

Review Article

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Assessment of eggshell-based material as a green-composite filler: Project milestones and future potential as an engineering material

<https://doi.org/10.1515/jmbm-2022-0269>

received September 12, 2022; accepted December 16, 2022

Abstract: The utilization of eggshell (ES) waste as a composite filler has increased significantly in the last 5 years. This increase in usage took place due to its unique characteristics, which improve the properties of the resulting composite. Adding a weight fraction of ES particles into a composite can improve its mechanical properties, although not all studies have shown this phenomenon. Studies on these composites' thermal and tribological properties are still limited, so more in-depth studies could be carried out. The degradation of composite performance due to friction or exposure to humidity and hot temperature is another area that is worthy of further study. In this work, we discuss changes in composites' mechanical, thermal, and tribological properties associated with the addition of ES particles, examining both untreated particles and those treated with carbonation. This work can serve as a guide for the utilization of ES particles as an environmentally friendly composite material.

Keywords: eggshell waste, mechanical properties, thermal properties, tribological properties, calcination, green composite

1 Introduction

Environmental issues have recently attracted the attention of researchers. This is because toxic and polluting materials can disturb the health and balance of nature. It is important to reduce, reuse, and recycle materials in order to preserve human life. The Central Bureau of Statistics of the Republic of Indonesia reported that the national production of chicken eggs reached 5.54 million tons in 2021 [1]. However, this production total was still below that of China, which reached 33,547 million tons, with Mainland China producing 33,090 million tons and the United States of America producing 6,707 million tons [2]. Egg production continues to increase and is expected to reach 90 million tons by 2030 [3]. One of the by-products of egg consumption is eggshell (ES) waste, with the weight of the ES comprising 10–11% of the egg's weight [4].

ES waste is abundantly available in Indonesia and has not been used optimally, so it has become a form of environmental waste. Using this waste could reduce the problem of household waste, help to conserve natural rock and soil resources, reduce global climate warming, aid in the development of new materials or green products, and provide a substitute for other natural calcium sources [5]. Another advantage of eggshell waste is that it is non-toxic and safe to use [6].

The exploration of environmentally friendly materials sourced from natural waste as a constituent of composite materials has increased recently [7]. One of these materials is ES waste. Some of its areas of use include plant fertilizers, bone tissue, oil adsorbents [8], medical applications, biodiesel production [9], polymer composite fillers [10–12],

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bioresources [13], eco-wall ceramic tiles [14], matrix composite reinforcement [7], pH modifiers [15], biodiesel catalysts [16–18], hybrid reinforcement [19], and adhesive modification [20]. The main component of ESs is calcium carbonate. This material is very effective in reducing emissions that have been shown to be harmful to humans [21]. The use of ES waste in composite paper [22] represents an effort to reduce the adverse effects of waste. For these applications, it is necessary to manage ES waste through initial treatments before sending it to industries that utilize ESs [9]. Excessive amounts of ES waste can cause problems for the environment, for example, (i) the transportation of waste to landfills leads to additional costs in the production process, and (ii) when dumped on the ground, the membrane attached to the ES can attract pests and cause the dispersal of energy [23].

In this work, we discuss the effect of the addition of ESs on the performance of composites. We examine polymer composites, metal composites, and hybrid composites. The ES pre-treatment, as well as the method and parameters used in the composite manufacturing process, significantly affects the mechanical properties, thermal properties, tribological properties, corrosion behavior, dimensional stability, and the response of the material to the environment. In this study, we also aimed to identify gaps in the research to aid in the development of environmentally friendly materials.

2 Eggshell

The ES is formed during the egg's journey in the oviduct *via* the sequential formation of the structure of the egg. The deposition of calcium in the ES occurs in the ES glands and takes about 17–20 h [24]. The ES is composed of 95% calcium carbonate in the form of calcite and 3.5% proteins, proteoglycans, and glycoproteins [25]. The outermost layer of the ES is the cuticle, which has a thickness of 10–30 μm and is insoluble in water. The cuticle layer is composed of an organic layer with a protein content of up to 90% and high levels of cysteine, glycine, glutamic acid, lysine, and tyrosine [26].

Researchers have shown interest in the use of ESs due to their numerous advantages, including their low density, low cost, renewability, and the fact that they are environmentally friendly and show good thermal stability at high temperatures [7]. ESs are abundantly available as a form of organic waste worldwide. Studies have also shown that ESs are a high source of calcium. Each bird's ES weight is different. For example, the weight of chicken ESs is

about 11% w/w, whereas that of ducks is 12.6% w/w [5]. Egg weight is related to the percentage and thickness of ESs, their shape index, and their specific gravity. Parameters used to observe the quality of ESs include shell weight, thickness, strength, and density [27]. The composition of a chicken ES is shown in Figure 1.

A strong ES is needed to reduce egg damage, protect it from infection, prevent water loss, and to provide a source of calcium [28]. A decrease in the ES' membrane thickness causes a decrease in the quality of the physical barrier and could affect the structure of the ES [29]. The age of the brood stock strongly influences the ES quality. Thin and weak ESs cause losses during production, transportation, and marketing. The losses for this industry have been estimated at 5–7%. Several factors that affect the ES strength are heredity, age, health, season, production level, diet, environment [30], time of oviposition, and the use of a cage system [27]. The crucial characteristics of ESs have been identified as egg weight, egg shape index, specific gravity, and shell ratio [20]. The quality of an ES can be identified based on its strength, namely, the ability of the ES to withstand external forces without cracking and breaking. Egg-specific gravity is an indicator of ES strength [20].

Several studies have shown that ESs can be used in fillers or composite reinforcement because they are abundant, cheap, lightweight, and suitable for low-load-bearing composite applications [31]. The review of ES characteristics presented here includes investigations of the chemical, physical, and mechanical properties; the wear rate; and the morphology of ES waste. Figure 2 shows that the utilization of ESs as a composite material increased significantly in the last 5 years, especially in relation to metal matrix composites (MMCs).

2.1 Preparation of ES particles

The preparation of ES waste for different applications involves relatively similar stages, that is, waste collection,

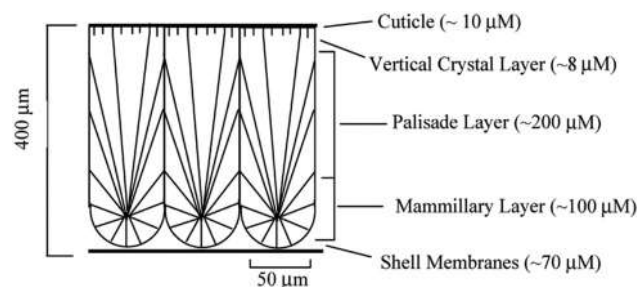


Figure 1: Schematic illustration of layers in a chicken ES [25].



Figure 2: Mapping of ES waste utilization for composite development. The graph is compiled and summarized based on the search data in [32].

flushing, drying, and milling. ES waste is relatively easy to collect from the food industry [32], the waste industry [33], poultry farmers [34], the cake industry [35], local markets [36–40], hotels [4], the egg and chicken processing industry [41], food industry [42], and household waste [43].

The washing of ESs is carried out to remove dirt or foreign particles, using warm water and soap [44], fresh water [31], and sodium hypochlorite (NaOCl) solution [45]. To remove the ES membrane, Lumlong *et al.* [46] soaked it in a vinegar solution for 30 min. The ES membrane floats and can be removed, whereas the deposited particles are calcium carbonate, which is a filler material. The separation of the inner membrane from the ES can be performed by mixing the eggshell powder (ESP) in NaOCl solution, stirring for 30 min, and then storing it at room temperature until two layers are formed. The top layer is removed, and the precipitated layer is washed 10–12 times with distilled water. The powder is then stirred in a mixture of 6% isophthalic acid in ethanol for 1 h [45]. In addition, Nayak *et al.* [47] treated ESs with sodium chloride solution at 70°C.

Drying is carried out to remove water, odor, organic membrane contaminants, and egg remnants from the shell. The moisture in ESs can be dried naturally or by heating. Forced drying has been carried out in heating ovens at various temperatures and with various heating times, such as 60°C for 60 min [48], 80°C for 24 h [40], 100°C for 2 h [46], 100°C for 4 h [45,47], 105°C for 2 h [49], 105°C ± 5°C for 24 h [50,51], and at a temperature of 140°C to obtain a constant weight [45]. Meanwhile, the natural heating approach uses the sun's heat [31]. To keep the ES particles dry and avoid contact with outside air, they should be stored in a desiccator [47].

Milling and refining ES particles can be carried out using a ball mill, grinding, and blender machine. Various speed settings for the ball mill machine have been used in previous studies, such as a rotating speed of 6,000 rpm for 6 h [44]; a speed of 750 rpm for 1 h [52]; and a low speed of 250 rpm, sieving for 20 min, and then repeating this process three times to obtain particles with a size of 106 µm [53]. The manufacturing of ES particles can also be carried out with a grinding machine [31,40,46]. The densities obtained are in the range of 2.5–2.6 g/cm³ [40]. A blender machine can also be used to grind the ES particles to obtain an average particle size of 100 µm [45].

