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Mechanical and flammability properties of ultrasonically processed silane-treated areca-banana fiber-reinforced epoxy composites for lightweight applications

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Abstract

Synthetic fiber-based polymers constitute a significant pollution source; however, their elimination is challenging due to their extensive applications. The properties of the natural fibers can be enhanced through chemical treatment. In this study, areca fibers, subjected to ultrasonic modification with silane, were utilized in various stacking sequences, and banana fibers were used without modification. The tri-layer epoxy composites were developed following four stacking sequences using a hand layup process. The mechanical, flammability, and morphological characteristics of the developed composites were analyzed. Fourier transform infrared spectroscopy results indicate the modification of fibers after silane treatment. Morphological investigations using scanning electron microscope revealed an excellent interfacial bond between the chemically treated fibers and the matrix, leading to a 15% improvement in ultimate tensile strength, 20% in hardness, 34% in ultimate flexural strength, and 18% in impact properties. This signifies the impact of surface modification on areca fibers and stacking sequence. The results showed that fiber-matrix interaction played a crucial role in controlling the performance characteristics of the developed composites.

Keywords Silane-treated areca fiber · Banana fiber · Tensile strength · Flammability · Impact strength

1 Introduction

Natural fibers have become more widely utilized, gradually replacing synthetic materials in numerous industries worldwide for various applications. The numerous advantages of

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natural fibers include their low cost, ready availability, and easily biodegradable [1-3]. Plant fibers are frequently employed in various situations and applications across different fields. The environment from which the natural fibers are sourced significantly influences their qualities [4, 5]. The conditions in which plants mature affect not only the crystalline content, such as cellulose, but also the amorphous content, which includes hemicellulose, lignin, and pectin [6]. It is imperative that natural fibers have a reduced amorphous content to minimize the need for chemical treatment. Mats made of natural fibers can be found in several forms, including bi-woven mats, mats made of single threads, and mats made of unidirectional fibers [7]. The process of combining different fiber mats with varied stacking sequences is known as hybridization and can be done in various ways. Recent ecological issues, such as rising energy demand, resource depletion, and plastic pollution buildup, have attracted scientists' interest in green composites made of biodegradable polymers and renewable resources [8, 9].

In research, Sathyaseelan et al. developed hybrid composites using areca and kenaf fibers through stacking sequence. The results revealed that the composite with kenaf as the skin layer exhibited the highest tensile and flexural strength of 17.26 MPa and 36.6 MPa, respectively [10]. James et al. previously stacked sisal and bagasse fibers in various sequences and assessed the performance properties of the developed composites. The test outcomes indicated increased properties when three sisal layers were stacked consecutively. It has also been shown that adequate interaction between the matrix and the fiber enhances various strength properties [11–13]. Similarly, Jayaraman et al. developed hybrid composites by stacking Indian almond and kenaf fiber. It was observed that kenaf/Indian almond/ kenaf epoxy composite exhibited a high storage modulus among the hybrid composites due to the presence of high stiff fibers at the skin layers [14, 15]. In another research over the effect of stacking sequence, Ganesh et al. produced composites of areca sheath and palm fiber using the hand layup method. These composites were then tested for their mechanical characteristics. The researchers concluded that composites with skin layers of areca sheath and a core layer of palm fibers exhibited superior characteristics [16]. This revealed that the chemical composition of the fiber also exhibits a significant influence over the mechanical properties along with the stacking sequence. In another research, Vinod et al. investigated the effect of stacking of jute and hemp woven fabrics. The results revealed that the presence of high strength hemp fiber as the skin layers improved the tensile and flexural strength up to 65.44 MPa and 121.20 MPa, respectively [17].

