double sided adhesive tape and mounted on the specimens' stubs. Prior to observation under SEM, the specimens were sputter-coated with platinum in order to create a metal conductive layer to prevent charging of the samples.

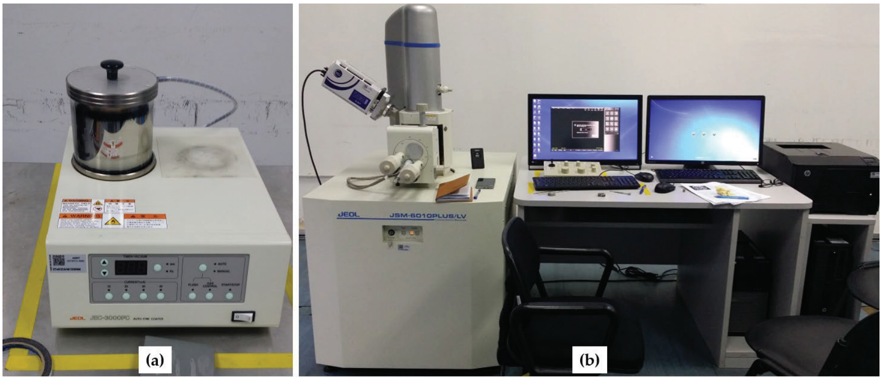

Figure 3: Morphology analysis: (a) sputter coater and (b) scanning electron microscope (SEM)

2.2.3 Thermal Properties

Samples of waste rubber crumbs and rubber crumbs from fresh glove were analysed for their thermal properties composition by using TGA analysis. The purpose of analysing both rubber crumbs are to determine if there are any significant differences or changes occurred in the waste rubber. The TGA analysis was conducted by using TGA Q500 TA Instruments thermal analyser (Figure 4). Sample of 5-10mg were heated from 30°C - 950°C at a nitrogen flow of 20mL/ min at a heating rate of 10°C/min.

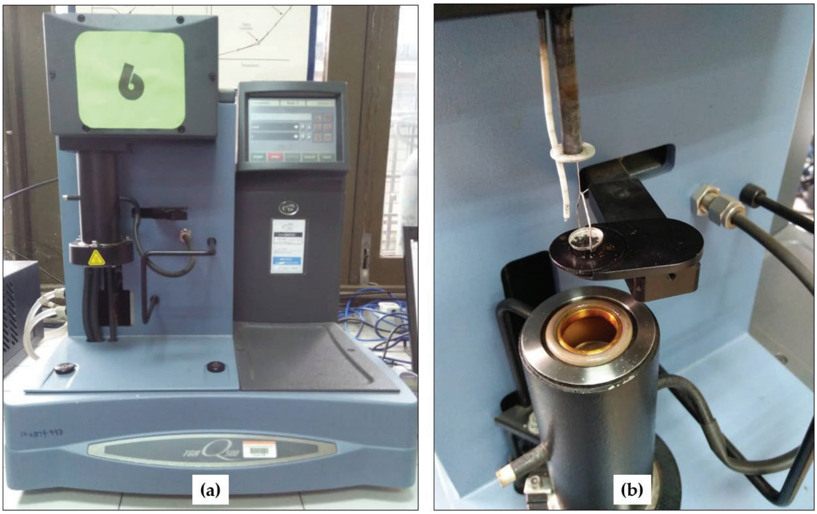
(a)
(b)

Figure 4: TGA analysis: (a) thermal analyser and (b) sample loading

3.0 RESULTS AND DISCUSSION

3.1 The Size of Rubber Crumbs

One of the key factors of producing composites with good physical and mechanical properties is by enhancing compatibilisation between filler and the matrix. Good compatibilisation between these two constituents, improves the filler-matrix adhesion. Hence, resulting better physical and mechanical properties of the composite. Therefore, filler size reduction is one of the main elements in providing better matrix-filler adhesion. Smaller size filler will increase specific surface area of the filler and allowing more area for adhesion with the matrix [18]. Cryogenic grinding has the ability to reduce the size of the rubber and it is capable of producing finer size of rubber crumbs up to 75μm [19]. Grinding rubber below the T_g changed elastic behaviour of rubber to brittle which ensured effective grinding in producing smaller crumbs. As a protective barrier, rubber glove possessed very high elongation and high strength to minimise glove rupture, puncture and tear during use [19–21]. High resistance to rupture, puncture, and tear resulted in the challenge of producing finer rubber crumbs through ambient grinding as highly elastic items such as gloves tend to stick to the grinder blade. The cryogenic condition had caused the rubber becoming brittle like glass. As a result, the brittle rubber was more easy to be ground. The rubber will gain back their elasticity once the rubber crumbs temperature returns to room temperature [7, 23].

