Wood Plastic Composites Produced from Postconsumer Recycled Polystyrene and Coconut Shell: Effect of Coupling Agent and Processing Aid on Tensile, Thermal, and Morphological Properties

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Expanded polystyrene (EPS) has been widely used as a disposable packaging material in many industries thanks to properties like low density, lightweight, high impact, and vibration damping. Although usage of EPS increases annually, recycling facilities often refused to process post-consumed EPS due to the poor economic viability associated with high logistics and transportation cost in collection, storage, and shipment of the material. The objective of this research is to enhance the value chain of postconsumed EPS by investigating its potential as feedstock in the development of sustainable wood plastic composites (WPC), thereby providing an attractive business opportunity that also increases interest in EPS recycling and indirectly continue the lifespan of disposed EPS. Varying compositions of recycled polystyrene (rPS), coconut shell (CS), maleated polystyrene (MAPS) and Ultra-Plast WP516 were compounded using a HAAKE internal mixer and compression molded to form WPC. The effects of material formulation on mechanical, thermal, and morphological properties of the composites were studied. The experiment showed that WPC formulated with 100 phr of rPS, 30 phr of CS, 3 phr of MAPS, and 1 phr of Ultra-Plast WP516 possesses higher modulus and tensile strength compared to the neat EPS, measured at 2.5 GPa and 27.5 MPa, respectively. Although the WPC experienced initiation of thermal degradation at a temperature lower than neat rPS, but the thermal stability of rPS/CS composites containing varying composition of MAPS and Ultra-Plast WP516 was better at high temperature. Furthermore, a 50% weight loss took place at a higher temperature. Nevertheless, the glass transition temperature of the rPS/CS composite with addition of MAPS and Ultra-Plast WP516 was found lower than the neat rPS. POLYM. ENG. SCI., 00:000–000, 2019. © 2019 Society of Plastics Engineers

INTRODUCTION

Wood plastic composite (WPC) is defined as composite materials having fine wood particles mixed together with thermoplastics like polyethylene and polypropylene. Besides fine wood, WPC can also contain other lignocellulosic fillers [1]. The global demand for WPC is increasing steadily, from a market value of about $3.3 billion in 2014 to an expected $6 billion in 2020 [2, 3]. The steep market growth of WPC can be attributed to a number of key stimulating factors, including but not limited to the high stiffness-to-density ratio, lower production cost, abundant availability of natural fillers from agricultural waste, green credential especially in reducing the logging and saving the forest, as well as the limitless customizable finishes and aesthetic appearance of WPC, that has vastly replaced usage of natural solid wood in furniture, decking, and so on [4–6].

In the past, fine wood has been traditionally used as a filler in the production of WPC. In recent years, numerous types of natural fillers recovered from agricultural wastes such as rice husk, kenaf fiber, wood flour, and coconut shells (CSs) have gained intent research interest as an alternative material for fine wood [7–10]. Found in the tropical regions, coconut (Cocos nucifera) is an important crop widely planted in countries like India, Malaysia, Indonesia, Thailand, Sri Lanka, Philippines and Brazil [11–14]. In the coconut industry, coconut water is sold as a favorite refreshing beverage while the tender kernel can be either eaten fresh or used to extract coconut milk and coconut oil [10]. However, the coconut husk and shell are commonly discarded as unwanted by-product from the industry, making up to about 3.18 million tons of CS residue annually [15]. In the past, there have been approximately 90% of CSs either sent to landfills or burned in open air. This has contributed to CO2 and methane emission [11]. In efforts to lessen the social and environmental impacts associated with the coconut industry, recent scientific research have reported the potential of converting wastage from coconut husks and shells to produce phenolic adhesive, charcoal, and activated carbon [16, 17] as well as fillers in WPC development [18, 19]. The current study further contributes to the green credential of the coconut industry by incorporating CS as key filler in the fabrication of WPC.