Sanjay et al. conducted research on jute, kenaf, and E-glass fibers, concluding that placing E-glass fiber on the outer layer enhanced the properties [18, 19]. Ramnath et al. developed a technique for producing hybrid composites using the hand layup method, resulting in materials composed of jute, abaca, and glass fibers [20]. They discovered that adding high-strength fiber to the central layer enhanced the characteristics. Cyprus and glass fibers were used to make hybrid composites for the final layers of the material using the hand layup method. The outcomes revealed that properties were better when jute was at the center layer, and Cyperus was the intermittent layer [21]. The natural fibers naturally exhibit impurities and amorphous constituents on the fiber surface. To remove the impurities and amorphous constituents from the fiber surface, the natural fiber was treated with an alkali solution. Alternatively, silane coupling agents were used to organize and functionalize the fiber surface, boosting the interfacial interaction between natural fibers and polymeric materials. Orue et al. examined the effect of environmental and processing variables on the mechanical, morphological, and thermal properties of PLA with sisal fiber (SF) composites [22]. They used several techniques, including NaOH treatment and silane coupling agent modifications, to alter SF. The results showed that the SF's surface became rough and thin after alkali treatment, improving interfacial adhesion between the SF and the PLA. Consequently, the interfacial compatibility of PLA/SF composites improved, leading to an enhancement in the composites' mechanical characteristics across a range of grades.

From the literature studies, it was observed that hybridization natural fibers have a significant influence over the thermal and mechanical properties of the hybrid composites [1, 4]. It was also observed that the areca fibers do not establish good interfacial adhesion between the fiber and matrix due to impurities and higher amorphous constituents. In contrast, the level of impurities and amorphous constituents of the banana fibers was comparatively lower than that of the areca fibers. Hence, the areca fibers were treated with a silane coupling agent to improve the fibermatrix interaction in the current work. Before the silane treatment, the areca fibers were pretreated with the alkali solution to remove the impurities on the fiber surface and expose more reaction sites for the silane molecules to the couple over the fiber surface.

Various composites were developed using the banana and silane-treated areca fibers through different stacking sequences. The influence of stacking sequence over the mechanical and thermal properties of the hybrid composites was evaluated according to the ASTM standard.

2 Materials and methods

2.1 Materials

Epoxy resin grade LY556 and hardener HY 951 were chosen for the current work. The resin hardener mixture was combined in a proportion of 10:1 (resin to hardener). These chemicals were supplied by HUNTSMAN India Private Limited. The 3-aminopropyl trimethoxylane silane modifier was procured from Sigma-Aldrich. Sodium hydroxide (NaOH) and ethanol were obtained from Analytical Chemicals. The areca-woven fiber mats of 300 GSM were procured from the "Go Green Composites" in Chennai, India. The banana stem fibers were extracted from the banana stem sheath.

2.1.1 Banana fiber extraction

Banana fibers were extracted from banana stem sheaths using a fiber extraction machine (Raspador). The process involved crushing the stem fibers, followed by cleaning and combing. Subsequently, the extracted fibers were dried in a hot air oven at 100 °C for 4 h to remove the moisture content.

2.1.2 Silane treatment of areca fiber

The woven mat of areca fibers was first washed in ethanol to remove the impurities present on the fiber surface. The cleaned areca fibers were then pre-treated with a 5% alkali solution to facilitate the removal of impurities and other excess amorphous constituents present over the fiber surface. After the alkali pretreatment, the fibers' pH was neutralized by washing them with distilled water. The alkali-pretreated areca fibers were then subjected to a 4% silane treatment. The silane solution was prepared by mixing 95% ethanol and 5% water, which was agitated in an ultrasonicate until a uniform aqueous solution was achieved. An aqueous solution containing 4 wt% aminopropyl trimethoxysilane (APTMS) was added and thoroughly mixed with gentle stirring. For silane treatment, the alkali-treated fibers were submerged in the silane-aqueous solution for 5 min. Finally, the ethanol was removed by baking the silane-treated areca fibers at 110 °C. Table 1 presents the chemical composition of natural fibers used in the current work.

