The influence of wood flour particle size and content on the rheological, physical, mechanical and morphological properties of EVA/wood cellular composites

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A B S T R A C T

Cellular composites reinforced with vegetal fibers are an emerging class of materials combining good mechanical properties with reduced density and superior impact energy absorption, as well as thermal and acoustic isolation compared to other composites. This research aims to investigate the effects of different particle sizes and contents of wood flour (WF) on the properties of cellular poly(ethylene-co-vinyl acetate) (EVA)/WF composites. The cellular composites were foamed in a heat press using azodicarbonamide as blowing agent. The results indicate that decreasing the particle size of WF increases the viscosity of the composite, which restricts the expandability of the composite. The presence of WF in the cellular composite increases the nucleation of cells, providing a larger number of smaller cells with increased filler content. Optimal homogeneity was observed with WF B (100–150 mesh), but the highest mechanical properties of tear strength were observed with WF C (150–270 mesh).

1. Introduction

Wood plastic composites (WPC) represent an emerging class of materials that combine the favorable performance and cost attributes of both wood and thermoplastics [1]. These composites have been gaining increasing degrees of acceptance because of their favorable properties, such as lower cost, improved stiffness, lower density, lower abrasiveness and better processability compared to other fillers, such as inorganic fibers [2].

Although WF is gaining increasing acceptance as a reinforcement filler due to its significantly properties, the applicability of WPC is limited due to their lower ductility, reduced impact resistance, and higher density in comparison with those unfilled thermoplastics and natural wood [3]. Reducing the weight of WPC is another challenge for these materials. The density of WPC is almost two times that of solid lumber.

The central idea behind producing a cellular composite is to create a large number of bubbles or voids from a single-phase composite-gas mixture by introducing a blowing (foaming) agent during processing. The concept of creating cellular foamed structures has been shown greatly to reduce the weight, improve the impact resistance, and improve the thermal and acoustic insulating properties of cellular WPC [4,5].

Polymeric composites are materials consisting of two or more phases; usually, the polymer is the continuous phase (matrix), and the fiber is the reinforcing phase [6]. In cellular polymer composites or expanded polymeric composites, there is a third phase referring to the voids (cellular structure) created by the blowing agent in the polymer matrix, which are called cells [7].

The basic mechanisms involved in the production of cellular WPC are usually associated with the incorporation of a blowing agent during composite processing by extrusion, injection or a batch process. The blowing agent is dissolved or finely dispersed in the polymer, after which a large number of bubble nuclei are generated such that they are uniformly dispersed in the polymer matrix. Bubble nucleation is followed by bubble growth due to the diffusion of the dissolved or evolved gas into these nuclei and, finally, by stabilization of the grown bubbles [2]. The blowing agent can be physical, such as liquids with low boiling points or solid chemical compounds that decompose at processing temperatures, thus liberating gases, such as CO₂ and/or nitrogen [8].

In most cases, the addition of WF in cellular composites resulted in narrower cell size distributions and lower average cell dimensions. Since the introduction of a solid phase in the foaming process creates sites of lower surface energy, wood particles are
believed to act as nucleating agents that enhance heterogeneous nucleation [9–11].

The surface area of WF particles varies for different particle sizes, which can have effects on the viscosity, the cell nucleation, and the cell expansion behavior during foaming of WPC. Conversely, the surface area may also have an effect on the amount of volatiles generated during processing, which, in turn, affects all of the earlier mentioned parameters, and not necessarily synergistically. However, notably few studies can be found concerning the effects of WF particle size on the foaming of WPC in the literature [12].

Most published works relate to rigid expanded composites using polymeric matrices, such as polypropylene (PP), poly(vinyl chloride) (PVC) and high-density polyethylene (HDPE). However, there are few references in the literature that use poly(ethylene-co-vinyl acetate) (EVA) to produce cellular composites. One of the most commonly used polymers in the manufacture of polymeric foams is EVA, a polymer that combines good mechanical properties and high flexibility with elastomeric characteristics, which can contribute to the production of a cellular composite with these characteristics. Cellular composites of EVA/WF, even partially crosslinked, are recyclable and have easy processability using various forming methods, such as hot press, batch process, injection and extrusion molding. Those composites may have huge potential for application in footwear products and/or construction industry. Given the shortcomings mentioned above, the objective of this study is to evaluate the influence of the size and content of wood flour on the properties of cellular composites of EVA.

