Mechanical and free vibration properties of clamshell particles/polyester composites

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Keywords: clamshell powder, calcium carbonate (CaCO₃), tensile properties, charpy impact, vibration characteristics

Abstract
In this work, an experimental investigation was implemented to identify the effect of adding clamshell powder (CSP) into the polyester matrix on the tensile and impact properties along with vibration characteristics of the particulate composites towards using eco-friendly reinforcement phase. Different weight ratios of clamshell powder, ranged from 0 to 20 wt%, were loaded into the polyester resin with particle sizes ranged from 25 to 75 μm. Tensile, Charpy impact and free vibration tests were performed to the specimens fabricated from the neat polyester and CSP-filled polyester. The results showed that the inclusion of CSP into the polyester matrix could improve the tensile modulus of the polyester up to 50% when the CSP weight ratio equals to 12%. Meanwhile, the strain-to-failure, tensile and impact strengths showed decreasing trends with increasing the CSP filler content owing to the weak adhesion (bonding) strength between CSP and the polyester matrix. Maximum improvements in the fundamental natural frequency and damping ratio of CSP-filled polyester were 24% (at 12 wt% of CSP) and 21% (at 8 wt% of CSP), respectively. Based on the results, the clamshell powder could be used as a very cheap bio-filler material within the polyester matrix if the high stiffness composites with improved damping properties are required.

Introduction
The shell waste is a big issue for the shellfish aquaculture industry as it can account for up to 75% of the total organismal weight [1]. The major component of the clamshell is the calcium carbonate (CaCO₃) which can be employed in several sectors. According to Kao et al. [2], there are about 8 000 tons of clamshell waste produced each year that could be considered as a public health problem. These residues could be reused in construction and the pharmaceutical industries [3]. Nowadays, great effort is being made to increase the mechanical properties of the composite materials without affecting the environment. Natural reinforcements in the form of fibres or particles are used efficiently in producing fully or partially green composites. Natural-particles derived from plant or animal-based are low-cost materials that can be used as reinforcements in the polymeric composites to improve certain mechanical properties of the polymeric materials [4, 5]. Clamshell powder (CSP) is an example of the natural materials that could be used as a reinforcement in the polymeric composites.

The addition of particulate fillers into the polymeric matrix could enhance some of the mechanical and physical properties [6]. Previous studies regarding this topic showed that the mechanical properties of the particulate composites might be largely affected by the properties of the particles themselves such as their size and shape, their distribution throughout the base material, surface traction and surface energy, stiffness and hardness, and adhesion strength with the polymeric matrix [7]. Previous studies showed that good enhancement in the tensile modulus and strength of the particulate composites could be obtained at smaller particle sizes [7–12]. Yang et al. [13] concluded that higher loading of CaCO₃ nano-particles into polypropylene matrix could easily agglomerate without significant effects on the flexural strength and modulus of the particulate composites. The results indicated that there was a specific filling weight ratio of CaCO₃ at which the mechanical properties of the composite reached their maximum values and then started to decrease with increasing the CaCO₃ wt%.
Eiras and Pessan [14] studied the effect of filling different weight fractions (3, 5, 7, and 10 wt%) of CaCO₃ as nano-particles into the polypropylene (PP) matrix on the tensile and impact properties of the particulate composites. Maximum improvements in the tensile stress (13%) and modulus (44%) were obtained for PP samples filled with 3 wt% of CaCO₃ when compared with the neat PP counterparts. Meanwhile, the impact resistance of PP filled with 3 wt% of CaCO₃ exhibited a maximum improvement of about 30% compared with the neat PP samples. Hongzhen et al.[15] showed that maximum improvements in the tensile, flexural and impact properties of wood/plastic composites were obtained when adding CaCO₃ (particle size equal to 10 μm) at weight ratios of 25%, 10%, and 30%, respectively. Othman et al.[16] studied the effect of adding different filler contents of CSP with particle size no more than 60 μm on the tensile properties of polyhydroxybutyrate (PHB) polymer. Maximum tensile strength and modulus were obtained by incorporating the CSP filler up to 30 wt%, but these values were still lower than those of neat PHB owing to the poor adhesion between clamshell particles and PHB polymer. Li et al.[17] examined the mechanical properties of polypropylene (PP) filled with shellfish shell powder (particle diameter less than 0.2 μm). Filling of PP with 5 wt% of shellfish shell powder decreased the tensile strength from 38 MPa to 33 MPa. Meanwhile, tensile modulus increased from 0.83 GPa to 1.47 GPa. Xia et al.[18] studied the effect of adding CSP (0 to 30 wt%) with particle sizes between 2–4 μm on the mechanical properties of PP matrix. Impact strength of the PP samples filled with 30 wt% of CSP showed improvement of 61% compared with neat PP counterparts. Flexural strength of PP filled with CSP did not show a noticeable change with changing the CSP content; meanwhile, the flexural modulus increased significantly. However, the tensile strength decreased significantly with increasing the content of CSP. Damia[19] stated that Young’s modulus of the polylactide matrix increased with increasing the CSP content (with particle size smaller than 63 μm) from 0 to 10 wt%. However, the tensile strength and elongation at break decreased with increasing the CSP filler.