Separating the membranes from ES particles can be carried out using precipitation. The most widely used mechanism involves adding distilled water to the ES particles and stirring them manually. After that, natural deposition is allowed to occur; the denser ES settles, whereas the lighter membrane floats. Thus, this membrane is easily removed by draining it out of its container [51].

2.2 Pre-treatment of ESs

Pre-treatment of ESs can increase the composite's bond strength by modifying the particle surface. This surface modification can be carried out by soaking the ES in a specific solution. In one study, raw ESs immersed in lard and palm oil showed increased hydrophobic properties, although the level of oil concentration had no significant effect on changes in their hydrophobic properties [46]. ES particles were also separated *via* immersion in a 10% NaOH solution for 6 min. The heavier ES particle layer was stirred in a mixture of 6% isophthalic acid and

ethanol for 1 h and dried at 80°C. As a coupling agent, isophthalic acid exhibited better strength and interactions in LDPE/ESP composites [54].

Another method for modifying the surface of ES particles is soaking with stearic acid. ES particles, ethanol, and distilled water were mixed in a ratio of 1:1:3 with a magnetic stirrer for 2 h at room temperature. The next step was heating the mixture at 80°C for 30 min and adding 2 wt% stearic acid solution. The mixture was continuously stirred for 2.5 h to ensure a homogeneous mixture. Particles treated with 2 wt% stearic acid were dried at 105°C for 24 h [51].

ES particles, either untreated or with calcination treatment, exhibited good interfacial bonds with various matrices, including both metallic and polymeric matrices. However, the calcination treatment of ESs resulted in better interfacial bonding when compared to that of raw ESs [10]. Calcination is the process of heating solids at high temperatures to remove volatile substances, oxidize some of the mass, or make the material more brittle. Calcination is also known as carbonation. The temperature and time of calcination treatment play important roles in changing the ESs' structure. The use of an appropriate carbonation temperature can make the carbon porous. The formation of pores on carbon was obtained using CaCO_3 . These conditions allow carbon to act as an adsorbent to remove various water and non-aqueous contaminants [38].

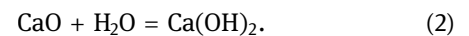
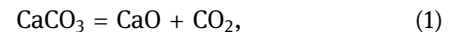
The carbonation of ESs at 500°C for 3 h was shown to be capable of removing carbon-containing materials. The carbon released during carbonation leaves through the pores. The use of carbonated ES particles as fillers in metal composites increased the volume of porosity and the light density, along with the percentage of ES added [7]. ESs that were calcined at temperatures of 600, 700, 800, 900, and 1,000°C for 2 h resulted in a more variable weight reduction in samples. The weight loss of each sample at these temperatures was 5, 5, 26, 46, and 52.8%. Temperatures of 600 and 700°C for 2 h did not have enough energy to cause the decomposition of CaCO_3 [15], but the surface area increased to 276 m^2/g [38]. When the temperature was increased continuously to 800°C for 2 h, the CaO and CO_2 phases began to form, although in small amounts [15], but there was a significant increase in the surface area, to 626 m^2/g [38]. Increasing the temperature to 900 and 1,000°C for 2 h caused the complete decomposition of CaCO_3 into CaO and CO_2 [15].

The optimum calcination temperature to produce pure CaCO_3 was observed at 700°C for 1 h. Calcium carbonate under these conditions was very effectively used

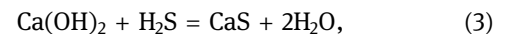
to synthesize hydroxyapatite (HAP) [55]. When the treatment time was increased to 5 h, CaCO_3 was observed to change into an elongated structure [56]. The decomposition of calcium carbonate began at a calcination temperature of 800°C for 1 h. In this condition, the CaO phase began to appear, with a microstructure resembling a flower and with a specific surface area of 8.71 m^2/g . The phase formed at this temperature is β -TCP, and this phase predominates [55]. When the duration of calcination was increased to 4 h with nitrogen media, a phase change to CaO occurred gradually, although in limited quantities, with CaCO_3 as the main component [16].

ESs calcined in a crucible at 900°C for 1 h left traces of Fe in the form of $\text{Ca}_2\text{Fe}_2\text{O}_{11}$, and showed a decreased calcium content and an increased oxygen content. ES calcination treatment resulted in a decrease in the flexural modulus, tensile strength, tensile modulus, hardness, and wear index, but the impact strength increased [57]. In duck ESs, researchers observed a change in the rhombohedral structure of CaCO_3 to CaO in the face-centered cubic form. Calcined duck ESs exhibited good dispersion and electrical properties and high porosity. Thus, duck ESs could potentially be used as dielectric materials, absorbents, and fillers [5].

Calcination at 900°C for 4 h with atmospheric and nitrogen media showed that a complete structure of CaO was formed. Calcination temperatures higher than 900°C are not appropriate carbonation temperatures because endothermic activation reactions can occur [44]. The thermal decomposition process of ESs is shown in Eq. (1). CaO is very hygroscopic by nature and quickly absorbs water from the atmosphere, thus forming Ca(OH)_2 , as shown by Eq. (2).



The decomposition of protein-containing sulfur in the egg white produces H_2S . H_2S cannot react directly with CaCO_3 to form CaS because H_2S is a weak acid. However, H_2S can react with CaO through H_2O , as shown by Eqs. (2) and (3). The CO_2 produced by reaction (1) can drive the activation process, as shown in Eq. (4), which leads to the formation of a pore network on the carbon.



ES particles in the form of Ca(OH)_2 were found to be very suitable for use as catalysts and additives when compared to CaCO_3 particles [56]. The Gibbs energy equation can be used to estimate the minimum thermal

decomposition temperature. In the calcination process of chicken ESs, the thermal decomposition temperature occurs at a temperature of 848°C [15]. Calcination temperature has a great effect on the formation of porous carbon. This porous carbon has been effectively used as an adsorbent to remove contaminants from water and non-aqueous solutions. Research has also shown that ES can be used as an activating agent in the manufacturing of carbon, and that it is cheap, effective, and environmentally friendly [38].

ESs treated with calcination up to a temperature of 1,200°C contain carbon in the form of graphite. ES particles obtained *via* calcination have a much smaller grain size, resulting in higher fineness. These conditions resulted in a relatively uniform distribution of particles in the metal matrix, and no agglomeration of ES particles occurred. The interface wettability between the ES particles and the metal matrix was the reason for the increase in the mechanical properties of the composite [58].

3 ES particles as a filler in MMCs

Three methods often used to produce MMCs are powder metallurgy, stir casting, and extruding. Composite performance has been shown to be affected by compaction pressure, mold temperature, molten metal temperature, and the time and speed of stirring.

3.1 Manufacturing of metal matrix composites

3.1.1 Powder metallurgy

Copper and ES particles were bonded together with an Al6061 matrix. The sample was formed with a compaction pressure of 15 tons, then sintered at 550°C for 10 min [53]. Demirdal and Aydin [52] used powder metallurgy and hot pressing to produce magnesium matrix composites. Magnesium particles and ESs were mixed using a planetary ball mill at a speed of 250 rpm for 30 min. This mixture was then compacted at a pressure of 50 MPa and a temperature of 600°C.

3.1.2 Stir casting method

The stir casting method is a comprehensive method that has been used in the manufacturing of metal-based

composites [19,59–61]. The critical parameters in this process are the mold temperature, the melting of the metal, and the duration and speed of stirring. This parameter affects the distribution of reinforcing particles in the metal solution, thereby causing a change in the mechanical properties of the composite.

Aluminum (Al) metal Al6061 and ESs were heated at temperatures of 500 and 750°C for 1 h, respectively. ES particles were added to the molten metal sequentially so that the particle distribution was uniform. The composite mixture was stirred at 500 rpm for 10 min and then poured into the mold [62]. Balan *et al.* [61] heated aluminum Al7075 in a graphite crucible at a temperature of 700°C and stirred it at a constant speed, with stirring times of 4, 10, and 15 min. The addition of glass fibers and ES particles was carried out gradually and the mixture was stirred until homogeneous, then poured into molds and allowed to cool. Moona *et al.* [60] carried out SiC and ES reinforcement of Al7075-T6. Al ingots were stored in graphite crucibles and heated in an electric furnace to a temperature of 900°C. The reinforcement, heated in a muffle furnace at a temperature of 500°C for 1 h, was poured into molten metal and stirred for a particular time. The mixture of metal and reinforcement was then subjected to an electromagnetic stirrer for 30 s and cooled.

