An experimental investigation on enhancing the strength and stiffness of GFRP co-cured composite joint: effect of glass powder addition

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Abstract
Co-cure adhesive joints are preferred by various industries, namely, automobile, marine and aerospace, to join two surfaces in structural applications, as a useful replacement for mechanical fastenings. The present work focuses on the mechanical properties and free vibration behaviour of co-cured glass fiber composites reinforced with glass powder. In the course of the experimentation, the adhesive is being reinforced concurrently with glass powder in four different weight percentages, such as 0%, 0.5%, 1%, 1.5%, and 2%. The mechanical testing results reveal that the addition of 1.5% of glass powder to the epoxy could relatively help in increasing the tensile strength and flexural strength of the co-cured glass fiber composites respectively to the degree of 11.68% (364.29 Mpa) and 24.75% (256.16 Mpa). The Single lap shear results show that the 0.5% glass powder reinforcement significantly increases the shear strength of the co-cured glass fiber composites by 20.91% (19.31 Mpa). Furthermore, the free vibrational study of 1.5% co-cured composites shows that they have a higher fundamental natural frequency than the glass powder reinforced co-cured composites that have a lower weight percentage. Furthermore, the addition of glass powder to the co-cured composites helps in increasing the damping factor of the composites due to the glass powder agglomeration. Neat and glass powder reinforced co-cured samples are further analysed afterwards, using the mechanical and shear test by scanning electron microscopy.

1. Introduction

Superior strength and stiffness along with better damping characteristics of lightweight composite materials, make them suitable alternatives in automobile, aerospace and other weight-sensitive applications than conventional materials [1–3]. In an assembly, thousands of sub-components are being merged through mechanical fastenings, such as bolt, nut, rivet, screw, etc. This leads to the degradation of the properties belonging to the advanced polymer composites and spoils the purpose of the intended application. Overcoming the problems that are associated with mechanical fastening can be resolved by introducing adhesive joining, which would help in solving the problems during the mechanical fastenings, especially in terms of the high-stress concentration that is most often found near the hole. However, an adhesive joint’s performance depends on the joint’s strength, surface preparation, toughness, type of reinforcement, type of adhesive, fabrication process and curing cycle.

Researchers carried out numerous research experiments to study the influence of mechanical fasteners on a composite’s load-carrying behaviour and found that creating holes during mechanical fastening not only increases the stress concentration, but also affects the load carrying capacity of the composite joint. Also, creating holes leads to the peeling of the plies, fiber pullout and affects the toughness of the matrix. Hence, it is vital to develop an alternative manufacturing process for joining the sub-components to further enhance the safety and durability of fiber reinforced composites during their services under various loads [4, 5].
Tremendous development in the composite manufacturing process for joining similar and dissimilar composite materials helps in enhancing the joint efficiency and performance of the composites by solving the problem associated with mechanical fastening, stress concentration and its distribution [6]. Secondary bonding, co-bonding and co-curing are alternative manufacturing processes that are most commonly used for mechanical fastening. This helps to improve the performance of the composite joint. Simultaneously, it reduces the final weight of the assembly and the assembly stress [7]. The main advantage of the adhesive bonding technique over mechanical fastening is the enhancement feature of the load-carrying behaviour and its ability to avoid premature delamination failure which is being done by improving uniform stress distribution over the joint area. It enriches the resistance against failure under loading and allows them to carry more load, making them compatible for intended applications [8]. Even though the secondary and co-bonding manufacturing process has its advantage over mechanical fastening, surface preparation of the composite adherent influences the joint efficiency. Co-curing techniques are introduced to avoid the problem associated with the surface preparation of the adherent [9].