Vibration characteristics such as the natural frequency and damping ratio of composite materials are very important especially in the fields of aerospace, automotive and construction [20]. Sometimes, vibration is a desirable physical phenomenon; however, successive oscillation in the structures often caused problems like noise generation and fatigue cracks initiation [21]. Accordingly, improving composite materials by increasing the damping properties is substantially important. It was stated that polymers alone have damping ratios much greater than fibres-reinforced polymer matrix composites since the bulk polymers have lower elasticity and higher viscosity than their composites [20, 22]. However, large interfacial regions could be obtained by incorporating small particles into the neat polymeric matrix [23, 24]. Damping resulted from frictional sliding at the particles/matrix interface regions could enhance the dissipated energy during the vibration. The dissipation energy due to the sliding friction depends on the particle/matrix adhesion, particles size and their distribution within the matrix. Improving the structural damping without sacrificing its stiffness is a challenging subject. From the vibration test, Huang and Tsai [25] indicated that adding 10 wt% of silica nano-particles (25 nm) into the epoxy resin had increased its loss factor from 1.43 to 1.57 and the modulus from 2.9 GPa to 3.27 GPa. Erklig and Bulut [26] studied the changes in the damping and natural frequency of the S-glass/epoxy laminated composite cantilever beam due to adding borax particles (<45 μm) with mass ratios of 0, 5, 10, 15, and 20%. The results showed that the natural frequency of the particulate hybrid composites increased about 17% after adding 5% of borax filler and it almost remains constant with increasing the content of the particles. Meanwhile, damping ratio increased by 29% when adding 5% of borax and decreased after further addition of borax filler. Alsaaedi et al.[27] investigated the effect of inclusion nano-silica particles (from 0.5 to 3 wt%) on the dynamic properties (natural frequency and damping ratio) of the carbon/Kevear/epoxy hybrid composite. Their study showed that maximum improvement (about 20.5%) in the natural frequency of the hybrid composite obtained at 0.5 wt% of nano-silica content, whereas damping ratio of this composite reduced by 37% compared to composites without nano-silica filler. Chandradass et al.[28] studied the free vibration characteristics of the chopped glass fibres/vinyl ester composites due to adding different contents of nano-clay particles (0, 1, 3, and 5 wt%). The maximum improvements in the fundamental natural frequency and damping ratio of the cantilever hybrid composite beam filled with 5 wt% of nano-clay were 43% and 20%, respectively. In summary, the mechanical and dynamic properties of the particulate composites are highly depending on the size of the added particles, loading (content) ratio, and the adhesion (bonding) strength between the particle and the molecules of the polymeric resin. Developing composite materials with enhanced static and dynamic properties without affecting the environment has motivated scientists to use bio-particle materials as fillers within the polymeric matrices. To the best of the author’s knowledge, no studies have been reported on the effect of adding CSP as a filler on the static and dynamic properties of the polyester.

