

Original Article

Flexural, impact and dynamic mechanical analysis of hybrid composites: Olive tree leaves powder/ pineapple leaf fibre/epoxy matrix



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ABSTRACT

The objective of this work is to determine the suitability of olive tree leaves powder (OTL) as reinforcement along with pineapple leaf fibre (PALF) in the epoxy composites. The parent and hybrid composites were fabricated using the hot press technique with the overall fibre loading at 40 wt% and OTL: PALF in the ratio of 50:50, 30:70 and 70:30, respectively. The fabricated composites were characterized using dynamic mechanical analysis (DMA), flexural and impact tests. As the proportion of PALF was increased in the hybrid composites, impact strength improved significantly. However, hybrid composites were not effective in withstanding the bending load where their bending strength and flexural modulus were lower than the parent composites. Hybrid composites outperformed parent composites with superior storage modulus, lower loss modulus and lower damping factor as per the DMA results.

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1. Introduction

Developing biobased materials has become significant in many industries owing to the need to preserve our environment. The biobased materials can be developed using biopolymers or synthetic resin reinforced with lignocellulosic fibres or synthetic fibres. Recently, the interest in using the lignocellulosic fibres is increasing rapidly due to their advantages, especially their higher specific strength and stiffness

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than glass fibre reinforced composites [1]. These fibres are found in a wide range of applications such as aerospace, automotive, marine, construction and building, biomedical, sports, and electronics [2–5].

Though the lignocellulosic fibres possess favourable properties, they have some demerits, such as water absorption, lesser thermal stability, degradation in strength, and lesser impact properties [6,7]. One way to improve the performance of composites using hybrid composites, whereby the single fibres are incorporated with several fibres into a single matrix. As a consequence, synergistic improvement can be obtained in composite properties [8-10]. Furthermore, the usage of agricultural waste residues is currently a matter of attention. Because the environmental problems can be minimized by reducing the huge waste of agricultural residues. Besides, agricultural waste residues can be an excellent alternative to plastic materials due to their various advantages such as (i) biodegradability, (ii) ease of availability, (iii) help to improve the economy of a country, and (iv) low CO2 emission [11]. The olive tree trunk that grows laterally is often pruned to allow it to grow to a larger height for its use as timber. The pruned trunk has hardly any purpose other than being used as firewood. Thus, this study attempted to use the olive trunk leaves (OTL) extracted from the trunk as reinforcement. When pulverized, OTL turns into powder form than as a fibre. Thus, it was used in the powder form as reinforcement, and their usefulness as reinforcement in the polymer composites were investigated.

Research works on using agricultural wastes as reinforcement have been well documented [12,13]. Further, agricultural wastes can be incorporated with several natural fibres and is said to improve the performance of the hybrid composites. For instance, Siti et al. [14] effectively hybridized bio-composite using bamboo-olive tree residues-epoxy matrix. Researchers reported an improvement in mechanical properties of hybridized composites than unhybridized olive tree residues/ epoxy composites [14]. It is well known that in the poultry industry, chicken feathers generate massive waste. Thus, Araya-Letelier et al. [15], used waste-based chicken weather fibres as reinforcement in adobe mixtures. It was reported that the chicken weather fibres could be used to reduce environmental challenges. Also, the chicken weather fibres effectively facilitated the improvement of the physical, damage, and durability performances of adobe mixtures. In another work, Prithivirajan et al. [16] developed bio-based composites using agricultural wastes such as rice husk and coir pitch. The researchers varied the particulate sizes from 10% to 50% and examined their mechanical performances. As expected, the mechanical results were improved by increasing the particulate sizes. Further, the researchers suggested the developed bio-based composites for low-load bearing applications. In another research work, Khristova et al. [17] utilized sunflower stalks to fabricate particle boards using tannin/ urea-formaldehyde resins. The sunflower stalks are considered agro-lignocellulosic wastes. The researchers optimized the particle board by varying the board density and resin content. Results obtained that the bending strength was higher than 16 MPa, thickness swelling, and water absorptions were less than 20% and 70%, respectively. Utilizing such raw materials would help to improve the country's economy.