Stevenson [10] compares the flexural strength of the stiffened composite panel that is fabricated through two different bonding processes: co-curing and secondary bonding. The Experimental results reveal that the co-cured composite panel’s load-carrying behaviour is higher than that of the secondary bonding process by 11%. Similarly, geometric accuracies of hat-stiffened panels are evaluated by the Research Centre for Aircraft Parts Technology, Gyeongsang National University, Republic of Korea [11]. Investigation reveal that the manufacturing process, and mould type, alter the strength and stiffness properties of the composites. Comparing it with the co-curing rubber mould, an inflatable mould increases the failure loading value up to 78.21%. Kim et al [12] found that the addition of the adhesive film helps enhance the load-carrying capacity of the hat stiffened composite panels that are fabricated through the co-cure manufacturing process under the buckling load. Similarly, the co-cure fabrication process helps in enhancing the structural capability of the blade-shaped stiffeners. The result shows 24% improvement in the tensile results of the co-cured composites [13]. A comparative study on the post-buckling behaviour of T-stiffened co-cured composite panels with co-bonding and secondary bonding was carried out by Ye et al [14] and it proves that the bonding method influences the buckling behaviour of the composites. Arndt et al [15] in the course of their experimentation fabricated dis-similar composite joints using fibre and aluminium. Furthermore, they employed a three-point bending to analyse the damage tolerance. In the course of their experimentation, they found that the shear properties of the composites are an essential factor in the energy absorption of the hybrid structure. Shino et al [16] found that in comparison to co-bonding, co-cured composite enhances the resistance of Mode-1 delamination. Following which, Pirondi et al [17] compared the tensile strength and fracture toughness of carbon fiber reinforced polymer (CFRP) co-cured and co-bonded composites. As a result of which they concluded according to the Mode-I failure analysis, that the co-cured joint enhances the fracture toughness compared to adhesive bonding. Likewise, Hou et al [18] from NASA proceeded to manufacture a honeycomb sandwich structure using the co-cured method following which they found that the manufacturing process enhances the properties of the composites. Nettles [19] chose to prepare face sheets using co-cured and precured honeycomb sandwich composites. A secondary bonding process was used to attach the pre-cured face sheets to the core. The Results of the experiment reveal that the undamaged strength is higher (by 33%) for the pre-cured laminate than for the co-cured laminate.

Other than the manufacturing process, the highly brittle nature which is associated with the epoxy matrix reduces the toughness of the composite joint [20]. Hence, it is essential to alter the nature of the epoxy matrix by reinforcing the filler material in the epoxy matrix as a secondary reinforcement. Carbon nanotubes (CNT), nano clay, silica powder, etc, are commonly being used by researchers to alter the properties of the epoxy composite [21]. Dhillipkumar and Rajesh [22] investigated the effects of various manufacturing techniques on adhesive joints in composites, and their findings show that the secondary bonded composites offer superior mechanical properties when it is being combined with MWCNT reinforcement up to 1 wt%. Deeraj et al [23] finds in his experiment that adding 1 wt% of polyimide (PI) nanofibers to the epoxy matrix enhances the modulus and fracture toughness of the composites up to 20%. The reason behind this enhancement in the modulus is that an excellent mechanical property which is associated with polyimide (PI) nanofibers enriches the load-carrying behaviour of the composite.

Similarly, MMT nano clay has been employed to improve the strength and modulus of natural fibre composites [24]. Reinforcing the clay with the adhesive enhances the strength and modulus of the natural fibre composite significantly due to the improved adhesion between the fibre and the matrix. Also, the nano filler in the matrix prevents crack formation and its random propagation. Hence, the composite becomes more rigid. Jakab et al [25] analyses the influences of a polymer filler and mineral filler on the tribological properties. They reach the conclusion that the fillers’ particle size significantly influences the wear rate. Sadik et al [26] found that the addition of waste glass powder in the high-density polyethylene matrix enhances the tensile strength and modulus.
Much research has been conducted on the strength behaviours of epoxy-based composites concerning the types of reinforcement, type of filler and its particle size and surface treatment. Many researchers have also taken on the job of carrying out experimental investigation on the mechanical properties of the mechanical fastened composite joint. The above literature attests to the fact that adhesive bonding techniques are an alternative technique which can be used to resolve the problem associated with mechanical fastening. However, adhesive bonding also has its own limiting aspects, such as the surface preparation, types of adhesives and curing time on the strength behaviour of a composite joint. Most of the research focuses on analysing the mechanical properties of the adhesive joined composite. Other than mechanical characterization, the dynamic properties of the composite joint fabricated through the co-curing technique are significant to make them safe during service. An efficient design of an adhesively bonded structure requires the knowledge of the adhesive joint’s static and dynamic behaviour under external loads. The vibration characteristics of the adhesive joints, such as natural frequencies, mode shapes and modal damping, are essential because of their vital applications in practice. Since the harmonic loads and short period impact loads may cause the structural adhesive joints to vibrate steadily or randomly, fatigue becomes important. Hence, the researchers have examined the adhesively bonded joints’ free and forced vibration behaviour. In the present investigation, the co-cure manufacturing technique and the addition of glass powder with weight percentage on the mechanical properties (tensile, flexural and shear) and free vibration characteristics (natural frequency and damping factor) of the composite are analysed.