In this study, the tensile, impact properties along with the vibration characteristics of the unsaturated polyester matrix filled with various weight ratios of CSP were investigated experimentally. Freshwater CSP with particle sizes ranged between 25 to 75 μm were used to reinforce the polyester matrix.
Materials and method

Raw materials
The unsaturated polyester resin was used in this work with 1.5% MEKP (Methyl Ethyl Ketone Peroxide) hardener. Mechanical properties of the unsaturated polyester resin and MEKP are given in Table 1. The clamshells shown in Figure 1 were collected from the cliff of Euphrates river, Iraq. The freshwater clamshells were cleaned using distilled water for removing the clay and dust before the grinding process. The cleaned clamshells were grinded and sieved to size between 25 to 75 μm. Physical properties of the clamshells are given in Table 2.

Fabrication procedures of the specimens
The clamshell powder was dried well inside the oven at 50 °C for 2 h prior incorporating it into the polyester. The CSP charge were weighed using a high accuracy digital balance and mixed well with the polyester resin. The relative weight ratios of CSP to the polyester resin used in this study were 0, 4, 8, 12, 16, and 20 wt%. The MEKP (1.5 wt%) was adding to the mixture of CSP filled and the polyester at ambient temperature equal to 27 °C. The final mixture was poured into closed moulds that were pre-configured according to the required shape and size of the specimens. The composite specimens were left to dry at room temperature for 48 h and then demoulded gently.

Tensile test
Tensile test was performed for the bulk polyester and CSP filled polyester samples as per ASTM D638 [29]. This test is important to find the tensile strength, tensile modulus, and strain-to-failure of the specimens. The dumbbell-shaped specimen (type I) was adopted for this test. The size of the specimen is 50 mm (gauge length) × 13 mm (width) × 3 mm (thickness). A hydraulic tensile testing machine was operated at a constant

Table 1. Mechanical properties of the unsaturated polyester resin and MEKP.

| Property                             | Value |
|--------------------------------------|-------|
| Tensile strength (MPa)               | 38.2  |
| Tensile modulus (GPa)                | 2.6   |
| Tensile failure strain               | 1.9   |
| Density (g cm⁻³)                     | 1.23  |
| Gelation time at 25 °C and 1.5% MEKP (min) | 11 ± 1 |

Figure 1. Clamshells used in the present work.

Table 2. Physical properties of Clamshells used in the present study.

| Property          | Value      |
|-------------------|------------|
| Colour            | Creamy white |
| Bulk density (g cm⁻³) | 1.31     |
| True density (g cm⁻³) | 1.62     |
| Porosity (%)      | 20.3       |
crosshead velocity of 2 mm min\(^{-1}\). To reduce the slippage of the specimen during the tensile test, the emery cloth (grade 80) was inserted into the interface between the specimen’s ends and the machine’s grips. The lab conditions during the test were 26 °C and 48% relative humidity. Five specimens were tested for each CSP loading and the mean values were calculated.

**Impact test**
Charpy impact test was used in this work to find the impact energy absorbed by the specimens subjected to swing impact load. The impact energy at impact moment is equal to 14.8 J. The unnotched impact test specimens were prepared with size equal to 55 mm (length) \(\times\) 10 mm (width) \(\times\) 4 mm (thickness). The mean values of the five replications were considered for each group of specimens (i.e. the bulk polyester and CSP filled polyester). The impact strength (IS) in kJ/m\(^2\) was calculated according to equation (1);

\[
IS = \frac{E_{bs}}{t \times b} \times 10^3
\]

where
- \(E_{bs}\): Represents the fracture absorbed energy in (J)
- \(t\): Represents the thickness of the specimen in (mm)
- \(b\): Represents the width of the specimen in (mm)