Expanded polystyrene (EPS) is a material that consists of nearly 98% air and 2% plastic [20]. The low cost, lightweight, and low density characteristics of EPS makes it a material of choice in various applications, for example, food packaging containers, protective cover for domestic appliances, consumer electronics and household devices that are commonly utilized for...
single-use. Furthermore, applications include sandwiched structured sport utility with high flexural rigidity and impact properties; as well as building materials with good thermal and acoustic insulation in construction [21, 22]. Packaging application represents a large proportion of EPS utilization and since it is for single use purposed, the EPS foam will be disposed by consumer as a municipal waste after serving its intended purpose or other end-use applications. Despite the low weight, it has been reported that about 3 million tons of EPS foam wastes are generated annually in the world [23, 24]. Most of these EPS foam wastes are dumped into landfills and contribute to environmental pollution as it is nonbiodegradable [25, 26]. Additionally, the lightweight EPS can be easily carried away in the wind or by water, turning them into harmful oceanic pollutant as consumption of polystyrene (PS) by wildlife can be fatal. Although disposed EPS is an imminent environmental issue, there is still a lack of waste isolation and collection policies on EPS as most recycling facilities are reluctant to recycle PS foam wastes. These solid, bulky, and lightweight structures take up tremendous storage space and transportation is very costly [27]. Hence, EPS foam wastes are specifically targeted in the current research to investigate and assess its efficacy as a matrix for WPC, which contributes to the reduction of EPS foam wastes, provide cost advantage on WPC materials and attract greater interest in the recycling of disposed EPS.

The production of WPC is complicated due to incompatibilities between hydrophilic natural fillers and hydrophobic plastic matrices that cause poor interfacial adhesion and result in low mechanical strength [28]. To combat such deficiency, various types of plastic additives have been used to enhance the properties of WPC. For instance, coupling agents, for example, maleated polyethylene and silane are used to improve filler–matrix adhesion [1] whereas lubricants and processing aids are added to improve the filler dispersion and processability of WPC [29]. Processing aid typically is polyolefin-based compound designed to improve polymer melt flow and processability of the materials. The coupling agent used in this study was maleated PS (MAPS) while the processing aid used was Ultra-Plast WP516 (Sabak Bernam, Malaysia). The CS particles were predried in oven maintained at 70°C prior to compounding. The MAPS coupling agent was supplied by Sigma Aldrich Malaysia (Selangor, Malaysia). Melt flow index of MAPS was 21 g/10 min (230°C/5.0 kg) and molecular weight of 1,600. The processing aid (Ultra-Plast WP516) was supplied by Performance Additives Sdn. Bhd. (Subang Jaya, Malaysia). The melting temperature of Ultra-Plast WP516 was 145°C. Both MAPS and Ultra-Plast WP516 were used as received.

Preparation of rPS

The collected PS foam wastes were manually cut into smaller size ranging within 4–2 cm and transferred to oven maintained at constant temperature of 130°C. The PS foam waste shrunk approximately 95% in size upon a duration of 15 min within the oven, signifying expulsion of entrapped gas from the EPS (as shown in Fig. 1). The rPS was stored in sealable plastic bag and ready to be used for producing WPC. The density of rPS was 0.98 g/cm³ and melt flow index was 12.8 g/10 min (200°C/5 kg).

Preparation of Composite Samples

The preparation and process flow of rPS/CS composite fabrication is displayed in Fig. 1. The rPS/CS composites were compounded using HAAKE Rheomix 600p internal mixer (Thermo Fisher Scientific Inc.). The composites were prepared according to the formulation listed in Table 1. Processing temperature and rotor speed were set at 190°C and 80 rpm respectively. The compounding procedures are: (1) weighted composition of premixed rPS and MAPS/ Ultra-Plast WP516 was introduced into the mixing chamber and compounded for 1 min until the mixtures completely molten; (2) after 1 min, weighted amount of CS particles was added into the mixing chamber and further compounded for 9 min to achieve homogeneous dispersion of the CS particles; and (3) the formed composite compound was removed from the mixing chamber and ready for molding process.

The rPS/CS composites were then compression molded into a square sheet of 15 × 15 cm² with 2 mm in thickness. The hot press machine used was Moore model (Kaohsiung, Taiwan) at the temperature of 190°C and pressure of 100 kPa, respectively. The rPS/CS composite sheets were then cut into tensile dumbbell Type I specimen with dimensions in accordance with ASTM D638. The molding procedures were to: (1) preheat the compound for 2 min until it softens; (2) fully compresses at set pressure for 1 min; and (3) cool the molded sheet for 20 min prior to removal from the mold.