In addition to the mechanical properties, determining the thermal behaviour of natural fibre reinforced composites is significant to knowing the suitability for a certain application. Sampathrajan et al. [18] developed low-density particle boards using a variety of five farm residues such as maize-cob, paddy-straw, coconut-pitch. Further, all these boards were subjected to mechanical and thermal properties. According to the results, the maize-cob board was suitable for interior applications. While the paddy-straw and coconut-pith boards were suggested for insulation purposes. Raju and Kumarappa [19] extensively studied the mechanical and thermal behaviour of groundnut shell/epoxy composites, whereby the groundnut shell can be considered agricultural waste. It was reported that the groundnut shell/epoxy composites could be promising materials for general applications.

The olive tree is botanically known as Olea europaea L [14,20]. In this work OTL was used as reinforcement. Along with this, pineapple leaf fibre (PALF) was incorporated for developing hybrid composites. Among the various natural fibres, the PALF possesses higher cellulose content (85%) [21] while the olive leaves have 11.28% [14]. Because the mechanical properties of fibres increase with increasing cellulose content [22]. Thus, hybridizing these two fibres was expected to synergistically improve the properties of developed composite materials.

Till date, the performance characteristics of OTL/PALF/ epoxy composites have never been explored as per the authors' knowledge. Thus, an attempt has been made to develop the hybrid composites using OTL and PALF as reinforcement



Fig. 1 - (a) PALF fibre and (b) OTL powder.

Table 1 – Fibre specifica	ble 1 — Fibre specifications.				
Description	OTL [14]	PALF [21]			
Density (g/cm³)	_	1.53			
Fibre diameter (µm)	250-500	800-1000			
Cellulose (%)	11.28	70.51			
Hemicellulose (%)	14.73	14.21			
Lignin (%)	16.33	2.93			
Others (%)	57.66	-			

in the epoxy matrix at different fibre weight ratios. Further, the developed composites were subjected to flexural, impact, and dynamic mechanical analysis properties.

2. Materials and method

2.1. Materials

OTL were taken from the Al-Jouf region in Saudi Arabia, and PALF was taken from a farmhouse in Malaysia. The OTL used here was obtained in the powder form in the size of $250-500 \mu m$ while raw PALF was pulverized using the ring flaker machine, and fibres of size 1-2 mm were separated with the help of a mechanical sieve (Fig. 1(a and b)). D.E.R.TM liquid epoxy resin and Jointmine hardener 905-3S were supplied by Tazdiq Engineering Sdn. Bhd., Selangor, Malaysia. The fibre specifications are presented in Table 1.

2.2. Fabrication

In this work, composites were fabricated by the hot press molding technique. Initially, OTL and PALF were mixed in the epoxy resin at different weight ratios (50:50, 30:70, and 70:30 represented as 1OTL/1PALF, 3OTL/7PALF, and 7OTL/3PALF) while the overall fibre loading was maintained to 40 wt%. The fibre and resin mixture is further mixed with hardener and placed in the 150 \times 150 \times 3 mm³ mold and cured at a temperature of 110 °C for 10 min under a pressure of 20 bar. The parent composites were also fabricated and characterized for comparison.

3. Characterization

3.1. Flexural test

Flexural test was done as per the ASTM D790 [23]with 30 kN Bluehill INSTRON 5567 and the average results of 5 specimens were reported. The crosshead displacement rate was 2 mm min⁻¹ and the span: depth was maintained to 16:1 respectively.

3.2. Impact test

Izod impact test was done using the INSTRON CEAST 9050 as per the ASTM D256-10 standard [24]. Average value of 5 specimens was reported.

3.3. DMA

DMA was carried out on the composite specimen with TA Instruments Q800 model in 3-point bending mode at a frequency of 1 Hz and a ramp rate of 5 $^{\circ}$ C min⁻¹ in the temperature range between 30 and 150 $^{\circ}$ C.



Fig. 2 - Flexural strength and modulus of PALF/OTL composites.