The rubber crumbs produced from the cryogenic grinding were in the range of 300 micron to 2mm and approximately 80% of the crumbs were in the range of 600 micron to 2mm. Figure 5 shows the different sizes of the rubber crumbs produced by the cryogenic grinding. This range of sizes were also obtained by other researchers that prepare their rubber crumbs under cryogenic condition as reported by Sienkiewicz et. al [12], Isayev [24] and Presti [25]. In term of incorporation the rubber crumbs into the polymer matrices, it is reported by Abu-Jdayil et al. [26-27] that they have incorporated the rubber crumbs of size ranging from 0.8mm to 2.0mm into the unsaturated polyester which the rubber crumbs they obtained from waste tyres.

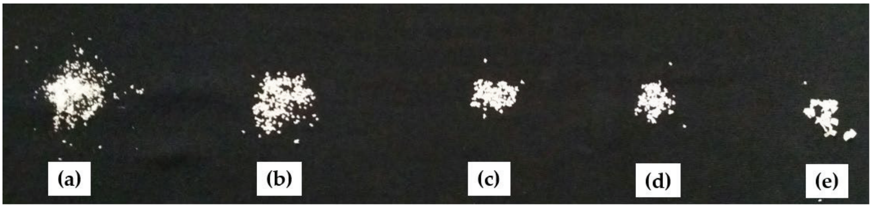

Figure 5: Different sizes of rubber crumb: (a) 300 to 450 μm, (b) 450 to 600 μm, (c) 600 to 850 μm, (d) 850 to 2 mm and (e) above 2 mm

3.2 Shape and Surface Characteristic

The morphology analysis as shown in Figure 6 revealed the shape and surface of the crumbs that produced from the cryogenic grinding. The crumbs were regular in shape and has sharp-angular edges. The fracture surface of crumbs is smooth and clean cut. Smooth fracture surface and sharp-angular edges showed rubber experienced brittle fracture as it embrittled below glass transition temperature during cryogenic grinding process. As discussed in many literatures, cryogenic grinding process produced sharp or edgy end with flat and smooth surface [9, 11, 14, 20, 23]. Adhikari et al. [29] had reported that the cryogenically ground rubber has a smoother surface as compared to the ambient grinding rubber. The smooth surface of the rubber has advantage which it will allows crumbs to disperse well without occlusion in matrix. Zefeng et al. [30] also mentioned in their publication that cryogenic grinding produced angular shape rubber particles with smooth surfaces. Meanwhile, Carli et al. [28] stated that the cryogenically ground rubber has smooth surfaces and spherical shapes. Although many findings mentioned that regular and smooth surface of filler may not be producing stronger adhesion with matrix as compared to irregular and rougher surface crumbs, it can be overcome by modifying the surface of the fillers or the crumbs with compatibiliser [13, 28–30].