2. Materials and methods

2.1. Materials

Poly(ethylene-co-vinyl acetate), grade EVA 1825 with 18.8% vinyl acetate, was provided by Quattor S.A. (Brasken, S.A.). The coupling agent PEGMA, grade Polybond 3029, was provided by Chemtura Corp. The chemical blowing agent azodicarbonamide (ACA), activated with zinc oxide (ZnO), was provided by Inbra Industria Química Ltda. The crosslinking agent (dicumyl peroxide), grade DCP 40 SAP, and the lubricant (Lub), grade Retiflux, were supplied by Retilox Química especial Ltda. The crosslinking agent PEgMA, grade Polybond 3029, was provided by Chemtura Corp. The chemical blowing agent azodicarbonamide (ACA), activated with zinc oxide (ZnO), was provided by Inbra Industria Química Ltda. The crosslinking agent (dicumyl peroxide), grade DCP 40 SAP, and the lubricant (Lub), grade Retiflux, were supplied by Retilox Química especial Ltda. The WF was collected from the stem of a Eucalyptus dunnii tree. The wood was previously dried in an oven at 70 °C for 5 h and milled in a MARCONI, model MA 580 knife mill with different sieves sizes. The wood samples were separated in a particle size classifier to particle sizes of: WF1 65–80 mesh (0.230–0.177 mm); WF2 80–150 mesh (0.177–0.1099 mm) and WF3 150–270 mesh (0.099–0.053 mm).

2.2. Composite preparation

The samples abbreviations are described in Table 1. The compatibilizer agent PEGMA was added to EVA (2% w/w) using a SEIBT model ES35 single screw extruder, L/D 20 with a temperature profile of 90, 120, 140 and 140 °C and a screw speed of 60 rpm. The additives and fillers were incorporated into EVA + PEGMA using an open roll mill developed by the University of Caxias do Sul (UCS), preheated at 85 °C in the front cylinder and 65 °C in the back cylinder to prevent the adhesion of the band to the back roller. The mixing process entailed sequentially adding each formulation component in the following order: EVA + PEGMA (100 phr), WF, lubricant (1 phr), ZnO (1 phr), ACA (2 phr) and DCP (2 phr). The duration of the mixing step lasted between 10 and 12 min.

After mixing, the composite was formed into preforms using a thermal press SCHULZ at 115 °C with 5 tons of pressure for 5 min using a 140 × 160 × 3 mm mold. The average mass of the preform was 80 ± 2 g. The size and dispersion of the wood particles in the matrix composite can be seen in Fig. 1, which corresponds to the micrograph obtained by the MO of the surface of the unfoamed preform composite under study with 30 phr of wood.

The expansion process for the expanded composite (e-EVA/WF) occurred in a heated press. The preform was placed in a 150 × 170 × 12 mm mold for free expansion in the absence of applied pressure at 175 °C. The foaming time of the samples in the heat press was 40 min.

2.3. Characterization

2.3.1. Melt flow index – MFI

The MFI of the composites was measured without the addition of either a blowing agent or a DCP crosslinking agent. The experiment was conducted using a DYNISCO equipment Kayeness Test Systems, model D4001 Hv at a temperature of 190 °C and 2.16 kgf according to ASTM: D1238-13.

2.3.2. Rheometry

The EVA crosslinking parameters were obtained through an oscillating disk rheometer TECHPRO, model Rheotech OD + with a frequency of 1.67 Hz, strain amplitude of 1° and temperature of 175 °C. The test specimen weight was 4 g, and the tests were conducted according to ASTM: D2084-11. From the obtained rheometer curve, the following parameters were evaluated: maximum torque (Mmax), minimum torque (Mmin), pre-crosslinking time (t1) and crosslinking time (t0).

2.3.3. Apparent density

The apparent density of the cellular composites was obtained by the ratio of the mass (g) and volume (cm³) of the sample. The density of seven specimens of each sample was assessed according to ASTM: D1622-08. The density test was performed at a temperature of 21 °C.