Table 2 – Flexural pr	operties of OTL/PALF h	ybrid composites	in comparison with the related hybrid composite works.		
Type of composite	Details of fabrication	Type of fabrication technique	Flexural properties	Observation	References
PALF/OTL/epoxy matrix hybrid composites	The PALF/OTL hybrid composites were fabricated by varying the fibre loadings in the ratio of 50:50, 30:70 and 70:30, respectively	Hot press technique	Pure pineapple leaf fibre reinforced composites exhibited higher flexural properties. In hybrid composites, the flexural properties were reduced by decreasing the fibre loading of pineapple leaf fibre.	Range of flexural strength of PALF/OTL/ epoxy matrix composites were measured between 16.41 MPa and 44.39 MPa Flexural modulus of composites observed between 2.04 GPa and 4.82 GPa	Present work
Sisal/silk/unsaturated polyester matrix hybrid composites	The composites were fabricated by varying the fibre lengths such as 1 cm, 2 cm and 3 cm. The fibre loading of hybrid composites was maintained as 1:1 (sisal:silk)	Hand lay-up	The flexural strengths were measured as 33.498 MPa, 46.181 MPa and 34.024 MPa for 1 cm, 2 cm and 3 cm fibre length hybrid composites, respectively	Higher flexural strength of hybrid composites was observed at 2 cm fibre-loaded composites	[31]
Banana/sisal/epoxy matrix hybrid composites	 Fibre length was varied from 5 mm to 20 mm (increasing order of 5 mm) Fibre weight percentage was varied from 8 to 20 	Compression moudling technique	Minimum and maximum flexural strengths were measured as 21.58 MPa and 57.53 MP at 8% and 16% of fibre-loaded composites, respectively.	The flexural strength of hybrid composites was observed to increase by 4% due to the addition of sisal fibre	[32]
Coir/silk/unsaturated polyester matrix hybrid composites	The composites were fabricated by varying the fibre lengths such as 1 cm, 2 cm and 3 cm. The fibre loading of hybrid composites was maintained as 1:1 (coir:silk)	Hand lay-up	Flexural strength of hybrid composites was measured as 37.419 MPa, 43.744 MPa and 39.692 MPa	The flexural strength of hybrid composites was increased by using 2 cm fibre-loaded composites	[33]
Sugar palm/glass fibre/ thermoplastic polyurethane	The sugar palm/glass hybrid composites were fabricated by varying the fibre loading of 30:10, 20:20 and 10:30	Melt compounding followed by hot press	Flexural strength was measured as ~ 17 MPa–31 MPa Flexural modulus was measured as ~280 MPa–~500 MPa	The flexural properties were dominated by glass fibre rather than sugar palm fibre	[34]

[32]	[30]
Optimum flexural properties were measured at a fibre loading of 1:1	The flexural modulus of hybrid composites was observed to reduce by hybridizing the fibres with the epoxy matrix
 The range of flexural strength of hybrid composites was observed between ~22 MPa. The range of flexural modulus was observed between 1900 MPa and 2200 MPa 	The range of flexural modulus of hybrid composites was measured between ~0.95 GPa and ~1.25 GPa
Compression moudling technique	Hand lay-up
The hybrid composites were fabricated by varying the fibre loadings of kenaf and pineapple leaf fibre were 30:70, 40:60, 60:40, 70:30. The total fibre loading of fibres was maintained at 40 wt%	 Hybrid composites were fabricated by varying fibre layering sequences using three different fibres: jute, hemp and flax. The total fibre loading of hybrid composites was maintained as 25% by weight.
Kenať/pineapple leať fibre/high-density polyethylene hybrid composites	Jute/hemp/flax/epoxy matrix hybrid composites

4. Results and discussion

4.1. Flexural properties

Flexural strength of PALF/epoxy, OTL/epoxy, and PALF/OTL/epoxy hybrid composites is presented in Fig. 2. The bending strength of epoxy matrix reinforced with PALF and OTL composites was increased by 69.75% and 42%, respectively, as compared to epoxy matrix. The flexural strength enhancement could be due to a reinforcing effect of PALF and OTL fillers. On the other hand, higher flexural strength was observed for PALF/epoxy composites as compared to OTL/epoxy. Thus, the PALF/epoxy composites could withstand higher stresses during flexural testing (or bending), and the strength of the composites was higher. A similar type of behaviour was observed by Maries et al. [25].