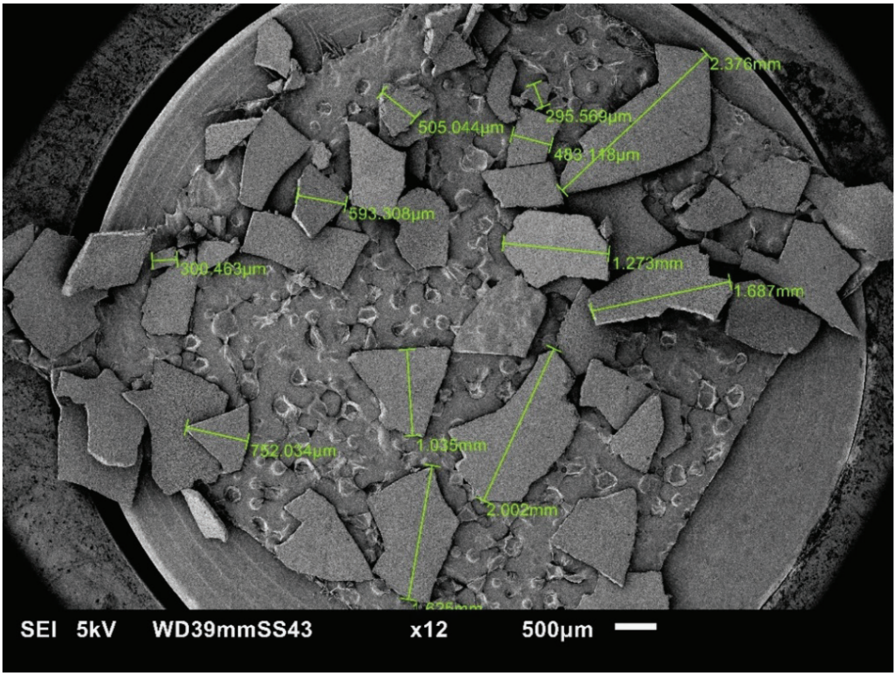

Figure 6: SEM image of rubber crumbs produced from cryogenic grinding

From the literatures, cryogenic grinding produces cleaner crumbs and the crumbs surfaces are free from oxidation unlike ambient grinding. Ambient grinding may cause the rubber heated up to 130°C due to friction which caused oxidation on the rubber crumbs surface [7]. The less heat generation during grinding helps preventing any potential polymer chains degradation in the rubber structure which may degrade its properties [31-32]. Thereby, effort of conducting cryogenic grinding on the rubber glove waste in this study is proven relevant and it offers a simpler method for reducing the size of waste rubber, consumes less energy and produces more desirable rubber crumbs.

3.3 Thermal Properties

The importance of conducting thermal analysis is to determine the thermal degradation of the rubber crumbs. Therefore, the analysis was conducted to find out whether crumbs obtained from waste gloves maintains the properties of crumbs from fresh glove. This analysis is also important in order to understand the thermal stability and heat resistance of crumbs. Hence, it is able to ensure the crumbs can perform well during application and product damage or deterioration can be prevented. Also, this information helps to define the level of temperature that rubber crumbs able to withstand during manufacturing and service. The thermogravimetric curves of weight loss for both of waste and fresh rubber crumbs are shown in Figure 7.

Three main steps were observed for both samples of rubber crumbs which represent crumbs degrade in 3 stages. The first weight loss occurred between 250°C and 400°C, with degradation starting at approximately 250°C. The weight loss was due to the degradation of main organic components in natural rubber which are poly-cis-1, 4-isoprene, protein and also some minor substances present in the glove's formulation [16, 26]. The degradation continued from 400°C to 700°C and the weight loss due to the degradation of subsequent rubber components and inorganic additives at temperature around 700°C [16, 23, 26]. According to Carli et al. [28], in their findings the weight loss occurred at 754°C due to the decomposition of inorganic elements such as $CaCO_3$ which released CO_2. The final steps between 700°C to 950°C were attributed by mass loss happened due to the decomposition of ashes and carbonaceous residue either from the earlier thermo-oxidation process of organic substances or elements that already present in the rubber.

The derivative curves as shown in Figure 8 represent derivative of the mass as a function of temperature yielded from weight loss activities. The first and the highest peak that presented in this curves as an evidence of sample degradation occurred from temperature 250°C to 700°C and most degradation of substances happened during this stage. The two curves at about 375°C and 700°C respectively has verified that the first two steps from thermogravimetric curves resulting from the combustion of the elements in the glove's ingredients. It can be seen that the first main step of the sample degradation in the thermogravimetric curves yielded a highest peak in the derivative curves. It confirmed that the first main step of weight loss happened between 250°C to 400°C. This is the temperature where most of the organic elements in rubber degrade whereby; the greatest thermal decomposition occurred at about 375°C. Approximately 75% of the substance degraded within this temperature range and the weight loss and volatilisation of the degradation products occurs rapidly during this stage. Similar finding was also discovered by Mathew et al. [34], they reported that the natural rubber starts degrade at 290°C and their derivative curve has a major peak at 364°C. They also reported that about 85% of the material was degraded during this stage. Meanwhile, work that has been conducted by Causin et al. [16] have revealed that the organic elements in rubber such as poly-isoprene, proteins, aldehydes, ketones and carboxylic acids experienced decomposition at temperature around 375°C. The smaller peak at around 700°C confirmed the subsequent degradation of these rubber substances such as inorganic elements in the glove as previously displayed by the thermogravimetric curves.