Al was heated at $700 \pm 50^\circ\text{C}$ until it melted, then was allowed to cool in the furnace to a semi-solid state at 600°C. Then, 2 wt% magnesium powder was added to the molten metal to improve the wettability of the metal by increasing the surface energy and lowering the surface tension and interfacial energy between the reinforcement and the matrix. ES particles calcined at 1,000°C for an hour were added to the semi-solid metal at various temperatures and stirring times. This mixture was then heated to $750 \pm 50^\circ\text{C}$ and stirred mechanically to be poured into a sand mold [59].

3.1.3 Extrusion

AA2014 metal composite/carbonated ES/SiC was made by means of electromagnetic stir casting with the following parameters: current: 12 A, time: 180 s, matrix temperature: 700°C. The composite was immediately extruded on a UTM machine at a pressure of 60 MPa [63]. Using disintegrated melt deposition, the Mg–2.5Zn composite, reinforced with ES, was arranged in layers in a graphite crucible and heated to a temperature of 750°C. To distribute the ESs evenly, stirring was carried out at 450 rpm. The molten metal was then poured into molds to make ingots. The cast ingot was heated at 400°C for an hour, and then hot extrusion was carried out at 350°C [64].

3.2 Mechanical behavior

The utilization of ESs as a reinforcement in MMCs had been widely carried out by researchers in recent years. ESs offer exciting prospects in reinforcements and fillers because of the characteristics of ESs observed in both untreated and carbonated conditions. This organic material is available in abundance, cheap, and non-toxic. The characteristics of the matrix determine the quality of the resulting composite. The mechanical properties studied include hardness, tensile strength, compressive strength, impact strength, and density.

3.2.1 Al composite Al6061

ES particles have been used as reinforcements in Al6061 composites *via* the powder metallurgy method. ES particles were evenly distributed in the matrix, forming interfacial bonds between the particles and the matrix and increasing the composite's hardness. The addition of ESs increased the hardness but decreased the composite's electrical conductivity and density [53]. This result is in line with Dwiwedi *et al.*'s research, which showed that the composite's strength and hardness increased with the addition of up to 4 wt% ESs. If the proportion of ES filler was above 4 wt%, they observed decreases in these properties due to increased porosity [62].

3.2.2 Al composite Al6351

Calcined ES particles were mixed with aluminum Al6351 and magnesium using the stir casting method. Response surface methodology (RSM) and artificial neural network (ANN) modeling were used to estimate the effects of process parameters such as the stirring speed, stirring time, and preheating temperature of ES reinforcements on the hardness and toughness of the composites. The ESs used in this study were treated with calcination at 1,000°C for 1 h. This study showed that RSM and ANN modeling were effective in estimating the relationship between the composite properties and process parameters [59].

3.2.3 Al alloy AA2024

Based on field emission scanning electron microscope (FESEM) analysis, ES particles were evenly distributed on the Al composite, and no agglomeration was formed. XRD analysis showed that the ES particles had good

wettability with the metal matrix, indicated by the presence of small peaks. The addition of ES particles at 10 wt% resulted in the composite's highest tensile strength and compressive strength, whereas the highest hardness was achieved with the addition of 7 wt% ES [65].

3.2.4 Composite Al–Zn

Researchers have successfully achieved the development of surface composites by mixing carbonated ES particles at temperatures of 400–600°C for 3–4 h with the magnesium alloy AZ31B using the friction stir method. Shell compositions ranging from 2–6 wt% significantly affected the thermal conductivity (TC) and the coefficient of thermal expansion (CTE) [66]. With the addition of ES particles at 20 vol% to the Zn–Al alloy, the composite's elongation and tensile strength increased, by 28.57% and 18.86%, respectively. A high energy difference between the ES' surface and the matrix could improve the adhesion between the filler and the matrix [67].

3.2.5 Composite Al–SiC

Carbonated ES particles exhibited better characteristics when compared to untreated ESs in the development of Al–SiC composites. The weight fraction of 12.5 wt% ESs in untreated and carbonated conditions exhibited the optimum values in terms of hardness, tensile strength, fatigue strength, and the corrosion rate. However, the toughness and ductility of the composite decreased as the percentage of ES particles increased. The higher the ES percentage, the higher the change in composite density [19].

3.2.6 Al–Si–Mg–Ti composite

Raw ESs were added to the Al–Si–Mg–Ti composite through the stir casting method, resulting in a dendritic structure and intermetallic particles released from the dendritic arms with the addition of 0.5 and 1.5 wt% ES. The release of intermetallic and CaO formed a rosette group, resulting in a significant decrease in the strength of the material. The addition of 3 wt% ES increased the composite's yield strength (18.92%), but the ultimate tensile strength and toughness significantly decreased by 24.06 and 28.94%, respectively. This was due to the formation of the Al matrix network structure, CaO dispersion, and the reduction in grain size to 103 μm [68].

3.2.7 Al–1.5Mg–1.5Sn composites

Magnesium (as a wetting agent) and tin (as a filler) can be evenly distributed in the Al matrix. The tin particles melt during the sintering process. The addition of ES particles causes the formation of higher porosity. A study on this method showed that the addition of ESs at 3 wt% improved the composite's compressive strength (40%) and hardness (15%). The lower porosity of the composite caused an increase in hardness and compressive strength [7].

3.2.8 Al–Cu–Mg composites

ES carbonation revealed contents of Ca, Si, O, C, Mg, and P. The C present during the carbonation process was in the form of graphite. The structure of the Al–Cu–Mg composite without ESs shows a eutectic phase containing Cu_3Al_2 and Al_6CuMg_4 in the Al matrix. Cu and Mg were present in the solid solution or precipitate as Cu_3Al_2 and Al_6CuMg_4 phases in grains and along grain boundaries. Characteristics of carbonated ES particles were better when compared to raw ESs. The higher the percentage of ES, the higher the hardness and tensile strength of the composite, but the lower the impact energy and density [58].

3.2.9 Al can waste composite

Waste Al cans were used to manufacture composites with the addition of ESs at proportions varying from 2 to 12 wt%. The distribution of the particles appeared to be even on the Al matrix. These composites exhibited improved mechanical and tribological properties. The modulus of elasticity, yield stress, tensile strength, hardness, and wear resistance increased with the increase in the ES particle composition. The most significant contribution to the increase in yield stress was observed with the addition of 4–6 wt% ESs [69]. Lubis *et al.* [48] demonstrated the same phenomenon, but they found that the modulus of elasticity and rupture reached a maximum with the addition of 20 wt%.

3.2.10 Pure Mg matrix composites

Magnesium particles with a size of 100 μm and a purity of 99.5% and ESs with a size of 10 μm were produced using powder metallurgy and hot-pressing devices at a

temperature of 600°C and a pressure of 50 MPa. Adding ES particles up to 10 wt% could improve the wear rate of the composite from 0.034 to 0.014 mm^3/m . Low and high load changes caused changes in the abrasive wear mechanism, leading to delamination. The presence of micro-galvanic activity negatively influenced the use of ESs [52].

3.2.11 Mg–2.5Zn composites

Mg–2.5Zn–3ES and Mg–2.5Zn–5ES composites with ES contents of 3 and 5 wt% showed low corrosion rates, whereas composites with 7 wt% ES showed an increase in the rate of micro-galvanic corrosion and a decrease in fracture strain. Microhardness, compressive strength, elongation, and damping capacity increased with the addition of ES particles. The addition of 7 wt% ES increased the damping capacity by 105.21%. Mg–2.5Zn composite reinforced with ESs showed a reduced corrosion rate. This decrease was caused by the formation of a bioactive apatite layer [64].

3.2.12 Mg–2.5Zn–1Mn composites

Magnesium is a light metal that is widely used in the automobile, aerospace, and electronics industries. The addition of ESs into the Mg–2.5Zn–1Mn composite was carried out using powder metallurgy. The density and hardness of the composites increased with increase in the ES composition up to 10 wt% [70].

3.3 Thermal behavior

The presence of ESs tends to increase the TC and the CTE. The addition of ESP to the AZ31B matrix alloy confirmed the improvements in the TC and CTE. An increase in the ESP percentage and an increase in the temperature from 50 to 400°C were observed to decrease TC and CTE. ES particles increased the formation of solid interfacial compounds in Mg_2Ca and Mg_2SiO_4 , as identified by means of XRD analysis, with ES particles acting as a thermal barrier, thereby decreasing TC [66].

A study showed that the thermal stability of nano-composite ESP (NCESP) was higher than that of regular ES particles. NCESP with silver-based nanoparticles generated *in situ* could be used as an antibacterial cleaning powder in household appliances. ESs could also be used

as an inexpensive antibacterial filler with a polymer matrix to manufacture antibacterial nanocomposites [71].