2.2 Manufacturing of composites

The hybrid composites with stacking sequences were developed through the hand lay-up process. These composites were created with three layers, and the composite designations are presented in Table 2. Initially, a flat dry surface was selected as a mold, and a layer of mold release agent (silicone spray) was applied to facilitate easy composite separation. For the hand lay-up process, a layer of resin-hardener mixture was first applied, followed by a layer of banana fiber. Another layer of resin was then coated using a mechanical roller, followed by a layer of areca fiber. This was repeated, with the final layer being banana fiber coated with the resin-hardener mixture. Finally, a top mold coated with a releasing agent was placed over the stacked sequence, and a load of 25 kg was applied to provide compression. A similar procedure was followed for manufacturing all the samples. The overall methodology and characterization techniques of fiber and its composites are presented in Fig. 1. The fiber treatment and the composite manufacturing procedures are presented

Table 2 Composite designation and its stacking sequence

Sl. no	Designation	Stacking order of fibers
1	A-1	Banana – banana – banana
2	A-2	Areca – banana – areca
3	A-3	Areca – areca – areca
4	A-4	Banana – areca – banana

in Fig. 1(a). The characterization techniques of the fibers and their composites are presented in Fig. 1(b).

2.3 Characterization of manufactured composites

2.3.1 Tensile and Shore D hardness

The test was performed using a computerized universal testing machine (UTM) according to ASTM D638-14 with a crosshead travel speed of 1 mm/min. The machine is equipped with a 400 kN loadcell and the corresponding tensile stress vs strain was recorded using a data acquisition system.

2.3.2 Flexural strength test

The flexural strength of the developed composites was analyzed using UTM according to ASTM D790-17 with a cross-head speed of 2 mm/min. The span length of the test specimen was set to 16 times its thickness. The flexural strength was calculated according to $\sigma = 3$ fl/2bd², where *f* is the maximum load (N), *l* is the span length (mm), *b* is the breadth (mm), and *d* is the thickness (mm). The machine was employed with a strain gauge along with a computer interface to measure deflections.

2.3.3 Impact strength and hardness

The impact test was performed according to ASTM D256-10 using a computerized Zwick Roell Charpy impact tester with a force of hammer being set at 6.5 J and at an angle of 150° release. The Shore D hardness values were tested according to ASTM D2240-15.

Table 1	Chemical composition	
of areca	and banana fibers	

Chemical composition	Cellulose (wt %)	Hemicellulose (wt %)	Lignin (wt %)	Moisture (wt %)
Areca fibers	54.25	14.53	16.32	11.67
Silane-treated areca fibers	75.32	7.91	6.86	6.32
Banana fibers	69.3	13.42	7.64	10.65



(b)

Fig. 1 a Fiber treatment and composite manufacturing. b Fiber and composite testing

2.3.4 Flammability analysis

Flame testing was conducted following the procedures outlined in the UL94 standards. Using specimens measuring 125×12 mm, a UL94HB typical horizontal burn test was

conducted along the length and at 45° across the width. A blue flame was applied for a duration of 30 s at an offset of 25 mm, at an angle of 45° from the horizontal. Afterwards, the flame was put out and the duration of extinguishment, as well as the rate at which the fire burned, was observed.

2.3.5 SEM analysis

The SEM images of the developed composites were obtained using a VEGA3 TESCAN scanning electron microscope. The SEM samples were sputtered with gold prior to testing.

3 Results and discussion

3.1 FTIR analysis of banana fiber and untreated and silane-treated areca fiber

The FTIR analysis of all the treated and untreated fibers is presented in Fig. 2. The first peak observed for banana, untreated, and treated areca fibers, between 2854 and 2944 cm⁻¹, represents the stretching of O–H bonds, indicating the presence of cellulose [23]. The next consecutive infrared transmittance spectrum observed at 1718 cm⁻¹ represents the stretching of C = O, indicating the presence of hemicellulose [24]. The transmittance peak observed between 1280 and 1288 cm⁻¹ is attributed to the stretching of C-O, representing the presence of acetyl groups in lignin [25]. The transmittance spectrum observed at 1099 cm⁻¹ is attributed to the deformation vibration in the secondary group of alcohol [26]. The final strong infrared transmittance spectrum observed between 1053 cm⁻¹ and 1059 cm⁻¹ is attributed to the bending vibrations of C-O, indicating the presence of acetyl groups in lignin [27].