Test Methods

The tensile tests were carried out by an Instron universal testing machine (model 5596; Norwood, MA) in accordance with standard test method ASTM D638. The crosshead speed was set to 5 mm/min. A specimen was tested at room temperature with 15 kN load cell. The applied force and displacement of the specimens were recorded by the Bluehill 3 software. The minimum number of specimens tested out for every formulated composite was 7. The tensile strength and modulus of the composites were analyzed and discussed.

The morphological analysis was conducted using scanning electron microscope (SEM, model: FEI Quanta 400, Hillsboro). This analysis was used to study the filler–matrix adhesion as well
as fracture behavior of the composites. The acceleration of the electron was set at 15 eV. Cross section of the selected tensile fractured surface was coated with a thin layer of palladium before analysis.

 Thermogravimetric analysis (TGA) was conducted using Pyris Diamond TGA (PerkinElmer, Waltham, MA) to determine the thermal degradation behavior of the WPC. Specimens were cut in small size weighing between 5 and 8 mg and placed into a ceramic pan. The specimens were then heated from 30 °C to 600 °C at a heating rate of 10 °C/min. TGA was carried out under nitrogen atmosphere utilizing gas flow rate of 20 mL/min.

 The thermal transition of the WPC was analyzed with the aid of differential scanning calorimetry, (model DSC 4000 PerkinElmer). The specimens were cut into small size weighing between 5 and 8 mg and placed in an enclosed aluminum pan.

### RESULTS AND DISCUSSION

#### Tensile Properties

One cycle of the heating and cooling in the range of between 30 °C and 120 °C was carried out to remove the thermal history of specimen. Upon completion of the initial heating–cooling cycle, the specimen was heated up from 30 °C to 150 °C and glass transition temperature ($T_g$) of specimen was recorded. The heating and cooling were set at 10 °C/min. The analysis was carried out under nitrogen atmosphere with the gas flow rate of 20 mL/min.

#### TABLE 1. Formulation of rPS/CS composites.

<table>
<thead>
<tr>
<th>Samples</th>
<th>CS (phr)</th>
<th>rPS (phr)</th>
<th>MAPS (phr)</th>
<th>Ultra-Plast WP516 (phr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>rPS/CS</td>
<td>30, 60</td>
<td>100</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>rPS/CS/MAPS</td>
<td>30, 60</td>
<td>100</td>
<td>3</td>
<td>—</td>
</tr>
<tr>
<td>rPS/CS/MAPS/WP516</td>
<td>30, 60</td>
<td>100</td>
<td>3</td>
<td>1, 3, 5</td>
</tr>
</tbody>
</table>

The load accuracy of Instron machine used for tensile test is ±0.5%, thus the potential errors from the machine is very little and it can be negligible. The tensile strength of neat rPS and rPS/CS composites is shown in Fig. 2a. In the absence of a coupling agent and processing aid, the tensile strength of rPS/CS composites decreased with higher loading of CS content. The addition of 60 phr of CS content measured an average of 55.2% reduction in tensile strength compared to the neat rPS. The reduction of the strength usually due to the following reason: (1) poor adhesion between filler and matrix; (2) geometrical factor of filler; and (3) low aspect ratio [6]. In this case, the CS used was largely in particle form and irregular in shape. These particle fillers typically have lower aspect ratio compared to fiber. As a result, the CS particles have lower ability to act as a load bearing component
in the composite owing to their lower effectiveness to facilitate stress transfer from matrix to fillers. The rPS/CS composite that was exhibited had approximately 28% lower tensile strength than rPS/durian husk fiber composite where both composites had 60 phr of filler content [27]. The comparison shows particles filler has lower load bearing capability compared to short fiber. In addition, the incompatibility between nonpolar rPS matrix and polar CS filler could be another reason for poor adhesion at their surface interfaces. Hence, the poor interfacial bonding between the CS and rPS matrix results in inferior tensile strength of the composite. This result can be further illustrated through the scanning electron micrograph observations as discussed in the section pertaining to morphological studies. Similar findings have also been reported in composites with CS as filler [18, 19].