Besides that, the hybrid composites exhibited lower flexural strength than unhybridized composites. The flexural strength of hybrid composites decreased with increasing OTL content. Because the OTL fillers have an irregular shape, it could not contribute to the stress transferred to the epoxy matrix [26]. In this way, the OTL just acted as a filler rather than a better reinforcement. Furthermore, many researchers correlated the mechanical properties of natural fibres with their main constituent named, cellulose [27]. The percentage of cellulose content can influence the mechanical properties among the constituents. For instance, higher cellulose in natural fibres can give better mechanical strength [28,29]. Since OTL has a cellulose content of 11.28% over PALF, which has 70-85%, hybrid composites with a higher proportion of OTL were not effective in resisting the bending load. A similar pattern was noticed in the case of the flexural modulus of the hybrid composites shown in Fig. 2. Though the hybrid composites possessed superior flexural modulus than the epoxy, their flexural strength was comparatively lower than the epoxy. Further, the flexural strength declined with the increase in OTL proportion in the hybrid composite. This trend indicates that OTL acts as a filler improving the rigidity while it remains ineffective in handling the applied bending load. Thus, the PALF acts as a load carrier and the decrease in their fibre loading leads to inferior strength.

Table 2 shows the flexural properties of different hybrid composites compared to the present work. PALF/OTL-based hybrid composites exhibited competitive results than other reported hybrid composite works. For instance, comparing the flexural modulus of 3OTL/7PALF reinforced hybrid composites with sisal/hemp/bioepoxy hybrid composites, the values were 3.22 GPa (present study) and ~2.69 GPa [30], respectively.

4.2. Impact strength

Impact strength of PALF/epoxy, OTL/epoxy, and PALF/OTL/ epoxy hybrid composites is presented in Fig. 3. The impact strength of PALF/epoxy and hybridized composites was found to be higher than the pure epoxy matrix. Further, it was clear from Fig. 3 that increasing the content of OTL within the epoxy and PALF resulted in lower impact strength.

Besides that, the impact strength of hybridized composites was higher than OTL/epoxy composites. It happened due to the addition of PALF. When the PALF loading in hybrid



composites increased from 10 wt% to 20 wt% and 30 wt%, the increase in impact strength was 57% and 97%, respectively. Thus, hybridizing the PALF with OTL, a synergisticallyimproved impact strength was obtained.

The scanning electron micrographs of impact-tested samples are shown in Fig. 4. Fig. 4(a-e) shows the SEM images of PALF/epoxy, 7OTL/3PALF, 1OTL/1PALF, 3OTL/7PALF, and OTL. The failure of impact strength was associated with many factors: fibre pull out, matrix fracture, and fibre-tomatrix debonding. Among these factors, fibre pull-out can be the significant energy dissipation mechanism in fibrereinforced composites [25]. A strong fibre-matrix bonding and lesser fibre pull-outs are observed in Fig. 4(a). In contrast, a lack of interfacial bonding and matrix crack is observed in OTL/epoxy composites due to the deposition of waxy layers (Fig. 4(b)). The energy needed for fibre pull-out depends on the fibre diameter and length and the interfacial shear stress. The larger the fibre diameter and length, the higher the fibre pullout energy; the fibre pull-out energy also increases with an increase in interfacial shear stress [37]. The interfacial shear stress contributes to the fibre-matrix debonding; the magnitude of this shear stress depends on the relative elastic modulus of the fibre to the matrix as well as fibre aspect ratio (i.e., a ratio of fibre length to diameter). High relative elastic modulus and fibre aspect ratio could enhance the composite properties by conferring high interfacial shear stresses during elastic stress transfer [38]. Fibre-matrix debonding must be avoided as this could limit the stress transfer and, henceforth, the stress uptake in fibre (as well as in the powder). This is because the interfacial shear stress regulating the stress transfer is constant of the applied load during plastic loading following fibre-matrix debonding [39]. Increasing the fibre

loading of PALF within the epoxy and OTL resulted in a noticeable improvement in impact strength, as shown in Fig. 3. Because the impact strength of hybrid composites was influenced by the PALF. Thus, reductions in fibre pull-outs increased fibre-matrix bonding, and lesser fibre fractures were noticed in Fig. 4(c-e).

Table 3 shows the impact properties of existing reported hybrid composite works compared to the present work. It is observed that PALF/OTL-based hybrid composites exhibited competitive results over other hybrid-reported works.