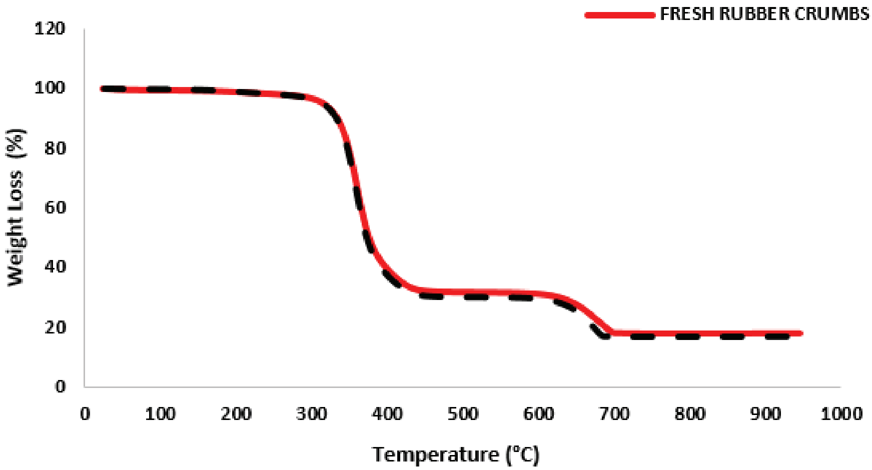

Figure 7: Thermogravimetric curves of fresh rubber crumbs and waste rubber crumbs

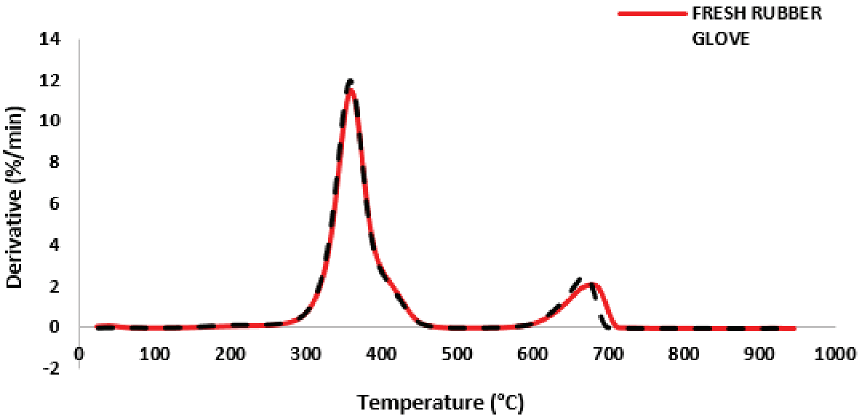

Figure 8: Derivative curves of fresh rubber crumbs and waste rubber crumbs

The thermogravimetric and derivative curves of waste rubber shows insignificant change as compared to fresh rubber which certify the waste rubber remains its elastomeric properties. These properties are vital to ensure the incorporation of waste rubber glove crumbs able to provide elasticity properties to the composite. Besides that, thermal analysis helps to define the thermal stability of composite as the size of rubber crumbs will play an important role in ensuring good thermal properties. Incorporating smaller rubber crumbs in the composite provides better thermal stability compared to larger crumbs. Smaller crumbs has higher surface area with high surface energy that allows good crumbs-matrix adhesion [35]. Good crumbs-matrix adhesion resulting stronger mechanical and physical properties of composites. Thus, composite with smaller rubber crumbs will have better thermal stability that able to serve well in various application.

4.0 CONCLUSION

Physical and thermal characterisations of rubber waste glove crumbs help in identifying the potential of rubber crumbs to be incorporated in the polymer composites. Cryogenic grinding is able to produce a smaller size of rubber crumbs that are suitable to become filler for the composites. Crumbs sizes obtained from cryogenic grinding were ranging from 300 μm to 2 mm. The small size of crumbs offers a higher surface area that could enhance filler-matrix adhesion. Good filler-matrix adhesion resulting better physical and mechanical properties of the composites. Cryogenic grinding produced crumbs with regular shape and smooth surface that allows a homogenous mixture of rubber crumbs and matrix. Although it might lessen the ability to adhere well to the matrix, it can be improved with surface treatment. The thermal properties analysis provides information of the waste rubber glove crumbs start to degrade at 250ºC and noticeable weight loss continues until the temperature is 400ºC. The weight loss, is mainly due to the decomposition of poly-cis-1,4-isoprene, the natural rubber main element. Thermal properties of the rubber waste crumbs show no changes as compared to fresh rubber which ensuring its elastomeric properties remains. Based on the results obtained, characteristics of rubber waste crumbs showed that they have huge potential as a filler for the polymer composite.

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