2.3.4. Mechanical property of tear resistance

The mechanical property of tear strength of the composites was performed using a universal testing machine, EMIC DL2000, in accordance with the ASTM: D624-12. The test speed was 500 mm min⁻¹ with a cell load of 20 kN. A type C tear strength model was used, and the test was performed on five specimens for each sample.

2.3.5. Morphology

The morphology of the cut surface of the cellular composites was evaluated by scanning electron microscopy (SEM – SHIMADZU SuperScan SS-550), previously coated with Au. The software used to measure the cell size was Image Tools for Windows, version 3.00. A universal stereoscope optical microscope also assessed
the morphology, mark ENTEX, coupled with the Photoscape software for editing the images.

The cell population density per unit volume of the foamed composites \( \left( N_f \right) \) was determined from the SEM micrographs using Eqs. (1) and (2) \[13\].

\[
V_f = \left( 1 - \left( \frac{\rho_f}{\rho_p} \right) \right)
\]

\[
N_f = \frac{\left( \frac{nM^2}{A} \right)^{\frac{3}{2}}}{\frac{1}{1 - V_f}}.
\]

where \( V_f \) is the void fraction (\%), \( \rho_f \) is the density of the cellular composite – foamed (\( \text{g cm}^{-3} \)), \( \rho_p \) is the unfoamed composite density (\( \text{g cm}^{-3} \)), \( N_f \) is the cell density (cell \( \text{cm}^{-3} \)), \( A \) is the area of the micrograph (\( \text{cm}^2 \)), \( M \) is the magnification factor for micrograph, \( n \) is the number of bubbles in the SEM micrograph (cells).

3. Results and discussion

3.1. Rheological properties

3.1.1. Melt flow index – MFI

The MFI is inversely proportional to the viscosity of the composite and its rheological behavior will depend on temperature and pressure \[12,14\]. In general, increasing the filler content and reducing the particle size of the filler in the composites entails a decrease in the MFI value. Thermoplastic composites reinforced with WF have higher viscosity (and lower MFI) with smaller WF particle sizes due to the increase in the contact area of the fiber–matrix interface. Small particles tend to agglomerate and aggregate in the polymer melt, which significantly hinders the mobility of the composite during processing \[12,15,16\].

The values for the MFIs of the composites with different WF particle sizes are shown in Fig. 2. After increasing the filler content in the composites, the MFI decreases due to the presence of load limits on the molecular mobility of the composite, causing an increase in viscosity of the same and corroborates what is described in the literature. The decrease in MFI is observed with decreasing particle size and increasing filler content and is more clearly observed in composites reinforced with WF3.

3.1.2. Rheometry

The crosslinking process in EVA foams chemically attaches the EVA polymer chains, forming a resilient three-dimensional network which confers elasticity and mechanical resistance to EVA that is required to support the action of gases from the blowing agent without the collapse and deterioration of the cells during composite expansion \[17\].

While the crosslinking process is primarily chemical, tests to evaluate the mechanism of crosslinking in EVA can be based on physical changes that occur in polymer at specific processing conditions, such as temperature and mechanical shear \[18\]. To determine the characteristics of the cellular EVA/WF composite crosslinking, the test was conducted by an oscillating disk rheometer, and the results are shown in Table 2, which presents the data for the \( M_\text{an}, M_\text{s}, t_1 \) and \( t_90 \).

The \( M_i \) reflects the composite viscosity at the considered crosslinking temperature and is thus indicative of processability. The viscosity is directly related to the formation and growth of cells in the cellular composite and reflected in the strength the gas must exert to start the formation and expansion of the cell in composite foaming \[5\]. The \( M_i \) is related to the stiffness of the crosslinked composite and is a result of a complex system involving the absorption of mechanical energy by both the crosslinked matrix as well as the influence of the WF fillers in the composite. In the EVA/WF composites, both the \( M_i \) and \( M_s \) increase with increasing filler content and decreasing WF particle size. These results indicated that the viscosity and stiffness increased in the composite with increasing filler concentration and decreasing particle size. This result corroborates the results from the MFI test that showed the same behavior of viscosity increasing with decreasing particle content.