3.4 Tribological behavior

The wear resistance of Al–1.5Mg–1.5Sn was increased up to 65% with the addition of 3 wt% ES particles. Based on the wear morphology, identified through SEM-EDX, the types of wear were abrasion, adhesive, delamination, and oxidative wear. The addition of ESs tends to lead to a higher porosity, which improves the composite surface's wear rate [7]. This result contradicts that the one reported by Agunsoye *et al.* [69], who found that the higher the content of ES particles (2–10 wt%), the lower the wear rate. Lower wear rates correlate with increased hardness and strength. Ononiwu *et al.* [72] showed that different carbonation temperatures of the ESs over a period of 2 h had differing impacts on the tribological behavior of the composite. Carbonated ESs at temperatures of 900 and 1,200°C as composite fillers showed excellent wear resistance compared to untreated ESs. This increased wear resistance was due to the ability of the ES particles to resist shear flow in the AA6063 Al alloy.

3.5 Corrosion behavior

Corrosion behavior is influenced by many factors, including water content, corrosive elements in the air, temperature gradients, and physical and chemical stability [19]. The addition of certain elements can reduce the corrosion rate of steel or other metal composites. Recent trends indicate that researchers have started to use organic materials for corrosion control. The method involves adding organic material to the metal composite or utilizing organic material as a coating material. Balan *et al.* [61] used ANOVA to determine the optimal composition of glass fiber, ES particles, and stirring time in the manufacturing of Al matrix composites. Their study showed that the use of 6 wt% ES and 15 wt% glass fibers as additional elements in the Al7075 composite, with a stirring time of 5 min, could significantly reduce the corrosion rate. Other studies have shown that corrosion resistance can be increased by adding ESs [70,73,74]. The addition of ES particles could reduce the corrosion rate of Al alloys. The corrosion rate reached the lowest value at 12.5% ES with the addition of ESs without treatment or with carbonation. The corrosion rate decreased from

5.79 to 5.24 mmpy and from 5.79 to 4.60 mmpy for untreated and carbonation treatment of ESs, respectively. The combination of heat treatment and ES carbonation resulted in the lowest corrosion rate, which was 4.28 mmpy [19]. Corrosion control was also carried out by depositing ES particles using the electrophoretic deposition method. Bakar *et al.* [75] experimented with depositing calcium carbonate from ESs on gray cast iron. Their study showed that the calcium carbonate layer formed prevented oxygen diffusion, inhibiting corrosion on the steel surface.

4 ES particles as a filler in polymer matrix composites

4.1 Manufacturing of polymer matrix composites

4.1.1 Injection molding

Injection molding is a method of injecting raw materials into the mold cavity under a certain pressure. The injection molding process consists of four stages: clamping, injection, cooling, and ejection [45]. A thermoplastic pellet is first compressed in a reciprocating screw and then pushed into the heating chamber. The softened material flows under pressure through a nozzle into a cold mold. After the injection of the raw material into the cavity, it is compacted to obtain the required shape. Finally, the shape is removed from the mold [45]. In one study, fine ES particles were mixed with linear low-density polyethylene (LLDPE) at 190°C in a single-screw injection molding machine and sampled for 2–5 min. After that, the sample was allowed to cool [39]. Resin and hardener, in the same ratio, were stirred in a beaker for 5 min at a constant speed. The weight percentage of ESs was added to the resin mixture, stirred for 5, 10, and 15 min, and then poured into molds [33].

4.1.2 Hot and cold press

In this method, low-density polyethylene was allowed to melt for 3 min, then ES particles were added to the Z-blade mixer for 3 min. The composite mixture was removed from the mixer and mixed at a temperature of 180°C, at a speed of 50 rpm. Preheating was carried out at 180°C for 4 min, then the sample was compressed at the

same temperature for 2 min and allowed to cool for 2 min [54]. Farahana *et al.* [76] used the same route, but the heating temperature was 190°C.

ES particles at 2 wt% were dissolved in ethanol and stirred for 20 min in a tumbler ball mill to break the agglomeration. Recycled low-density polyethylene (RLDPE) was added to the ES solution and stirred. After that, it was heated at 80°C until all the ethanol evaporated. Palm kernel oil was used in the mixture as a plasticizer. The RLDPE/ESP composite sheet was made in a two-roll polymer mill at 150°C. The composite sheet was hot-pressed using hydraulic compression molding [77]. High-density polyethylene (HDPE) granules were melted using a Brabender two-roll mill at a temperature of 170°C and a speed of 50 rpm. ES particles were added to the liquid HDPE and mixed for 8 min. The resulting composite was hot-compacted at a temperature of 170°C, with a pressure of 15 kPa for 10 min [40].

The polyester composite was manufactured *via* compression molding with a composition of 10 wt% jute fiber, 10 wt% coir, 77 wt% polyester matrix, and 3 wt% filler. The filler used was ESs and nano-clay. Woven jute fiber and coir fiber were cut into 300 mm × 300 mm sections with a fiber ratio of 1:1 with a weight of 10 wt%. ES and nano-clay were mixed using mechanical stirring and a shear mixture at 500 rpm for 1 h at room temperature. The mixture was held in a vacuum oven for degassing. Then, 1.5 wt% methyl ethyl ketone peroxides was poured into wax-coated molds with a brush. The woven fibers were placed one by one, and the matrix was placed between the woven fibers. The pressure was applied to the mold for 6 h at room temperature [78].

4.2 Mechanical behavior

4.2.1 Natural rubber composite

ES particles can be used as a filler or reinforcement, although they are more precisely used as fillers. Adding ESs to natural rubber (NR) composites can increase the modulus and tensile strength. In a study by Lumlong *et al.* [46], the ES surface was modified by immersing it in palm oil and lard. The results showed that ESs without surface modification increased the hardness, as well as increasing the tensile strength by 25% and the modulus by 30%. On the other hand, the ESs with surface modification increased the tensile strength and modulus by 30%. ESs used as fillers for NR composites were heated in a furnace at a temperature of 600°C for 2 h. Bao *et al.*

[49] showed that increasing ES loading increased the tensile strength and fracture elongation, but the hardness and hysteresis loss ratio decreased.

4.2.2 Glass fiber reinforced polymer composite (GFRP)

The use of GFRP composites composed of polyester resin, glass fibers, and ES particles affects the flexural strength and hardness of the material. The flexural strength and hardness of GFRP composites exhibit optimal values. Studies have shown that the addition of ES filler can limit the rate of crack propagation and delamination [79,80]. A similar study showed that a composite with ES particle filler showed increased stiffness and decreased combustibility. The addition of ESs in that study also showed that with an increased weight fraction of ESs, the tensile strength also increased, but the water absorption decreased.

4.2.3 Epoxy resin composites

Epoxy composites reinforced with ESs increased the hardness and tensile strength of the composites, but water absorption decreased with the addition of ESs. The addition of ES particles at 16 wt% increased the tensile strength, flexural strength, impact strength, and hardness, but water absorption and the elongation percentage decreased [81]. Iloabachie *et al.* [82] showed an increase in flexural strength with the addition of up to 25 wt% ESs. Meanwhile, the composite hardness of epoxy resin/ES particles reached the maximum value with the addition of 20 wt% ES. The poor distribution of ES particles in the epoxy matrix could decrease the composite hardness.

Owuamanan *et al.* [51] obtained different results, namely, a decrease in tensile strength, flexural strength, and impact strength as the percentage of ES weight increased. Stearic acid treatment on ESs did not significantly change the material's mechanical properties. The results of this research were in alignment with those of Nayak *et al.* [47], who observed a decrease in tensile strength when the percentage of ESs increased. However, the tensile modulus and impact strength of the E-glass-reinforced epoxy composite was higher. The tensile modulus and impact strength reached the highest value with the addition of 6 wt% ES particles, at 6.63 GPa and 75.38 kJ/m², respectively. The tensile strength of the E-glass/epoxy composite without a filler was 172 MPa.

ES nanoparticles positively impact tensile strength, tensile modulus, flexural strength, hardness, and

resistance to erosion in epoxy bio-composites at a certain weight fraction. Adding ES nanoparticles in the range of 2–3 wt% resulted in optimal values of its mechanical properties. The distribution of ES particles in the epoxy and the use of nano-sized ES particles in the form of CaCO_3 increased the wear resistance of the composite in regard to surface erosion [83]. The use of ES microparticles in a polymer composite increased its tensile strength by 22% with the addition 10 wt% ES particles, but the elongation at break decreased. Another advantage was that the ES particles exhibited good wettability with the matrix [42].

The use of plastic waste and ES particles to manufacture composites could be achieved by using polymer composites and implementing the stirring method. The combination of 30 wt% plastic particulates and 15 wt% ES particles with stirring for 10 min resulted in excellent hardness [33]. Carbonated ESs have different characteristics. Carbonation treatment is more effective for epoxy composites that require hardness, tensile strength, and flexural strength. The addition of 10 wt% ESs into the compound matrix composite conferred high impact and compressive strength values [84].