4.3. Dynamic mechanical analysis

Fig. 5 illustrates the storage modulus plot for the OTL and PALF-based hybrid composites. All the composites displayed a typical trend of higher storage modulus at room temperature, whereas it was the least value at elevated temperatures. This decrease in storage modulus over the increasing temperature represents the phase transition from a glassy state to a rubbery state in the polymer composites, which usually occurs beyond the glass transition temperature (T_g) [45]. The peak storage modulus for the composites was found to be in the following order: 1OTL/1PALF > 3OTL/7PALF > 7OTL/ 3PALF > PALF > OTL. It can be noticed from the trend that all hybrid configurations had superior peak storage modulus values than the parent composites with the individual fibre. Since the storage modulus is equivalent to the stiffness or elastic modulus of the composite, higher values for the hybrid configurations are an advantage. The highest value of peak storage modulus was observed for the 1OTL/1PALF with 50:50 fibre loading, whereas as the filler weight was varied at 30:70, the peak storage modulus was significantly lower. The





superior storage modulus for the hybrid composites can also be equated to the uniform filler dispersion according to the recently published works [46–49].

Fig. 6 presents the loss modulus plot as a function of the temperature. Above T_g , the segmental motion of the polymeric chains at a molecular level is initiated. However, the segmental motion is restricted in the transition region, leading to increased loss modulus as more energy dissipates. On the other hand, a much lower force is required to dissipate energy well beyond T_g ; hence loss modulus decreases. The initial increase and the decreasing trend were prominent for the OTL composite, whereas PALF and hybrid configurations displayed a flattened curve, indicating lower energy dissipation characteristics. The energy dissipation characteristics of the hybrid composites imitated the parent laminates and were dependent on the proportion of the parent fibres in the hybrid composite. Among the hybrid composites, 10TL/1PALF

exhibited the peak loss modulus value of 223 MPa, slightly higher than the OTL (156 MPa), which was then followed by 3OTL/7PALF (122 MPa), and 7OTL/3PALF (103 MPa) with values equivalent to the PALF (112 MPa).

Peak tan delta or damping factor obtained from Fig. 7 is said to be in the following order: OTL > 1OTL/1PALF > 7OTL/3PALF > 3OTL/7PALF > PALF. It can be noticed from the trend that hybrid composites displayed a lower damping factor than OTL composites. Lower damping factor for the hybrid composites reinforced with the bamboo fibre and olive fibres from different parts of the tree, such as from leaf, big and small branches, respectively [48]. According to Saba et al. [50], a lower value indicates better interfacial bonding and uniform filler dispersion characteristics. Thus, it can be concluded that both OTL and PALF had better fibre—fibre interaction and fibre-matrix compatibility, which led to lower damping for the hybrid composites. In particular, the stress uptake in fibre during

Type of composite	Details of fabrication	Type of fabrication technique	Impact strength	Observation	References
PALF/OTL/epoxy matrix hybrid composites	The PALF/OTL hybrid composites were fabricated by varying the fibre loadings in the ratio of 50:50, 30:70 and 70:30, respectively	Hot press technique	Pure pineapple leaf fibre-reinforced composites exhibited higher impact strength. In hybrid composites, the impact strength was observed to decrease by reducing the pineapple leaf fibre content	The range of impact strength of PALF/ OTL composites ranged between 5.97 J/m to 43.40 J/m	Present work
Calotropis Gigantea Fibre/ Areca Fine Fibres/phenol formaldehyde	Fibre loading was varied for 25 wt%, 35 wt% and 45 wt%	Hand lay-up	~1.3 kJ/m ² to 1.4 kJ/m ²	At 35 wt% of fibre loading, the composites exhibited improved performance	[40]
Bamboo/jute/epoxy	Composites were fabricated by varying the fibre layering sequences	Hand lay-up	~0.10 J—0.34 J	When the bamboo fibres were used as outer skinned layers, the respective hybrid composites exhibited higher impact strength	[41]
Sisal, sorghum and jute fibres/unsaturated terephthalic polyester resin	 Fibre loading varied from 70 wt% to 95 wt% for sisal and jute fibre. Sorghum varied from 5 wt% to 30 wt%. 	Hand lay-up	20.3 J—26.3 J	The higher impact strength was observed for 40:30:30 (sisal: jute: Sorghum bicolor	[42]
Roselle fibre/sisal fibre/ polyester	Fibre loading varied from 10 wt% to 30 wt%	Compression molding technique	~1.25 kJ/m ² to ~ 1.5 kJ/m ²	No significant changes were observed by varying the fibre loading	[43]
Banana/sisal/epoxy matrix hybrid composites	 Fibre length was varied from 5 mm to 20 mm (increasing order of 5 mm) Fibre weight percentage varied from 8 to 20 	Compression molding technique	2.15 kJ/m ² to 13.25 kJ/m ²	The impact strength was increased by 35% by increasing the addition of sisal fibre	[32]
Jute/banana/epoxy matrix hybrid composites	Hybrid composites were fabricated by varying the fibre layering sequences	Hand lay-up	~50 kJ/m ² to ~ 110 kJ/m ²	When the jute fibres were used as outer skinned layers, the respective hybrid composites exhibited higher impact strength	[44]