2. Material and methods

2.1. Materials
In the present work, a composite adherent is prepared with the help of glass fibre reinforcement and epoxy matrix. 600 GSM bidirectional glass woven fabric reinforcement and epoxy adhesive and hardener (HY951 & LY956) have been purchased from Sai sakhthi fibres, Chennai. Composite adherent is utilized in place of the joints using glass powder modified epoxy matrix. Glass powder has been bought from Ashwin Ceramics, Chennai, Tamil Nadu. Glass powder particle size is measured as 43.03553 μm with Laser Scattering Particle Size Distribution Analyzer (LA-950).

2.2. Glass powder/epoxy adhesive preparation
In this research, composite adherents are joined through the co-cure technique using glass powder modified epoxy adhesive. An ultrasound liquid processor is being employed to achieve the uniform dispersion of low-density glass powder in the epoxy matrix. The uniform dispersion of glass powder is then mixed with acetone and then it undergoes sonication for one hour at 40 kHz. Further, it is placed in an ice cube container to avoid the temperature rise during sonication, when the process is stopped at a steady interval of ten minutes and manually stirred to enhance the possibility of uniform dispersion. In continuation, a magnetic stirrer at 800 rpm is being used to stir the glass powder depressed epoxy resin for one hour at 80 °C. At 80 °C, acetone starts to evaporate and it is then kept in the degassing oven for 30 min. As a result of this, the entrapped air is removed during mixing. Further, a hardner is gradually being added in the ratio of 10:1 (by weight) and a mechanical stirrer is used to mix it up.

2.3. Fabrication of the composite laminate
Through the hand lay-up method, the composite laminate with a dimension of 300 mm × 300 mm × 2.5 mm is prepared using the co-cure technique. Two numbers of composite adherents are being prepared with a size of 300 mm × 300 mm × 1.25 mm using the steel mould, which had a top and bottom steel plate. Initially, four numbers of 600 GSM bidirectional woven glass fabrics are arranged on the workbench. To achieve the stiff composite adherent, the glass woven fabric is dipped in the epoxy matrix and the excess resin over the fabric is removed using a small plastic spoon. A sufficient amount of epoxy resin is then poured over the glass fabrics. The excess resin and air bubble formation between the glass fabrics are both removed with the assistance of rollers. After preparing two sets of 1.25 mm thick composite adherent, they are then combined using the glass powder dispersed epoxy adhesive.

In continuation, to study the shear strength of the co-cure composite laminate, lap joint composite specimens are fabricated with the help of a glass powder dispersed epoxy matrix. To manufacture the composite lap joint, 1.25 mm composite adherent is arranged in a lap joint manner with an overlap length of 25.4 mm × 25.4 mm. After placing the 1.25 mm composite adherent, glass powder mixed with the epoxy matrix is poured on to the overlapping area. Furthermore, this arrangement is placed at one bar pressure in the vacuum bag setup. This helps in removing the excess resin and entrapped air in the composite adherent. Followed by the one bar pressure for 45 min, the next cycle of arrangement is being kept for 24 h at room temperature for curing and the industrial oven is used for 24 h at 60 °C for the post-curing period.
2.4. Mechanical characterization

After preparing the composite joint through the co-cure technique, it is sized as per ASTM D3039 and ASTM D790 for analysing the tensile and flexural strength. Tensile specimens are then sized in 250 mm × 25 mm and tested with a crosshead speed of 2 mm min⁻¹ in universal testing machines (UTM). Similarly, the flexural test (specimen size was 127 mm × 12.7 mm × 2.5 mm) is carried out using 3-point bending method at a crosshead speed of 2 mm per minute. 101.6 mm × 25.4 mm dimension is being used for the shear analysis at a 2 mm per minute crosshead speed. The average value obtained for the five samples under each test has been used to analyse the strength behaviour of glass powder dispersed epoxy glass fiber reinforced polymer (GFRP) cured composites and the standard deviation for the test results of tensile, flexural and single lap shear is calculated and plotted.