4.2.4 Polyamide composites

Asha and Sekhar compared the performance of a composite using a polyamide and black nylon matrix reinforced with ES particles. The tensile strength, flexural strength, and impact strength of the black polyamide/nylon composite increased with the increase in the percentage of ESP, but the use of black nylon exhibited a better performance when compared to polyamide composites [45]. The increase in Young's modulus in polypropylene (PP) composites was caused by the limitation of motion and stiffness of the filler, but the impact strength decreased with the increase in the percentage of ES filler. ES bio-fillers were able to provide better matrix continuity. This was because of the laminar shape, specific surface area, and grain size of the ES particles, so there was little free space at the PP composite interface [85].

4.2.5 Polyethylene composite

The elongation percentage decreases with the increase in the composite's particle size and the filler content in the composite. Incorporating ESs into LLDPE could increase the stiffness and dimensional stability of the composite, whereas water absorption and fire resistance increase

with the increase in the filler content [39]. Hussein *et al.* also showed the same phenomenon, observing that the tensile strength, tensile modulus, and hardness of HDPE composites decreased with the increase in the weight fraction of ES particles, but the elongation and impact strength increased. The percentage of water absorption reached its maximum value with the addition of 5–10 wt% of ES particles [86].

4.2.6 Rigid polyurethane foam composites

The addition of ESs to rigid polyurethane foam composites significantly affected their material properties. The addition of ES particles increased the composites' density and compressive strength. Other advantages were low water absorption and brittleness, high dimensional stability, and non-toxicity. This study showed that an ES particle load of 20 wt% resulted in optimum characteristics for the chemical industry [87].

5 ES as a hybrid composite filler

ES particles are organic materials that are compatible with forming good bonds with various types of matrices, metals, and polymers. To increase the advantages of the use of ESs, they can be used together with other organic materials. Here we examine the advantages of using ESs with other fibers for hybrid reinforcement.

5.1 ES–silicon carbide (SiC)

ESs were treated with carbonation at 500°C for 3 h to remove the ES membrane. The secondary reinforcement of the hybrid composite used SiC with the aim of increasing its strength; modulus; abrasive wear resistance; thermal stability; and resistance to acids, alkalis, and salts up to 800°C. Carbonated ES and silica particles could be evenly distributed in the Al alloy AA2014. The maximum specific strength, thermal expansion, porosity, density, and fabrication cost reached the minimum values at 7.5 wt% ES and 2.5 wt% SiC [63]. Girimurugan *et al.* [88] used an Al6061 Al matrix with the same hybrid reinforcement. This research showed that adding a hybrid reinforcement increased the tensile strength, percentage of elongation, yield strength, compressive strength, and hardness by more than 50%. However, the impact energy and

impact strength decreased. This decrease was due to the weakening of the interfacial bond between the reinforcement and matrix.

5.2 ES–SiC–Al₂O₃

ESs increased the fatigue strength of the Al7075–T6 hybrid composite material containing ES reinforcement, SiC, and Al₂O₃. Using the Taguchi method, researchers found that the stirring and composition parameters obtained with the addition of 1.5 wt% ESs, 1.5 wt% SiC, and 1.5 wt% Al₂O₃, and a mechanical stirring time of 360 s produced the highest fatigue strength in light-load applications [60].

5.3 ES–glass fiber type-C

The stir casting method was used to manufacture aluminum matrix composite with type-C glass fiber reinforcement and ES particles. Process parameters were optimized using the Taguchi method. Hybrid reinforcement significantly contributed to its corrosive behavior and tensile strength. This research showed that for the reinforcing composition of 15 wt% glass fiber and 6 wt% ES particles, a stirring time of 15 min produced the optimum tensile strength. A stirring time of 5 min was required for the corrosive rate to be low [61].

5.4 ES–mengkuang fiber

The combination of mengkuang fiber and ESs increased the tensile strength and impact strength of the NR/HDPE hybrid composite. The strong bond between the ES filler and the matrix and the more even distribution of the ES particles were the main reasons for the increased mechanical properties. The higher the weight fraction of the ES, the better the tensile strength and impact of the composite. The same was observed for the tensile modulus, which reached its maximum value at the fiber and filler ratio of 15/5. The high fiber content in the composite led to high water absorption as well. This was due to the hydrophilic nature of the fiber. The treated fibers exhibited better interfacial bonds and water absorption, thus preventing the dispersion of water molecules [89].

5.5 Bio-composites

The hand lay-up method was used to make a thin film from a bio-composite material. The bio-composite thickness swelling increased with the increase in the banana peel/ES filler ratio, for both banana peels that were given treatment or for those without treatment. The lower the ES content, the greater the thickness swelling of the bio-composite [90]. Coconut jute fiber and ES particles in a different bio-composite were bonded with gelatin gel. The weight fraction of 99% ES and 1% coconut jute fiber showed the optimal hardness. This study showed that gelatin gel could form an interfacial bond between the filler and the fiber [91].

5.6 ESs–*Bauhinia racemosa* fiber

Balan *et al.* optimized the effect of ES content and *Bauhinia racemosa* fiber on hybrid composites using ANOVA. ES filler significantly affects the hardness and water absorption of polymer composites. The composition of the fiber and ES must be designed appropriately according to the desired conditions. The maximum composite hardness was observed when the fiber and filler composition reached the maximum level. However, the amount of fiber must be minimal to ensure that there is minimal water absorption. This is due to the nature of the fiber, which absorbs water upon exposure to water. A small ES particle size could lead to reduced porosity. Chemical treatment of the fiber reduced its hydrophilic properties [43].

5.7 ES–water hyacinth–basalt fiber

A hybrid composite reinforced with basalt fiber with ESs and a water hyacinth filler was created. Manikandan *et al.* compared the characteristics of hybrid composites using an epoxy matrix with the addition of ES and water hyacinth particles. The 3% ES and 3% water hyacinth composition showed excellent flexural strength, tensile strength, and impact strength. This research also confirmed that adding ESs or water hyacinth improved the composites' observed mechanical properties [92].

5.8 ES–rice husk–coir fiber

A composite of coir fiber, rice husk, and ESs was made using the hand lay-up method. The composition of 40 wt

% coir fiber, 48 wt% epoxy resin, 12 wt% ES particles, and rice husk offered the best mechanical properties. The composites' tensile strength, flexural strength, and impact strength increased with an increase in the filler content from 0 to 12 wt% [93].

5.9 ES–montmorillonite nanoclay (MMT-NC) jute fiber–coir fiber

Ganesan *et al.* compared the behavior of hybrid composites in dry and wet conditions (with exposure to seawater and river water). The composites reinforced with jute fiber and coir fiber showed increased mechanical properties with the addition of ES filler and nano clay. This study showed that a composition of 1.5 wt% ES, 1.5 wt% MMT-NC, 10 wt% jute fiber, 10 wt% coir, and 77 wt% polyester matrix showed an increase in tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength under all environmental conditions under study. In general, the addition of filler led to reduced water absorption [78].

5.10 ES–rice husk–cassava peel–palm kernel shell

Particleboard hybrid composites provide a means of using agricultural waste for the creation of indoor furniture with good mechanical properties. Composites with a composition of 50% rice husks by weight, 25% ESs by weight, and 25% cassava peels by weight showed the best density, thickness expansion, and water absorption values [94].

5.11 ESs–*Achatina fulica* snail shells

Snail shells and ESs were crushed with a ball mill to 50 nm. Composites were made using the conventional resin casting method. Tensile strength, impact strength, stiffness, hardness, and water absorption reached the optimum values with the addition of conch shell particles, ESs, or a combination, at 5–10 wt%. An increase in the ES content beyond 10 wt% tended to decrease the mechanical properties of the epoxy composite [95].

5.12 ES–glass fiber–burlap fiber

Ramamurthy *et al.* [96] compared the mechanical behavior of glass and jute fibers-reinforced hybrid composites with and without ESs. The ultimate tensile stress, flexural strength, and composite hardness decreased with the addition of ES particles to the hybrid composite, although the density increased.

5.13 ES–glass fiber–kenaf fiber

Adding ES particles into these hybrid composites led to significant improvements in their mechanical properties. The best composition was 5 wt% glass fiber, 15 wt% kenaf fiber, and 9 wt% ESs, resulting in a composite with excellent tensile and flexural strength. Water absorption increased as the number of ESs increased. An increased kenaf fiber content led to increased water absorption [36].

5.14 ES–talc

ESs positively affected the properties of PP/ES/talc hybrid composites. ESs were highly compatible with PP composites and provided a composite strengthening effect compared to the traditional calcium carbonate applications. Another interesting fact was that ESs were able to replace the role of talc without decreasing the mechanical properties of PP–talc composites [97].