![Fig. 1](image1.png)

Fig. 1. MO micrograph of the surface of the unfoamed EVA composite: size and dispersion of the wood flour particles in the composites with 30 phr of (a) WF1, (b) WF2 and (c) WF3.

![Fig. 2](image2.png)

Fig. 2. The MFI of unfoamed EVA/WF composites.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( M_i ) (dN m)</th>
<th>( M_s ) (dN m)</th>
<th>( t_1 ) (min)</th>
<th>( t_90 ) (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>EVA</td>
<td>5.29</td>
<td>36.96</td>
<td>1.46</td>
<td>7.38</td>
</tr>
<tr>
<td>EVA/WF1 (10)</td>
<td>5.02</td>
<td>26.53</td>
<td>1.55</td>
<td>7.90</td>
</tr>
<tr>
<td>EVA/WF1 (20)</td>
<td>5.06</td>
<td>29.09</td>
<td>1.86</td>
<td>7.72</td>
</tr>
<tr>
<td>EVA/WF1 (30)</td>
<td>5.17</td>
<td>35.79</td>
<td>1.58</td>
<td>7.68</td>
</tr>
<tr>
<td>EVA/WF2 (10)</td>
<td>5.84</td>
<td>42.38</td>
<td>1.43</td>
<td>7.90</td>
</tr>
<tr>
<td>EVA/WF2 (20)</td>
<td>7.48</td>
<td>41.46</td>
<td>1.50</td>
<td>7.62</td>
</tr>
<tr>
<td>EVA/WF2 (30)</td>
<td>8.82</td>
<td>59.28</td>
<td>1.14</td>
<td>5.64</td>
</tr>
<tr>
<td>EVA/WF3 (10)</td>
<td>8.14</td>
<td>39.60</td>
<td>1.94</td>
<td>7.72</td>
</tr>
<tr>
<td>EVA/WF3 (20)</td>
<td>10.23</td>
<td>46.96</td>
<td>1.44</td>
<td>5.90</td>
</tr>
<tr>
<td>EVA/WF3 (30)</td>
<td>11.56</td>
<td>55.39</td>
<td>1.48</td>
<td>6.97</td>
</tr>
</tbody>
</table>

Table 2

Rheometric properties of unfoamed EVA/WF composites.
size and increasing WF content. Another factor associated with increased $M_d$ may be the interaction of wood with peroxide during crosslinking of EVA. Free radicals formed by peroxide decomposition can react with wood particles and thus, can improve the fiber-polymer interface and provide increased stiffness in the EVA/WF composite [4].

The $t_{s1}$ indicates the safety process time, that is, the time limit before the crosslinking starts. It can be observed that the $t_{s1}$ did not vary significantly with the increasing of filler content and particle size reduction. The optimum crosslinking time $t_{90}$ presents a slight reduction in the EVA/WF$_2$ (20) and EVA/WF$_3$ (20 and 30). This reduction can be attributed most likely to the lower number of crosslinks resulting from the lower amount of polymer available in the mixture consequent to the higher WF content. Another factor related to the decrease in $t_{90}$ may be the separation of polymer chains due to interference from the wood particles in the EVA/WF composite.

### 3.2. Morphology

Figs. 3 and 4 illustrates the SEM and MO micrographs of the e-EVA/WF composites with different WF particle sizes. In cellular composites reinforced with vegetable fibers, the morphology is generally a complex system, due to presence of non-spherical and non-uniform cells, co-existence of open and closed cells and deformed cells due to chemical and physical influence of vegetal particle/fiber on the cellular composite formation [3,19,20].

Increasing the WF content at various granulometries decreases the size and increases the number of cells in the cellular EVA/WF composites because the WF acts as a nucleating agent in the foaming process, and WF increases the viscosity and melt strength of the mixture during expansion, limiting cell growth [13,21].

Although WF can act as nucleating agents, the inherent moisture, as well as the volatiles extractives present in wood are released during processing, causing significant deterioration of the foam cell structure, that is, a non-uniform cell distribution and a large average cell size, as shown in Table 3. Furthermore, the large amount of WF causes irregular cell nucleation and cell growth, generating foams with low cell density and a large, non-uniform cell size with possible deterioration of properties [10,11].