2.5. Material characterization

SEM morphology analogy is employed within this research to understand the influence of glass powder reinforced epoxy adhesive on tensile, flexural load strength and shear behaviour.

2.6. Free vibration analysis

The modal analysis study is utilized in this research to gain a further understanding of the influence of glass powder reinforced epoxy adhesive on the fundamental natural frequency and damping factor of the first three bending modes under the fixed-free condition of GFRP co-cured. A modally tuned impulse hammer and light weight accelerometer is used to capture the time domain signal. The DEWE data acquisition system is then used to convert the time domain signal into a frequency response function with the use of the FFD algorithm. A 250 mm × 25 mm × 2.5 mm composite beam is then sized.

3. Results and discussion

3.1. Tensile test results

The effect of adding glass powder to co-cured glass fibre composites is then demonstrated using figures 1(a), (b). Combining glass powder with epoxy resin improved the tensile qualities of the material. Figure 1(a) shows that various weight percentages of glass powder in the matrix material help in boosting the load-carrying capability of the co-cured composites by up to 1.5%. The Results also reveal that a higher weight percentage of glass powder reduces the tensile strength and tensile modulus of the co-cured composites. Figures 1(b), (c) shows that the addition of glass powder with adhesive could increase the tensile strength value by 11.68% and it can also increase the bonding between the glass powder and the epoxy resin. Addition of excess glass powder with epoxy increases the agglomeration of glass powder and it reduces the tensile strength value.

Table 1 shows the tensile behaviour of co-cured glass fibre reinforced composites with varying glass powder weight percentages. The weight proportion of glass powder as and when it is added to the epoxy resin is shown in the first column. The table shows the average values of five samples under each kind. The load-carrying behaviour of the co-cured glass fibre composites is then improved by adding 1.5 wt% glass powder. The tensile strengths of 0.5%, 1%, 1.5% and 2% co-cured composites are 5.02%, 7.75%, 11.68% and 10.08%, increasing with respect to weight percentage of glass powder compared to neat composite. As a result, glass powder in co-cured joints increases the specimen’s tensile strength and modulus. The homogeneous dispersion of glass powder particles in the epoxy matrix is thought to be responsible for the higher tensile strength and modulus with 1.5 wt% glass powder addition. Further, it could reduce the breakage of fibre delamination through the process of better load transfer from the matrix to the fibre.

The Scanning electron microscope (SEM) images of the tensile tested specimen surfaces of co-cured composites are shown in figure 2. The SEM images of with and without glass powder in co-cured composites of tensile tested specimens clearly show that the addition of glass powder could enhance the property of the composites.

Figures 2(c), (d) shows that the tensile strength and modulus of the co-cured composites increased with the addition of 1.5 wt% of glass powder due to the uniform dispersion and good interfacial bonding between the fibre and the glass powder. Furthermore, 0 wt% co-cured composites have less interfacial bonding between the fibre and the matrix material, reducing the tensile properties of the composites and the 0.5 wt% of glass powder composites has lesser interfacial bonding as it leads to lesser tensile properties of the co-cured composites when compared to the 1.5 wt%.

3.2. Flexural test results

Figure 3(a) reveals that 1.5 wt% of glass powder co-cured composites have higher flexural strength among other combinations and 3(b) shows the flexural load and displacement of different weight percentages of glass powder
reinforced composites. The addition of glass powder into the epoxy matrix material helps in controlling the displacement values of specimens and makes it a better joint with the fibres.

The influence of glass powder with the adhesive on the flexural strength of the co-cured composites is presented in Table 2. It explains that adding glass powder to the co-cured composites could increase its subsequent failure resistance and load-carrying capacity and also improve the flexural strength up to 1.5 wt% of glass powder.