6 ESs as a green material

A hybrid adsorbent composed of lignin deposited in black liquor and ESs was very effective for SO₂. The composition of 50% lignin in the lignin–ES mixture with carbonation treatment at 800°C for 50 min produced an SO₂ release of 52 g/kg in both wet and dry conditions. Carbonation temperatures above 750°C facilitate gasification reactions between the carbon matrix and CO₂ due to calcite decomposition. This can lead to pore formation and the development of porosity [41].

The carbonation-calcination loop of the CaO–CaCO₃ process has shown the potential to capture CO₂ gases. The exploration of ESs as a CO₂ gas catcher has been

carried out based on several considerations, such as their abundance, renewability, relatively straightforward preparation methods, and excellent thermal stability [98]. Recycled ESs are thus an environmentally friendly alternative material for CO₂ absorption. Fresh ESs showed a CO₂ absorption capacity of 0.57 g CO₂/g sorbent, whereas for CaO conversion, they showed the capacity to absorb 0.25 g CO₂/g sorbent. The average absorption of ES decay was 19.75 mg CO₂/cycle. Increases in the adsorption capacity of the sorbent were obtained through treatment with acetic acid, citric acid, and formic acid, with absorption rates of 5.65, 6.20, and 5.02 mg CO₂/cycle, respectively [99].

Hsieh *et al.* [100] also modified the surfaces of ESs using Na₂CO₃ and NaOH solution. ES–Ca(OH)₂–CaO, obtained from ES purification, showed a pore structure with a pore size of about 7 nm. This indicates that the material had an excellent CO₂ adsorption capacity. The addition of an amino-containing polymer, followed by calcination, resulted in polydopamine–CaO with a pore size of 12.6 nm, which exhibited the best adsorption capacity of up to 0.4 g CO₂/g sorbent.

Pure ESs produced *via* the calcination process at a temperature of 700°C were very effective as a calcium source for the synthesis of HAP. The higher the calcination temperature, the higher the powder particle size. Another exciting observation was that the direct synthesis method, consisting of dry mixing followed by heat treatment, was shown to affect the powder's morphology [55]. The adhesive bonds of the brake lining increased with the addition of increased concentrations of microparticles of ES waste. SEM analysis showed that the ES microparticle filler had good wettability to the matrix [50].

The need for lightweight engines, changing vehicle architectures, and limited space have led to significant challenges in terms of noise, vibration, and harshness (NVH) of applications. One essential material to overcome NVH issues is carbon black, which is obtained *via* petroleum processing. However, due to its non-renewable nature, it is necessary to find a replacement material. ES composites exhibit static damping and stiffness and do not exhibit the harmful aging of materials [101].

7 Discussion

Applying heat at a specific temperature to ESs can change the characteristics of the material. Heat treatment of ESs can be performed through carbonation or calcination. The temperature and treatment time have

the most significant influence on the material's behavior. ESs heated at a specific temperature and time were found to be able to trigger the presence of carbon in the form of graphite [58].

7.1 Structure and morphology of ES particles

Calcination treatments with different temperatures and times have been shown to result in particles with different sizes and structures. The higher the temperature and time of calcination, the larger the size of the ES particles. The morphology of the ES changes sequentially from oval, to elongated or to bean-shaped with changes in temperature [56]. The intensity of CaCO₃ decreases with the increase in the calcination temperature and the formation of CaO [16]. The structure of the calcined ES resembles a wasp's nest. Boiled ESs contain very low calcium concentrations and very high oxygen contents. Calcination treatment allows the formation of many pores. This is indicated by the ES particles' increasing circumference, area, and pore diameter. The increase in pore size occurs due to the incorporation of smaller pores and the growth of CaO grains in high-temperature treatments. Larger pores can provide space for the accumulation of moisture and dirt [17]. Awogbemi *et al.* showed that the different treatments of ES particles affected their calcium, carbon, and oxygen content concentrations, as shown in Table 1.

Risso *et al.* [102] obtained contrasting findings showing that ESs calcined at a temperature of 900°C for 4 h had a pore volume of 0.0020 cm³/g, a pore diameter of 26 nm, a crystallite size of 44 nm, and an activation energy of 18.1 mol/min kg. SEM observations showed that the differences in the particle sizes of ESs with calcination treatment were more significant than those of ES without calcination [57]. A comparison of changes in pore dimensions in various treatments is presented in Table 2. Commey and Mensah [15] showed that calcined ESs were

Table 1: Chemical compositions of ES particles with various treatments [17]

Element	Chicken ES weight (%)		
	Raw sample	Boiled sample	Calcined sample
Calcium (Ca)	41.31	21.91	46.08
Carbon (C)	14.93	26.40	6.22
Oxygen (O)	43.76	51.68	47.70

Table 2: Details regarding the porosity of ESs [17]

Chicken ES	Perimeter (nm)	Area (nm ²)	Diameter (nm)
Raw	75.73–90.79	210.75–43.14	28.33–30.55
Boiled	24.93–62.80	32.87–112.38	8.90–20.76
Calcined	129.02–184.41	192.60–1917.78	37.68–58.51

suitable for pH modifiers. PH is an essential parameter in the electrochemical reduction process of carbon dioxide and carbon monoxide. Schouten *et al.* investigated the effect of pH on copper electrodes. Their study indicated that methane formation was strongly influenced by pH, whereas ethylene had a different dependence on pH [103].

The treatment of ESs affected the density of the composite, although the change in density was not very significant. Bose *et al.* [19] calcined ESs at 650°C for 3 h and used them as a reinforcement for MMC material. An increase in the ES percentage decreased the density of the composite. This trend differs from that reported by Hassan *et al.* [10], who found that the density of polyester composites increased with an increase in the ES percentage. The composite's density became

progressively lower in the ESs subjected to calcination treatment [10,19].

7.2 Mechanical characteristics

The characteristics of carbonated ES particles have been found to be superior to those of raw eggshells. The higher the ES percentage, the higher the hardness and tensile strength of the composite, but the impact energy and density decrease, as shown in Figure 3 [58]. Oladele *et al.* [57] observed a slightly different phenomenon in measuring the tensile strength of the composite. Figure 4 shows that the tensile strength of the composite reinforced with calcined ES particles decreased. In general, the mechanical properties of the composite reached the optimum value at the addition of ESs at 9 wt%. ES particles with calcination treatment improved impact properties and wear resistance [57]. Several other studies have shown that the hardness of composites reinforced with carbonated ESs exhibited better values compared to non-calcined ESs [10,84].

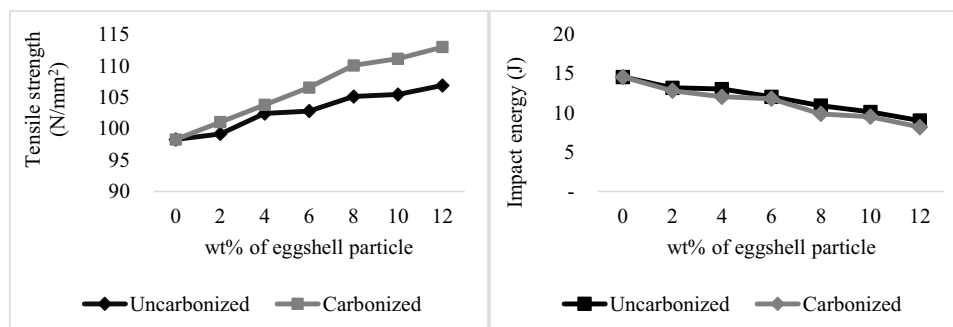


Figure 3: Comparison of tensile strength and impact energy values of raw ES particles and particles subjected to carbonation. Figures are produced based on the data in [58].

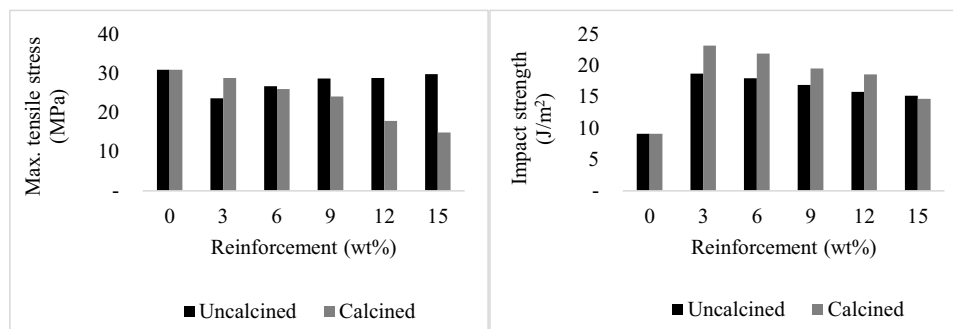


Figure 4: Comparison of tensile strength and impact energy values between raw ES particles and particles subjected to calcination. Figures were produced based on the data in [57].