3.2 Tensile strength of different stacked areca/ banana fiber

The tensile properties of the composites are influenced by the orientation of the fibers within the system. The tensile strength and modulus of the developed composites are shown in Fig. 3. Among the composites, A3 has the highest ultimate tensile strength at 88.34 MPa. The areca-arecaareca sequence coated with silane exhibits higher tensile strength values than other hybrid-sequenced composites. The increase in cellulose content influenced the tensile strength of the composite [28]. When specimens were subjected to tensile loads, the topmost layer experienced the most stress, which was then transferred to subsequent layers. The efficient stress transfer was due to the increased interfacial adhesion, where the silane coupling agents established good bonding with the matrix [29]. Furthermore, the increase in cellulose content increased the tensile modulus of the A3 composite to 1.66 GPa, which was higher than the other hybrid and banana composites. The tensile strength of the hybrid samples A2 and A4 exhibited ultimate tensile strengths of 82.67 MPa and 83.29 MPa, respectively, with



Fig. 2 FTIR graph of treated and untreated areca fiber and banana fiber

tensile moduli of 1.62 GPa and 1.63 GPa, respectively. It was noted that composites with hybridized stacking sequences did not show significant differences in tensile strength. However, their tensile strength was lower than that of pure areca composites and higher than that of pure banana composites. The lower tensile strength of A2 and A4 composites may be due to the lower interfacial adhesion between the banana fibers and the epoxy matrix, resulting in inadequate stress transfer [30]. On the other hand, the lower tensile strength and modulus of the A1 composite were due to the lower interfacial adhesion of banana fibers with the matrix and the lower cellulose content compared to the silane-treated areca fibers.

3.3 Flexural strength of different stacked areca/ banana fiber

The flexural properties of the developed composites are presented in Fig. 4. During flexural testing, a complex





mechanical process occurs within the composite. When the flexural load is applied, the composite is subjected to various stresses, including tensile, shear, and compressive stresses [31]. From Fig. 4, it was noted that the flexural properties of the composites follow a similar trend to the tensile properties. Composite A3 exhibited the highest flexural strength and modulus, at 228.36 MPa and 3.63 GPa, respectively. In the flexural test, the hybrid stacking sequences showed quite different behavior. Composite A2 exhibited the highest flexural strength of 197.54 MPa compared to its counterpart A4 composite, which exhibited a flexural strength of 183.87 MPa and a modulus of 3.32 GPa. Composite A1 exhibited the lowest flexural strength and modulus, at 125.71 MPa and 2.86 GPa, respectively. It was noted that the pure areca composite showed the highest flexural strength and modulus because of the good interfacial adhesion between the fiber and matrix due to the peralkaline followed by silane treatment [32]. There was good stress transfer between the subsequent stacking layers. In the case of the A3 composite, the skin layers were made of areca fibers, which had an increased cellulose content compared to the banana fiber due to chemical treatment. During the application of flexural load, the skin layers could withstand more stress before transferring it to the core banana layers. The lower flexural strength of the A1 and A4 composites was due to improper stress transfer between the stacking layers caused by inadequate adhesion [33]. The high amorphous constituents in the banana fibers, noted in Table 1, contributed to the decreased flexural strength and modulus of the A1 composites.