An almost similar trend is being followed in the co-cured composite in the flexural test like the tensile test. 0.5%, 1%, 1.5% and 2% of glass powder can improve the flexural strength of the composites to 7.69%, 11.5%, 25.5% and 23.5% when compared with the neat co-cured composites. Figure 3(b) reveals the flexural load-carrying capacity of co-cured composites under transverse loading condition. Reinforcement of 1.5 wt% of glass powder provides a better flexural load-carrying behaviour of the co-cured composites than other composites up.

| Glass powder wt% | Tensile strength (MPa) | Tensile modulus (GPa) | Maximum load (kN) |
|------------------|------------------------|-----------------------|-------------------|
| 0                | 321.74 (24.56)         | 9.12 (0.28)           | 35.313            |
| 0.5              | 338.22 (14.30)         | 9.85 (0.15)           | 35.549            |
| 1                | 348.98 (14.75)         | 10.17 (0.30)          | 37.193            |
| 1.5              | 364.29 (8.38)          | 11.04 (0.65)          | 37.555            |
| 2                | 357.35 (8.70)          | 9.988 (0.25)          |                   |

Table 1. Tensile test results. (standard deviation).

Figure 1. Tensile properties of glass fibre co-cured composites (a) load versus displacement (b) tensile strength and modulus versus different weight percentage of glass powder (c) tensile strength versus strain.
to 687.67 kN and an addition of 2 wt% of glass powder reduces the load-carrying behaviour of the co-cured composites.

Figures 4 (a), (b) illustrates the microstructure of glass powder reinforced co-cured composites under flexural loading conditions. Figure 4 (a) shows the evidence that the addition of 1.5 wt% of glass powder reinforcement decreases the fibre breakage and delamination of the co-cured composites under flexural loading and figure 4 (b) shows that poor bonding between the fibre and adhesive material and agglomeration of glass powder.
powder in the co-cured composites can lead to fibre breakages. Thus, it minimizes the capacity of the glass powder-enhanced co-cured composite in flexural loading. The Addition of glass powder to the epoxy can increase the flexural behaviour since glass powder transfers the flexural load to the fibre.

3.3. Single lap joint shear test results
Shear-tested samples of the co-cured composites are shown in figure 5. It shows that 0 wt% and 2 wt% co-cured samples have an adhesive failure during shear loading and 0.5 wt% glass powder reinforcement on co-cured composites reflects the higher shear strength during shear loading, which increases the resistance against failure. In co-cured composites, adding 0.5 wt% glass powder reinforcement makes a good bonding between the

![Figure 3. Flexural properties of glass fibre co-cured composites (a) flexural strength versus different weight percentage of glass powder and (b) load versus displacement.](image)

| Glass powder wt% | Flexural strength (MPa) |
|------------------|-------------------------|
| 0                | 192.76 (0.28)           |
| 0.5              | 208.92 (0.15)           |
| 1                | 217.01 (0.30)           |
| 1.5              | 256.16 (0.65)           |
| 2                | 251.19 (0.25)           |
adherent and the adhesive due to this combination of cohesive and adhesive failures. However, the addition of higher wt% of glass powder promotes adhesive failure.

In figures 6(a), (b) shear strength versus weight percentage of glass powder and load versus displacement curves are plotted. It shows that adding 0.5% of glass powder to the matrix material increases the load-carrying capacity of the single lap joint composites. Meanwhile, the neat composite specimens have less shear strength and load-carrying capacity.

From table 3, it is observed that the addition of 0.5% glass powder to the adhesive enhances the shear strength and load-carrying behaviour of the co-cured composites in shear loading in comparison with other weight percentages of glass powder reinforcement in the epoxy. However, the addition of glass powder into the matrix material can help in improving the load-carry capacity of the composites.

Researchers are also under the belief that (21, 26) the addition of higher weight percent filler materials in the epoxy reduces the shear strength of the composites. The same is being observed in table 3 wherein excess amount
of glass powder in the epoxy is utilised to reduce the shear strength of the co-cured composites than the neat co-cured composites.

The Scanning electron microscopy images of the single lap co-cured composites are shown in figures 7(a), (b). Figure 7(a) results showcase that a 2 wt% addition of glass powder in epoxy reduces the shear strength of a single lap joint. In addition, agglomeration of glass powder in the epoxy that is being caused by increasing the weight percentage of glass powder could lead to the reduction of the shear strength of the co-cured composites. From the SEM morphology of the single shear lap images of 2 wt% of glass powder addition co-cured composites, void formation is also seen to reduce the shear load carrying behaviour.