Stress–strain curves are used to show the tensile strength behavior and Young’s moduli of polylactic acid (PLA) composites. The addition of calcined ESs at a low level (10 wt%) resulted in good tensile strength and moduli. At a large percentage, the presence of calcined ES reduced the composites’ tensile strength [104]. The addition of calcined ESs up to 7 wt% increased the degree of crystallinity of the PLA composite. An even distribution of ES particles increased Young’s modulus but decreased the tensile strength, fracture elongation, and impact strength. The presence of ESs lowered the glass transition temperature, thus making the movement of molecular chains more manageable at low temperatures [105].

The differences in the various discussions of the tensile strength and impact strength of composites obtained using carbonated ESs and untreated ESs were fascinating to study. Changes in the phase, structure, and particle size of ESs from CaCO_3 to CaO need to be studied more deeply. This recommendation is based on several studies which stated that ES particles could capture CO_2 , which would enable environmentally friendly composites to be developed in the future.

7.3 Characteristics of tribology

Wear resistance is indicated by a wear index. The wear indices of hybrid composites were found to increase with the addition of ES particles and sisal fiber. The increased wear resistance of the composite was due to the very soft molecular nature of the epoxy and the presence of $\text{Ca}_2\text{Fe}_7\text{O}_{11}$. Figure 7 shows that the addition of calcined ESs increased the resistance of the composite surface to wear. Adding ES particles at 3 wt% offered the highest wear resistance, with 0.38 mg of calcined ES particles [57].

Oladele *et al.* [106] investigated bio-composites using HAP synthesized from chicken ESs. As shown in Figure 5 (right), they determined that the higher HAP loading in HDPE composites could increase the wear resistance of composites by up to 40 wt% HAC.

7.4 Thermal characteristics

With the calcination treatment, the thermal stability of ES particles was found to be excellent because the weight loss only reached 5.5% at a temperature of 450°C and then stabilized up to $1,000^\circ\text{C}$ [17]. The cold crystallization temperature at 112.83°C exhibited a perfectly crystalline structure. The ES phase lowered the glass transition temperature, T_g , T_{cc} and T_m decreased when the ES increased because the perfection of the crystalline structure could be induced more quickly when the ES particles were dispersed in the PLA matrix [105].

ESs with calcination treatment, at a specific temperature and holding time, form CaO , and when this comes into contact with water, it forms Ca(OH)_2 . Increasing the temperature increases the value of TC and specific heat but decreases thermal diffusion at temperatures up to 363 K. Thermal diffusivity increases when the temperature is above 363 K. An increase in temperature triggers an increase in the kinetic energy of the phonons; therefore, the TC increases [107].

Figure 6(a) shows the division of the weight losses. At temperatures $200\text{--}400^\circ\text{C}$, a weight loss of 4.46% occurs due to the loss of water content and organic matter. The weight loss of 40.84% observed between $600\text{--}800^\circ\text{C}$ is caused by the phase change of CaCO_3 to CaO . Figure 6(b) shows the active CaO content using full carbonation under N_2 atmosphere. This finding showed that there was a minute difference between the actual CaO fraction

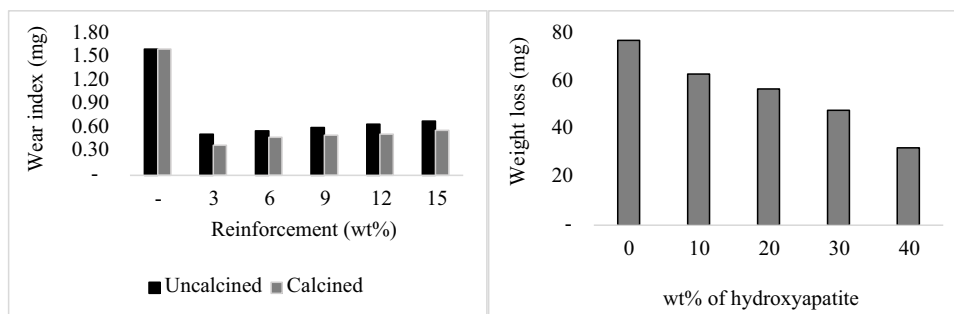


Figure 5: Wear indices of uncalcined and calcined ESs [57] and weight losses of composites [106]. Figures are produced based on the data in [57] and [106].

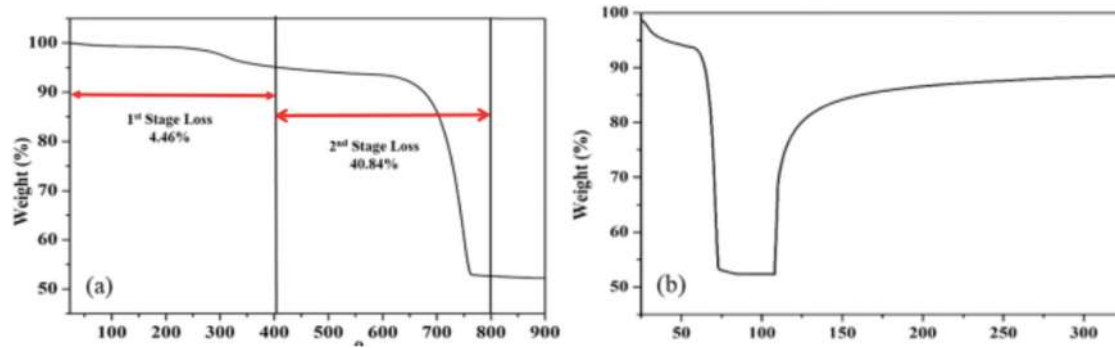


Figure 6: (a) TGA of fresh ESs and (b) carbonation test for the detection of the actual CaO fraction [99].

and the full carbonation test [99]. Another study showed that the weight losses of calcined ES samples were relatively shallow [17].

7.5 Water absorption characteristics

In one study, HAP, synthesized from ES particles, was used to close the pores in the bio-composite, thereby reducing water absorption [106]. This study was in line with that conducted by Gbadeyan *et al.*, who showed that water absorption decreased along with the increased content of ES and snail shell particles. This decrease was due to the shell particles' hydrophobic nature and the filler's interconnecting bonds. ES particles also acted as a barrier to crack propagation, so water absorption decreased. However, an excessively high number of shell particles would cause agglomeration and stress concentrations would occur, which would ultimately weaken the bond between the particles and the matrix [95].

Panchal *et al.* demonstrated a different phenomenon: water absorption increased with the increase in the addition of the particulate filler. The highest increase in water absorption occurred in salt water compared to fresh water and moist soil. This finding was due to the presence of amine and carboxylic functional groups in ES particles [11]. A higher filler content and higher surface area showed better water absorption ability. The higher the filler content, the higher the agglomeration formation, so the dispersion of filler in the matrix became challenging. This led to an increase in water absorption in the composite. The smaller the filler, the higher the water absorption [39].

Panchal *et al.* [11] compared the effect of the ES content in mineral water, salt water, and moist soil environments. Figure 7 shows that the higher the ES particle content, the higher the swelling thickness under

various conditions. However, the increase in thickness was significant in saltwater and moist soil environments. Yusuf *et al.* [90] studied the effect of silane treatment on ES particles in terms of water absorption properties and thickness development. Thickness swelling and water absorption increased with ES content, but ESs with silane treatment performed better. This increase was due to the hydrophilic nature of the ES filler. Arsene *et al.* [108] showed that when in aqueous media, silane was partially or entirely hydrolyzed to form dimers or oligomers.

7.6 Antibacterial characteristics

ESs can inhibit the growth of bacteria, and this effect was found to increase with an increase in the percentage of ES particles in a PLA composite bio-filler [104]. CaO, in its pure state, was a very effective antibacterial agent. The presence of CaO in the composite was able to kill bacteria. ESs calcined at a temperature of 800°C for 2 h were very effective as a chromium heavy metal releaser in wastewater [109]. ESs in a raw state were not effective at killing bacteria. Meanwhile, ESs calcined at a temperature of 900°C for 2 h form CaO, which is a potential source of antibacterial material [110].