**Fundamental natural frequency and damping ratio**
Dynamic properties such as the natural frequency and damping ratio were measured using ceramic shear accelerometer model 352C22 from PCB Piezotronics. National Instruments (NI) with Data Acquisition (DAQ) were used to acquire the accelerometer dynamic signal during the vibration. A cantilever beam with cross sectional area of 20 mm (width) \(\times\) 3 mm (thickness) was adopted to measure the dynamic properties of the specimens fabricated from the polyester resin filled with various weight fractions of CSP. The lightweight accelerometer (0.5 g) was attached at the free end of the cantilever as shown in figure 2. Best signal-to-noise ratio was obtained from placing the accelerometer close to the free end of the cantilever beam. Accordingly, the measured dynamic properties are for a cantilever beam with an attached end mass (accelerometer mass). The active length of the cantilever was 250 mm. The free end of the cantilever specimen was moved 10 mm downward and then released to vibrate freely. The acceleration data was then analysed using FFT algorithm that built in LabVIEW software. Subsequently, the frequency-amplitude domain for each specimen was calculated. The highest peak in the frequency-amplitude domain represents the fundamental natural frequency \((f_s)\) at resonant. Damping ratio \((\zeta)\) of each specimen could be determined from the frequency-amplitude domain using half-power bandwidth method as depicted in figure 3 [30, 31]. Therefore, damping ratio corresponds to this peak could be determined as per equation (2);

\[
\zeta = \frac{f_2 - f_1}{2 f_o}
\]
Results and discussion

Chemical composition of clamshells

Table 3 shows the chemical composition of the clamshell powder using an x-ray Diffraction (XRD). It is clear that CaCO$_3$ is the dominant component of the clamshells (∼95%) and other components can be considered as insignificant constituents [32].

|      | CaCO$_3$% | SiO$_2$% | MgO % | Al$_2$O$_3$% | SrO % | P$_2$O$_5$% | Na$_2$O % | SO$_3$% | Total % |
|------|-----------|----------|-------|--------------|-------|-------------|------------|--------|---------|
| Value| 95.416    | 0.926    | 0.682 | 0.501        | 0.427 | 0.333       | 0.861      | 0.854  | 100     |

Tensile properties

The tensile modulus of the polyester resin filled with different weight ratios of CSP are shown in figure 4. The tensile modulus of the polyester increased as the CSP loading was increased up to 12 wt% and then decreased. The maximum improvement in the tensile modulus was about 50%. Adding the CSP to the polyester resin would increase the polyester stiffness probably due to impeding the polyester chains from slipping on each other when deformed. Beyond 12 wt% of CSP, the particles were not evenly distributed and started to aggregate due to van der Waals force. Consequently, the tensile modulus decreased gradually with increasing the CSP content. However, adding the CSP into the polyester showed a declined trend in the tensile strength as shown in figure 5. The decline in the tensile strength of the polyester in the presence of CSP possibly resulted from the weak interfacial bonding between the CSP filler and the molecules of the polyester matrix. This in turn result in
inefficient transfer of the stresses between the matrix and CSP filler [33–35]. The composite’s strength depends mainly on the weakest path of fracture within the material. Hard clamshell particles affect the composite’s strength in two different trends. The first trend is the reinforcing effect by obstructing the crack propagation. The second trend is the weakening effect by the concentration of stresses they may produce [36]. Stress concentration, interface adhesion, spatial distributions and defect size are the most important parameters that affect the fracture behaviour of the particulate composite materials. The weakest strength path within the microstructure of the CSP-polyester composite is the dominant criterion of failure rather than the average values of its macrostructure properties. Figure 6 shows the effect of the filler content on the strain-to-failure of composite specimens. Clearly, the brittleness of the polyester increased with increasing the CSP loading. The decline in the strain-to-failure is attributed to the low elongation of CSP that constrains the flow of polyester polymer molecules past each other. The failure strain has decreased steadily from 1.87% to 1.02% when increasing the CSP filler content up to 12 wt%, and then declined slightly. The CSP filler restricted the mobility of polyester chains to move easily during the deformation [37].

**Impact strength**
Several factors influence the impact strength of the particulate composite including reinforcement toughness, reinforcement-matrix adhesion strength, and the nature of the matrix fracture [18, 38]. Figure 7 shows the...
impact strength of the bulk polyester and CSP filled polyester specimens. As the bonding between the CSP filler and the polyester matrix was weak, the CSP composites gives lower impact strength than that of neat polyester counterparts. At higher CSP loading, the filler-matrix interaction became weaker and this resulted in lower impact strength of the CSP composites. On the other hand, adding hard and brittle particles to the polyester resin could increase the brittleness of the composite if the interfacial adhesion is weak [36]. Therefore, the impact strength value has declined steadily as CSP filler loading increased. The highest impact strength (7.5 kJ m$^{-2}$) was observed for the bulk polyester specimens and it significantly dropped by 66% when adding 4 wt% of CSP. It is expected that as the CSP filler loading increased, the aggregation of particles increased due to the uncontrolled dispersion of CSP filler within the polyester matrix at high filler loading. On the other hand, the weak interfacial bonding between the CSP particles and the surrounded polyester contributed the low impact strength of particulate composites.