The formation of cells in the cellular composites is influenced by the WF because the existence of micropores or voids in the polymer-fiber interface provide the migration of the gas generated by the blowing agent to these regions and thereby provide the cell initiation and propagation from this site. Another factor observed by Rizvi et al. [3] is the presence of moisture and extractives in wood particles that initiate cell nucleation. The volatilization of moisture and extractives, which occurs below 175°C, generates a small quantity of gas, which generally migrates to the polymer-fiber interfacial region and thereby starts the formation of a small void space. The gas from the blowing agent migrates to the forming void space in the polymer-fiber interface and continues cell growth from this place. Thus, the greater the number of WF particles, the greater the presence of nucleation sites for new cell formation in the cellular composite [3].

With decreasing WF particle size, especially evident in samples produced with WF$_3$, a greater tendency to form closed cells is observed. This result is due to distribution of the WF particles in the walls of cells, whereas with larger particles, there is a greater tendency to break the cell wall, causing a greater concentration of open cells. The cellular composites produced with WF$_3$, which have lower cell density (low amount of cells per area, as shown in Table 3), have the thickest cell boundary walls. This characteristic may be related to action of WF particles during cell nucleation. As EVA/WF$_3$ composites have the smallest particle size, considering a single particle, this particle has a smaller polymer-fiber contact area in comparison with composites produced with WF$_1$ and WF$_2$. Possibly, initiation areas for nucleation were insufficient to initiate gas prop-
agation from this region, and the gas from the blowing agent, instead of forming new cells, migrated to another cell already forming, thereby increasing the size of an existing cell.

According to Chen et al. [22], larger particles contain more trapped air and promote the formation of larger cells. However, when the gas pressure is low, only large particles originate new cells during the expansion process. Thus, small particles of WF with low air concentration have a lower degree of nucleation, which affects the cell density. When comparing the cellular composites produced with WF1 and WF2, the cellular composites produced with WF1 showed higher cell sizes according to data presented in Table 3 and Fig. 5.

The uniformity and heterogeneity of cell size in the composites are based on the peak intensity on the Y-axis and the amplitude of the X-axis in the cell size frequency distribution graph. The samples containing WF1 and WF2 obtained larger variations in cell size, as can be observed through the broadening of the X-axis amplitude in the distribution curve, while the composites produced with the WF2 had smaller cells size and were more homogenous, as can be observed through the narrower amplitude in distribution curve and the largest peak intensity on the Y-axis. These results are in accordance with Figs. 3 and 4 and Table 3.

Doroudiani and Kortschot [23] related that different cellular morphologies can be obtained depending on the type, size and concentration of the vegetable fiber used in the cellular composite. Most of the energy absorption (mechanical and thermal) is directly influenced by the ratio of cell size and fiber size in the cellular

### Table 3

Morphological properties of cellular composites.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Average cell diameter (μm)</th>
<th>Cell size variation (μm)</th>
<th>Number cells per micrograph</th>
<th>Nf (cells cm⁻²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>e-EVA/WF1 (10)</td>
<td>1267 ± 519</td>
<td>616–2570</td>
<td>9 ± 1</td>
<td>5.02 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF1 (20)</td>
<td>815 ± 294</td>
<td>102–1470</td>
<td>24 ± 4</td>
<td>20.91 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF1 (30)</td>
<td>616 ± 297</td>
<td>90–1260</td>
<td>26 ± 2</td>
<td>28.62 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF2 (10)</td>
<td>848 ± 244</td>
<td>538–1385</td>
<td>10 ± 2</td>
<td>5.54 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF2 (20)</td>
<td>671 ± 238</td>
<td>269–1346</td>
<td>23 ± 7</td>
<td>19.60 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF2 (30)</td>
<td>454 ± 232</td>
<td>115–1192</td>
<td>30 ± 5</td>
<td>35.00 × 10⁸</td>
</tr>
<tr>
<td>e-EVA/WF3 (10)</td>
<td>1191 ± 457</td>
<td>384–2192</td>
<td>10 ± 1</td>
<td>5.32 × 10⁵</td>
</tr>
<tr>
<td>e-EVA/WF3 (20)</td>
<td>773 ± 282</td>
<td>192–1570</td>
<td>24 ± 1</td>
<td>17.68 × 10⁵</td>
</tr>
<tr>
<td>e-EVA/WF3 (30)</td>
<td>597 ± 263</td>
<td>153–1240</td>
<td>27 ± 2</td>
<td>19.09 × 10⁵</td>
</tr>
</tbody>
</table>