8 Prospects of ESs as a future green material

The use of ES particles could have positive effects in the manufacturing of composites or in overcoming environmental problems. ES waste could be utilized for engineering applications. Several studies have shown the positive effects of ESs used as fillers or reinforcements

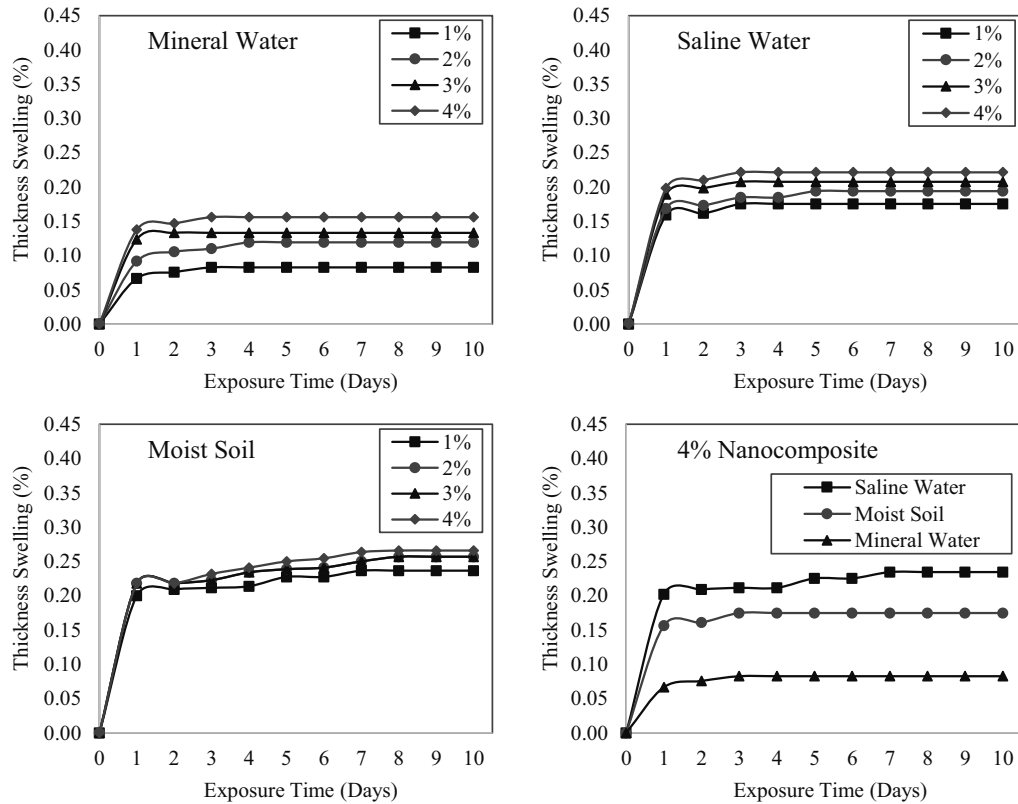


Figure 7: Thickness swelling of ES filler composites under different environmental conditions. Figures are produced based on the data in [11].

for polymer composites, ceramic composites, and metal composites, and as absorbents or catalysts.

The findings of this review indicated that there are still problems concerning the use of ES particles which have not been solved. Our understanding of the characteristics of ESs used as fillers and reinforcements in composites are still largely based on their mechanical and physical properties. Chemical, tribological, thermal, and water absorption reviews have not been widely carried out. Another aspect of this field of research that has not been studied in detail concerns the composite characteristics of ESs treated with calcination.

8.1 Pre-treatment of ESs

Pre-treatment of ESs could improve their mechanical properties, thermal properties, tribological properties, water absorption, and thickness swelling properties. The studies discussed above have shown that calcination treatment can improve composite performance, although decreasing some material properties. This review makes it possible to observe the changes in the behavior of materials caused by the presence of ES particles, both in their

untreated form and with calcination treatment. Previous studies showed that ES particles subjected to calcination treatment showed an increase in contact area, which resulted in an increase in their mechanical properties. Silane treatment of ES particles also showed an increase in the composite performance, especially in terms of thickness swelling. Thick swelling indicates high water absorption by the fiber and the filler. The ability of fibers and fillers to absorb water is determined by their hydrophilic properties. This weakness can be improved through silane treatment. The addition of a silane layer on the particles or filler can limit the water absorption rate so that the degradation of the composite material properties is minimized.

ES particles showed good distributions in composites, especially in MMCs obtained using the stir casting method, which showed improved material performance. However, an excessive ES particle content leads to agglomeration. Agglomeration was observed to cause stress concentration and to weaken the bonds between particles in the composite. The pre-treatment of ESs can be divided into two categories. The first of these is pre-treatment at room temperature, such as the immersion of raw ES particles in NaOH, or silane solution, which has not

been studied in depth. Research on the effect of the solution concentration and time of immersion of ESs in the solution in regard to composite performance is still limited. The second category of pre-treatment is pre-treatment at high temperatures, such as temperature and time calcination, which have also been carried out by researchers. However, this form of treatment of calcined ESs has not been carried out. These gaps in the research must be explored so that the utilization of ESs as an environmentally friendly green material can be achieved. Composites using calcined ESs as a catcher for greenhouse gas emissions have also not been studied in detail.

8.2 ESs as friction materials

In recent decades, researchers have observed that particulate matter (PM) induced by brake linings can have a harmful effect on human health. PM 10 and PM 2.5 air particles were identified as being particularly hazardous. These particles can be formed of dust, dirt, soot, or smoke. PM 2.5 particles are produced by ash, volcanic ash, burning coal, burning forests, and burning biomass. The concentration of these particles is limited to a maximum of $65 \mu\text{g}/\text{m}^3$, whereas that of PM 10 is limited to $80 \mu\text{g}/\text{m}^3$.

PM 10 particles have been found in building construction, garbage disposal, agriculture, forest fires, dust, and bacterial fragments. PM 10 emissions in relation to heavy vehicles have been observed to be caused by exhaust emissions (41%), the suspension of road dust (38%), and brake wear (21%). For light vehicles, exhaust and brake wear were identified as the greatest contributors, at 63 and 33%, respectively [111]. A reduction in this form of emissions could be achieved by formulating and modifying brake linings and discs. Substances that are harmful to human health should be reduced.

One of the materials that is not good for human health is copper. Copper has been widely used due to its excellent TC. Wei *et al.* [112] stated that the TC of disc materials can influence the formation and destruction of contact planes and thus affect the particle size distribution in the air due to heat accumulation at the shear interface. The substitution of copper with other materials with equivalent TC could lead to a decrease in PM 10 emissions.

Using ESs, whether calcined or untreated, would undoubtedly have an effect on PM 10 emissions. Composites containing ESs produce relatively lower PM 10 emissions, which are relatively safe for health. CaO

has demonstrated an excellent ability to absorb CO_2 in high-temperature conditions. Hsieh *et al.* [100] showed that CaO synthesized from ESs could absorb CO_2 , while reducing environmental waste. The utilization of ESs as a friction material has not been widely carried out, and the literature on this topic remains still limited. It is known that particles from hazardous materials can interfere with health and cause disease in humans or pollute the soil, which can interfere with soil fertility. For this reason, it may be beneficial to replace the hazardous components of friction materials with ESs, which are more environmentally friendly. For this reason, observations of their tribological characteristics must be carried out.

9 Conclusion

ES particles offer unique characteristics compared to other calcium carbonate sources. There is a need for in-depth explorations of their use as commercial materials in engineering. This review represents the first step towards observing the behavior of ESs, both in their untreated form and with specific treatments, such as silane treatment or calcination. Encouraging findings have been obtained regarding the utilization of ESs as fillers or reinforcements to be applied in environmentally friendly materials. ESs could even be a solution for reducing greenhouse gas emissions. Further in-depth research is still required in order to respond to these challenges. The key findings noted in this review were as follows:

- 1) The pretreatment of ES particles at room temperature or through calcination has not been widely carried out, so it is not known whether this pretreatment could affect the bond strength between particles and their matrix.
- 2) ES particles primarily containing CaCO_3 can affect the mechanical, thermal, and tribological properties of materials, as well as their dimensional stability, although contradictory results were also found. This finding was understandable because ES particles have complex material compositions.
- 3) ES particles treated *via* calcination or carbonation can be used at high temperatures, so these materials are suitable for applications as friction materials. Calcined-ES-reinforced composites were characterized by excellent thermal stability.

The utilization of ESs that have undergone calcination treatment is feasible and further investigations should be conducted to explore the behavior of ESs

which have and have not undergone calcination treatment under various conditions. In the future, ESs may be identified as an environmentally friendly commercial material with various advantages. This will also provide an immediate solution to the problem of ES waste.

Abbreviation

ANN	artificial neural network
CTE	coefficient of thermal eExpansion
ESP	eggshell powder
GFRP	glass fiber reinforced polymer composite
HAP	hydroxyapatite
HDPE	high-density polyethylene
LDPE	low-density polyethylene
LLDPE	linear low-density polyethylene
MMC	metal matrix composite
MMT	montmorillonite
NC	nanoclay
NCESP	nanocomposite eggshell powder
NR	natural rubber
NVH	noise, vibration, and harshness
PLA	polylactic acid
PM	particulate matter
PP	polypropylene
RLDPE	recycled low-density polyethylene
RSM	response surface methodology
SEM	Scanning Electron Microscope
TC	thermal conductivity

Funding information: The authors state no funding involved.

Author contributions: All authors have accepted responsibility for the entire content of this manuscript and approved its submission.

Conflict of interest: The authors state no conflict of interest.

Data availability statement: All data generated or analyzed during this study are included in this published article.

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