The addition of 3 phr of MAPS coupling agent is capable of enhancing the surface adhesion between the rPS matrix and CS filler, where the tensile strength of the composite was approximately 16.5% greater than those without MAPS. The maleic anhydride carbonyl groups from MAPS are highly reactive and capable of forming ester bonds with hydroxyl groups of the CS filler, a mechanism that allows long PS chain from MAPS to chemically attached onto surface of CS filler. In addition, the PS chains of MAPS facing away from CS filler capable of forming an “adhesive bridge” between CS filler and rPS matrix through physical chains entanglement, thereby increasing the interfacial adhesion between CS filler and rPS matrix. The greatest tensile strength was achieved with 30 phr CS fillers, 3 phr of MAPS, and 1 phr of WP516. The greater tensile strength of composite with 30 phr CS fillers may be attributed to the more uniform filler dispersion. The excessive loading of CS fillers at 60 phr potentially induced particles agglomeration and resulted in lower particle surface-to-volume ratio for effective stress transfer. Thus, the addition of more CS fillers reduced the tensile strength of the composite. Figure 2b demonstrates the tensile modulus of neat rPS and rPS/CS composites. Therefore, the tensile modulus of the composites increased with the filler content. When compared to the neat rPS, the tensile modulus was about 29.2% higher with 60 phr of CS fillers. A similar trend was observed by Pao and Chan [31] where higher amounts of CS particles were added into rPS/CS composites that
showed an increase in the tensile modulus. With 3 phr of MAPS coupling agent, the tensile modulus was found to further increase to approximately 10.9% higher than those without MAPS. Since the CS particles are stiffer and more rigid than plastic materials [13]. The addition of MAPS enhanced interfacial adhesion between rPS and CS. Thus, the addition of CS often increased the modulus of the composites. Besides, it is postulated that the presence of CS particles tends to introduce restriction to chain mobility of the rPS matrix, which reduces the efficacy of chain slippage and disentanglement. Hence, this causes the composite to become more brittle but with greater modulus.

In the present study, different concentrations of Ultra-Plast WP516 have also been incorporated to the rPS/CS composite with MAPS. The effect of varying Ultra-Plast WP516 content on tensile strength and modulus of rPS/CS/MAPS composites are shown in Fig. 4. The result showed that optimum tensile strength and modulus were achieved with 1 phr of Ultra Plast WP516. As the Ultra-Plast WP516 processing aid used was a fatty acid amine compound, the amine group was capable of establishing a hydrogen bond to the CS filler surface leaving fatty acid tails facing away from the filler [29]. At 1 phr, the fatty acid amine-based compound might attach onto the CS surface as illustrated in Fig. 5a. Besides, the presence of both MAPS and Ultra-Plast WP516 would improve filler–matrix bonding as well as filler dispersion. However, the Ultra-Plast WP516 content above 1 phr adversely affected and decreased the tensile strength and modulus of the composites. This is because part of the excessive Ultra-Plast WP516 might not be able to find their way and attach to the filler surface due to the reason that a majority of the filler surfaces had fully bonded with MAPS and Ultra-Plast WP516. These excess Ultra-Plast WP516 most likely diffused and intercalated between the rPS matrix and MAPS (Fig. 5b) and imposed a lubrication effect that inhibited the rPS chains from forming effective entanglement with the PS chain of the MAPS. Then, it weakens the interfacial bonding between the CS filler and rPS matrix. As a result, the tensile strength and modulus of composites were significantly reduced.