**Fig. 4.** MO micrographs of (a) e-EVA/WF1 10; (b) e-EVA/WF1 20; (c) e-EVA/WF1 30; (d) e-EVA/WF2 10; (e) e-EVA/WF2 20; (f) e-EVA/WF2 30; (g) e-EVA/WF3 10; (h) e-EVA/WF3 20 and (i) e-EVA/WF3 30.
composite. When the cellular composite is exposed to mechanical deformation, the energy can be absorbed and dissipated by fiber pullout, matrix fracture or dislocation of the fiber in the matrix at the polymer-fiber interfacial region.

In general, in cellular composites, when the cells of the composite are large and the fibers are small, the fibers are dispersed into the cell wall such that the mechanical strength of these composites are influenced by the mechanical properties of the polymeric matrix, the thickness of the cell wall and adhesion strength of polymer and fiber. In composites which the cells are small, and the fibers are large, the fibers are dispersed throughout the cellular structure, puncturing multiple cells, and the mechanical strength is strongly dependent on the mechanical properties of the fiber and the interface bonding strength polymer-fiber [23,24].

![Fig. 5. Cell size frequency distribution in cellular composites produced with (a) WF1, (b) WF2 and (c) WF3.](image1)

![Fig. 6. SEM micrograph of cut surface of (a) e-EVA/WF1 30, (b) e-EVA/WF2 30 and (c) e-EVA/WF3 30.](image2)

![Fig. 7. Density and tear strength properties of the cellular composites.](image3)
Fig. 6 illustrates the arrangement of the WF in the cellular composites. It can be noted that all three particle sizes of WF are deposited in the cell wall of the composites and moderate adhesion properties are observed in the polymer/fiber interface region, even with the presence of a coupling agent. This weak interaction between the particle and the matrix can be a function of the low compatibility between the two components of the composite or and the gas migration from the blowing agent to the fiber matrix interface region.

3.3. Mechanical and physical properties

The density and the mechanical property of tear strength of the cellular composites with different particle sizes are shown in Fig. 7. The decrease in particle size of the composite provides a tendency of increase in the mechanical property of tear strength of the cellular composites. The composites produced with WF3 had superior mechanical properties compared to the other composites and, at levels of 20 and 30 phr, had similar tear strength than pure e-EVA. Morphological analysis shown in Fig. 3 provides evidence that the composites produced with WF3 had thicker cell walls, which may have contributed to the improved mechanical properties, specifically, the tear strength, of these samples.

The density of the cellular composite was also reflected in the mechanical property of tear strength. In general, the cellular composites showed a reduction of approximately 65–60% by weight compared to non expanded EVA, the density of which is between 0.90 and 0.95 g cm⁻³ [17]. The highest densities were observed in the cellular composites produced with WF3, most likely associated with the fact that the higher viscosity affects the foaming process. With the higher density, fewer voids exist in the cellular composite; therefore, there are higher concentrations of polymer and higher wall thickness, which affects the composites’ mechanical properties.

4. Conclusions

Particle size acts directly on the viscosity of cellular composites, providing reductions in MFI with reduced particle size and increased filler content. In the rheometric test, the Mf and Mn increase with decreasing particle size and increasing filler content. In morphology of cellular composites, WF acts as a nucleating agent during the expansion process and provides an increase in cell density (number of cells per area) with increasing filler content. Wood particle size influences the cell size of the cellular composite due to the capacity of gas migration from the blowing agent to the fiber/matrix interface region. The highest cell density and cell size homogeneity were observed in the composites made with WF3.

The mechanical properties of tear strength are directly related to the cellular morphology of the composites. The highest tear resistance properties were observed at concentrations of 20 and 30 phr of WF3. The fine wood particles (WF3) provide higher density in the composite and larger thickness in the cell borders wall, which have influenced the mechanical responses of these composites.

References