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Fig. 5 - Storage modulus-temperature plot for OTL and PALF-based hybrid composites.

fibre—fibre interaction depends on the fibre-matrix properties, namely the relative elastic modulus of the fibre to the matrix. High relative elastic modulus confers enhanced stress transfer from the matrix to the fibre, leading to high-stress uptake in fibre [51]. Also, Tg obtained from the peak tan delta is the least

for the parent composites (96 °C for 80 °C for OTL and PALF, respectively), whereas it was approximately 150 °C for the hybrid composites. Higher T_g indicates a delay in the phase transition from glassy to rubbery state, which can be interpreted as superior thermal stability for the hybrid composites.



Fig. 6 - Loss modulus-temperature plot for OTL and PALF-based hybrid composites.



Fig. 7 – Tan delta-temperature plot for OTL and PALF-based hybrid composites.

5. Conclusion

In this work, hybrid composites reinforced with OTL and PALF at different weight ratios, such as 50:50, 30:70 and 70:30 were fabricated. Following were the conclusions from the flexural, impact, and dynamic mechanical analysis:

- The pure pineapple leaf fibre-reinforced epoxy matrix composites exhibited higher flexural strength of 44.39 MPa than other counterparts. Hybridizing the pineapple leaf fibres with olive tree leaves powder showed reductions in flexural properties. The range of flexural strength of hybrid composites was between 16.41 MPa and 28.95 MPa.
- Regarding the flexural modulus, the pure pineapple leaf fibre-reinforced epoxy matrix composites exhibited higher values, such as 4.82 GPa. The flexural modulus of hybrid composites was reduced by decreasing the fibre content of pineapple leaf fibres. The range of flexural modulus of hybrid composites was between 2.04 GPa and 3.22 GPa.
- Though the flexural strength was observed lower in hybrid composites, the flexural modulus of 3OTL/7PALF hybrid composites was 8.4% higher than olive tree leaves powder-reinforced epoxy matrix composites.
- Similarly, the impact strength of hybrid composites exhibited 3 to 8 times higher than the olive tree leaves powder-reinforced epoxy matrix composites. Thus, adding pineapple leaf fibre was beneficial, resulting in hybrid composites with better impact resistance.
- DMA results reported that hybrid composites presented higher storage modulus. The order of storage modulus of composites was 1OTL/1PALF > 3OTL/7PALF > 7OTL/3PALF > PALF > OTL.
- In loss modulus, 10TL/1PALF exhibited the value of 223 MPa, followed by 30TL/7PALF (122 MPa) and 70TL/3PALF (103 MPa) with values equivalent to the PALF (112 MPa).

 Regarding the tan delta, hybrid composites presented lower damping than the parent composites, indicating better fibre dispersion characteristics and good interfacial bonding.

The developed hybrid composites can be used in the building and construction as door panels, window frames and handrails.

Author contributions

K. Senthilkumar: Investigation, Methodology, Writing - review & editing, Formal analysis; M. Chandrasekar: Investigation, Writing - review & editing; Othman Y Alothman: Investigation, Methodology, Conceptualization, Writing - review & editing; Funding; Hassan Foud: Investigation, Writing - review & editing; Mohammad Jawaid: Conceptualization, Investigation, Methodology, Supervision, Writing - review & editing; M.A. Azeem: Investigation, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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