Figure 7(b) shows that adding 0.5 wt% of glass powder enhances the load-carrying property of single lap co-cured composites. Glass powder acts as a good interfacial lock with the resin and the development of uneven surfaces enhances the shear property of the specimens. Wrinkle formation in 0.5 wt% co-cured composites gives a better load-carrying behaviour over the smooth surface co-cured composites.

| Glass powder wt% | Lap shear strength (MPa) | Maximum load (N) |
|-----------------|-------------------------|------------------|
| 0               | 15.26 (0.37)            | 9848             |
| 0.5             | 19.31 (1.98)            | 12459            |
| 1               | 17.49 (1.32)            | 11289            |
| 1.5             | 13.14 (0.71)            | 8481             |
| 2               | 13.13 (0.97)            | 8476             |

Figure 6. Single lab shear test results of co-cured glass fibre composites. (a) shear strength versus different weight percentage of glass powder and (b) load versus displacement.
3.4. Free vibration test

Fundamental natural frequency and corresponding damping factor values of the co-cured composites are shown in Table 4. The dynamic behaviour of the co-cured composites is then analysed using an impulse hammer test in a fixed-free boundary condition. The experimental setup model for the modal analysis is shown in Figure 8.

From Table 4, it is found that the addition of 1.5 wt% of glass powder with the matrix materials could enhance the dynamic response of the co-cured composite. Even with the excess addition of glass powder to the epoxy, the natural frequency of the co-cured composites reduces. However, the values are comparatively higher than those of the neat epoxy composites. The Free vibrational behaviour results show that the damping behaviour of the co-cured composites with a 0.5 weight percentage of glass powder is higher than that of the other composites.

Table 4. Free vibrational characteristics of co-cured composites.

| Specimen No. | Glass powder wt% | Mode 1 | Mode 2 | Mode 3 |
|--------------|------------------|--------|--------|--------|
|              |                  | Natural frequency (Hz) | Damping factor | Natural frequency (Hz) | Damping factor | Natural frequency (Hz) | Damping factor |
| 1            | 0                | 54.6   | 0.0648 | 321.1  | 0.015  | 867.2  | 0.007  |
| 2            | 0.50             | 56.2   | 0.09   | 332.8  | 0.0094 | 881.3  | 0.0089 |
| 3            | 1                | 58.5   | 0.0498 | 349.21 | 0.005  | 942.96 | 0.0115 |
| 4            | 1.50             | 62.5   | 0.096  | 358.56 | 0.014  | 952.79 | 0.012  |
| 5            | 2                | 58.4   | 0.103  | 350    | 0.007  | 935.9  | 0.022  |
The dynamic behaviour of the co-cured composites improves due to the good bonding between the fibres and the epoxy. Thus, the addition of glass powder acts as a mechanical interlock between the fibres and the matrix. Finally, it increases the composite’s fibre breakage, crack formation and propagation during loading.

The higher addition of glass powder increases the agglomeration area in the co-cured composite, and it, in turn, increases the stress level and reduces the bonding between the fibre and the adhesive.

4. Conclusion

In the present work, a composite joint has been prepared using the co-cure manufacturing technique and then it is used to analyse the influence of the addition of glass powder on the mechanical properties, such as tensile, flexural, shear strength and free vibrational characteristics like fundamental natural frequency and corresponding damping factor. The Results showed that 1.5 wt% of glass powder addition improves the tensile strength up to 11.68% and flexural properties up to 24.75% of co-cured composite specimens due to interfacial bonding between the fibre and the epoxy resin. The addition of 0.5 wt% of glass powder enhances the shear strength of the single lap co-cured composites to 20.91% due to uniform dispersion and it improves the bonding between the adhesive and the glass powder with respect to uneven surfaces. Thus, it changes the failure mode from adhesive to cohesive failure. Adding a higher weight percent of glass powder causes the formation of agglomeration in the epoxy, which increases the local stress concentration and leads to the early failure of the single lap co-cured composites. The free vibrational test reveals that 1.5 wt% reinforcement of glass powder enhances the natural frequencies of the composites compared with the other composites and indicates that the addition of lower wt% of glass powder has a higher damping value than a higher percentage. These findings indicate that, due to the improved bonding found between the fibre and the adhesive, the inclusion of glass powder could increase the mechanical and dynamic properties of the co-cured composites by up to 1.5 wt%.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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Conflicts of interest

The authors declare no conflict of interest.

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