**Free vibration characteristics**

The fundamental natural frequency and damping ratio of the polyester filled with different contents of CSP particles are listed in table 4. Clearly, increasing the CSP filler content up to 12 wt% had increased the fundamental natural frequency of the polyester from 11.08 Hz to 13.69 Hz and then started to decline with additional loading of CSP. This behaviour is mainly attributed to the direct effect of the elastic modulus of the composite on the composite beam stiffness. The higher the elastic modulus, the higher the natural frequency.

Damping is the most sensitive indicator of the molecular transitions and structural heterogeneities in polymers. The incorporation of little content of CSP (4–8 wt%) within the polyester matrix could offer a large amount of interfacial region, which enhances the energy dissipation by hysteresis damping. The microparticles with very low aspect ratios could highly increase the damping of the composite as stated by Finegan and Gibson [39] and Alsaadi et al [27]. Incorporating of CSP within the polyester could also produce a weak interface with the polyester matrix. The weak adhesion between the reinforcement phase and the polymeric matrix could increase the dissipated energy during vibration [40]. The interface regions around the incorporated CSP fillers have a distinctive damping effect to the polyester matrix. The interfacial relative displacement might also result in debonding and friction during sliding in the interface region, which could be considered as the fundamental energy dissipation resource during vibration [23]. The slippage of particles within the surrounded matrix of a vibrating particulate composite material is another damping resource of polymeric composites. Accordingly, the

![Figure 7. Effect of filling different CSP contents on the impact strength of the polyester.](image)

**Table 4. Vibration properties of polyester filled with different contents of CSP.**

| Property                        | CSP wt% |
|---------------------------------|---------|
|                                 | 0       | 4       | 8       | 12      | 16      | 20      |
| Fundamental natural frequency (Hz) | 11.08   | 12.34   | 13.51   | 13.69   | 12.31   | 11.45   |
| Damping ratio × 10$^{-3}$        | 35.43   | 40.23   | 42.82   | 39.33   | 31.98   | 31.45   |
damping of the CSP filled polyester showed increasing trend as the CSP content was increased up to 8 wt%. The damping ratio decreased slightly when the content of CSP was beyond 8 wt% due to the aggregation of CSP and hence these particles would decrease the internal friction and the load transfer between composite’s constituents, which is in consistent with the observation by Zhou et al. [41] and Assadi et al. [27]. At relatively higher CSP weight fractions (i.e. higher than 8 wt%), there is CSP-polyester interface overlaps. The loss of interface by overlaps also takes place due to particles agglomeration as stated by Patel et al. [42].

Conclusions

In this study, effects of adding different contents of CSP into the polyester matrix on the tensile, impact and free vibration properties have been investigated. From the results that have been obtained from this work, the following conclusions can be drawn:

1. The tensile strength and strain-to-failure of the unsaturated polyester have decreased as the CSP content was increased, which means increasing the brittleness of the particulate CSP/polyester composite.

2. A maximum tensile modulus equal to 3.77 GPa (50% improvement) has been obtained for the polyester filled with 12 wt% of CSP.

3. The optimum CSP filler contents that offer the highest natural frequency and damping ratio were 12 wt% (24% improvement) and 8 wt% (21% improvement), respectively.

The current study suggested that the incorporation of CSP as a bio-filler material into the polyester matrix could improve the stiffness of the polyester and the vibration properties; meanwhile, tensile and impact strengths have been sacrificed. Therefore, this partially bio-composite material could be used in the secondary structural applications such as homemade, automotive interior parts that could attenuate vibration and noise and some indoor housing elements towards the production of cost-effective and eco-friendly structures.

Acknowledgments

The authors wish to express their gratitude to University of Babylon for the help in performing the experimental tests.

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