Morphological Properties

The SEM micrographs of fractured surface of the rPS/CS and rPS/CS/MAPS composites are illustrated in Fig. 6. All SEM micrographs showed brittle fracture with faceted surface nearly perpendicular to the direction of the applied loading. Pronounced holes or voids can be observed in rPS/CS composites with 30 and 60 phr of filler contents, Fig. 6a and b, respectively. The number of holes was more severe in composite with 60 phr filler content, as depicted in Fig. 7b. The presence and number of holes can be linked to the ease of CS filler detachment (pullout) from the rPS matrix under external tensile load, which signifies weak interfacial bonding between CS particles and rPS matrix. For the rPS/CS composites with MAPS coupling agent, most CS particles were embedded and well covered by the rPS matrix, as shown in Figs. 6c and d. This can be attributed to the addition of MAPS that have enhanced the filler–matrix bonding at the interfacial region. These observations proved that strong interfacial bonding was established between the CS filler and rPS matrix, which supported the findings in tensile properties of the composites reported in the earlier section of this article.

Figure 7 presents the SEM micrographs of fractured surface of rPS/CS/MAPS composites with different concentrations of Ultra-Plast WP516 processing aid. The SEM micrographs of the composites revealed more occurrences of matrix tearing with increased loading of Ultra-Plast WP516 from 1 to 5 phr. The observations suggest that the addition of Ultra-Plast WP516 imposed a lubricating effect on the composites at concentrations higher than 1 phr and tends to become more severe at higher concentrations.
content. At 1 phr of Ultra-Plast WP516 and 3 phr of MAPS, the SEM micrograph showed that the CS particles were well embedded within the rPS matrix, Fig. 7a. This finding showed evidence that 1 phr of Ultra-Plast WP516 yielded the optimum tensile strength and modulus of the rPS/CS/MAPS composites. In contrast, numerous fillers pull out and CS particles detachment found in Fig. 7b and c that further proved that addition of excess Ultra-Plast WP516 tend to weaken the interfacial bonding between the filler and matrix.

**Thermal Properties**

For this TGA equipment, the accuracy of balance of equipment is ±0.2% and the temperature precision is ±2°C. Hence, the potential error from the equipment is insignificant. Figure 8a displays the TGA curves of rPS/CS and rPS/CS/MAPS composites with selected filler content, neat rPS, and CS. The temperature at 50% weight loss ($T_{d50\%}$) and char residue at 600°C are recorded in Table 2. It can be seen from Fig. 8a that the CS endured
was due to the removal of moisture and volatile compounds. The major weight loss of CS can be attributed to the thermal degradation of lignin, hemicellulose, and cellulose. At higher temperature, the thermal degradation of lignin yielded high amount of char residue [10, 18, 28]. For the rPS/CS composites with 30 and 60 phr of CS filler content, the composites started to thermally degrade at temperature above 260°C. When compared to neat rPS, both rPS/CS composites encountered earlier thermal degradation due to the addition of CS. Nevertheless, the rPS/CS composites showed better thermal stability than neat rPS at temperature above 400°C, where the $T_{d50\%}$ of rPS/CS composites were higher, at approximately 430°C while the $T_{d50\%}$ of neat rPS was measured at approximately 407°C. On the other hand, higher CS content in the rPS/CS composites resulted in much greater char residue. This suggested that higher presence of CS filler leads to formation of more char residue which provide a certain degree of thermal shielding effect and delayed the thermal degradation process of the remaining composites. Hence, the composites show higher thermal stability at temperature beyond 400°C, the temperature where neat CS produced pronounced char residue. This finding was similar to research works reported earlier on CS filled composites with different type of matrices [10, 18]. The results also showed that rPS/CS composites containing MAPS have slightly higher $T_{d50\%}$ and more char residue, indicating that the presence of MAPS surrounding the CS particles formed a coating layer that increased the thermal stability of the composites. The current findings agree with previous studies that reported the maleated copolymers were able to provide a better thermal protecting layer on the filler surface and led to better thermal stability [25].

The TGA curves of neat Ultra-Plast WP516 and rPS/CS/MAPS composites with various Ultra-Plast WP516 are shown in Fig. 8b. The results demonstrated that neat Ultra-Plast WP516 experienced initiation of thermal degradation at temperature above 220°C and its thermal stability was obviously lower than the composites. For composite that had an addition of Ultra-Plast WP516, the $T_{d50\%}$ showed a slight decrease to a lower temperature, from 430.5°C at 1 phr to 427.6°C at 3 phr of Ultra-Plast WP516. Owing to the lower thermal stability of neat Ultra-Plast WP516, the addition of more Ultra-Plast WP516 would slightly reduce the thermal stability of the rPS/CS/MAPS/WP516 composite. Nevertheless, the $T_{d50\%}$ rPS/CS/MAPS/WP516 composite demonstrated better thermal stability compared to rPS/CS composite without additives. The char residue of rPS/CS/MAPS/WP516 composite did not show any significant changes with the content of Ultra-Plast WP516.

The accuracy of this DSC equipment for scanning $T_g$ of polymer is ±0.3°C, thus the results obtained using heating rate of 10°C/min is within the limit of acceptable experimental error. The DSC curves of neat rPS, rPS/CS, and rPS/CS/MAPS composites at different filler contents are shown in Fig. 9a. The glass transition temperature ($T_g$) of samples is tabulated in Table 3. It was found that an increase in CS content slightly increased the $T_g$ of the rPS/CS composite. This is because the addition of CS particles restrained the chain mobility of the rPS matrix. The $T_g$ of rPS/CS/MAPS composites was lower than the rPS/CS composites. The MAPS is a low-molecular-weight copolymer and its $T_g$ was lower than the neat rPS. Hence, introducing MAPS reduced the $T_g$ of...
For the rPS/CS/MAPS composites with various contents of Ultra-Plast WP516, the DSC curves are as displayed in Fig. 9b and data in Table 3 reveal that the $T_g$ of the rPS/CS/MAPS composite decreased with increasing Ultra-Plast WP516 content. This could arise due to the lubricating effect of Ultra-Plast WP516. Lee et al. [29] also found that the addition of excess fatty acid-based compound would cause lubricating effect on composite materials.

CONCLUSIONS

Although global concerns in society responsibility and sustainability towards a circular economy have induced intense researches on ways to eliminate landfill and minimize wastes; however, there have been generic lack of published research work tackling on the reuse and recycling of PS foam. The current work demonstrated an economical and yet sustainable way of extending the lifecycle of postconsumed PS foam waste as binding matrix for agricultural waste CS filled composite. The key findings of MAPS coupling agent and processing aid on the properties of varying formulation of CS/rPS composite are summarized. The tensile strength of rPS/CS composite was lower than the neat rPS, but the tensile modulus of the composite was found better than neat rPS. The increase of CS filler content from 30 to 60 phr in rPS/CS composite resulted in lower tensile strength but higher tensile modulus. The addition of 3 phr of MAPS significantly increased both the tensile strength and modulus of the composites. However, the tensile strength of rPS/CS/MAPS composite was still lower than neat rPS. Optimum content of Ultra-Plast WP516 for rPS/CS/MAPS composite was 1 phr. The rPS/CS/MAPS composites with 1 phr of Ultra-Plast WP516 had a higher tensile strength and modulus compared to rPS/CS/MAPS composites without Ultra-Plast WP516. The rPS/CS/MAPS/WP516 composite at 30 phr of CS content showed higher tensile strength and modulus than neat rPS. In addition, all composites showed earlier thermal degradation when compared to neat rPS due to the presence of volatile, hemicellulose, and lignin in CS. Thermal degradation of lignin yielded high amount of char residue, this char residue provided thermal shielding and delayed the thermal degradation of the remaining composites that increased its thermal stability at higher temperatures. The presence of MAPS slightly improved thermal stability of rPS/CS/MAPS composite and contributed to higher char residue. However, the increase of Ultra-Plast WP516 content reduced the thermal stability of the composites without any significant changes in char residue. Although the $T_g$ of rPS/CS composites became higher when added with more CS content, the inclusion of MAPS and Ultra-Plast WP516 reduced the $T_g$ of the composites. The rPS/CS/MAPS/WP516 with 30 phr of CS content showed lower $T_g$ than neat rPS. The addition of MAPS and Ultra-Plast WP516 showed a significant improvement on the tensile and thermal properties of rPS/CS composite.

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REFERENCES


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<tr>
<th>Samples</th>
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<td>rPS</td>
<td>105.4</td>
</tr>
<tr>
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<td>90.3</td>
</tr>
<tr>
<td>rPS/CS:100/30</td>
<td>107.1</td>
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