3.4 Shore D hardness of different stacked areca/ banana fiber

The hardness values of the developed composites are presented in Fig. 5. Hardness reflects the material's resistance for the indenter to penetrate the sample [34]. From the hardness values, it was noted that the A3 and A4 composites exhibited hardness values in the range of 72-76. This is because the skin layers of these composites comprise silane-treated areca fibers. In the silane-treated fibers, the initial phase of alkali treatment disintegrates the fiber surface, removing impurities and excess amorphous constituents [23]. This exposes more reaction sites and brings the cellulose molecules closer together, thus increasing the density and crystal size of the fiber. During the silane treatment, the silane molecules graft onto the hydroxyl groups of the fiber surface, filling the pores with graft molecules and increasing the fiber's density [35]. When these fibers are used as reinforcements in the skin layer, the hardness of the composite increases. In the case of A1 and A4 composites, the skin layers are made of banana fibers, which have lower density and higher amorphous constituents than silane-treated areca fibers. This results in lower hardness values when the indenter penetrates the sample [36].

3.5 Impact strength of different stacked areca/ banana fiber

The impact properties of the developed composites are presented in Fig. 6. The composite A1 exhibits the highest impact strength of 4.1 J/mm, the highest compared to the

Fig. 4 Flexural properties of developed composites

A4

A3

A2

A1

0

. 10 . 20

Composite Designations



Fig. 5 Shore D hardness values of the developed composites

30

. 40

Shore D Hardness

50

other composites. It was noted that the impact results of the composites did not follow the same trend as the tensile or flexural results. This is because the impact strength depends on how much energy the composite can absorb before failure [37]. From Fig. 6, it was noted that the A1 composite exhibited the highest impact strength of 4.1 J/mm, while the A3 composite exhibited the lowest impact strength of 3.22 J/ mm. The highest impact strength of the A1 composite is due to the presence of high amorphous constituents in the banana fiber, which are less stiff compared to the silane-treated areca fiber [38]. Moreover, the banana fibers could not establish good interfacial adhesion with the epoxy matrix compared

Fig. 6 Impact properties of the developed composites

to the treated areca fibers, leading to lower stiffness of the composite. Hence, during the application of impact load, the composite could absorb more energy and dissipate it before crack initiation [39]. Similarly, in the hybrid stack of the A4 composite, the skin layers were made of banana fibers, resulting in higher impact strength than other composites. In the case of A2 and A3 composites, which are reinforced with silane-treated areca fibers in the skin layer, the increased stiffness of the silane-treated areca fibers compared to banana fibers increased the brittleness of the composite. Consequently, the composite could not withstand the



Fig. 7 Flammability of the developed composites

impact load, and cracks appeared immediately as the hammer hit the composite specimen.

3.6 Flammability tests of different stacked areca/ banana fiber

The flammability test results of the developed composites are presented in Fig. 7. It was noted that chemical treatment, fiber chemical composition, and fiber-matrix adhesion play a significant role in determining the flammability properties of the composite [40]. From Fig. 7, it was noted that the flammability resistance was very low for the A1 and A4 composites. This phenomenon was mainly due to higher amorphous constituents in the fibers and improper bonding between the fibers and the matrix [41]. The lower interfacial adhesion between the fibers and the matrix allows the flame to propagate further because of the air gaps at the fiber-matrix interface. Additionally, the larger quantity of flammable amorphous constituents in the fibers, as noted in Table 1, contributes to this effect. The flammability levels of the A1 and A4 composites were 18.14 mm/min and 21 mm/min, respectively. In the case of the A2 and A3 composites, which consist of silane-treated areca fibers in their skin layers, the areca fibers had good interfacial adhesion with the epoxy matrix due to the silane treatment. Due to this higher interfacial adhesion, there were no air gaps at the fiber-matrix interface [42]. Hence, the flammability resistance was higher for the A2 and A3 composites.

3.7 Thermogravimetric analysis of the developed composites

TGA analysis was conducted to determine the composite's weight loss at various temperatures. The TGA curves of the A2 and A3 composites are presented in Fig. 8(a). They exhibit three stages of weight loss during the experimentation. The first stage occurs at a temperature range of 30-80 °C, attributed to the release of moisture. The next degradation takes place between 180 and 290 °C, corresponding to the breakdown of hemicellulose and lignin in the fiber and polymer linkages. The final stage of degradation occurs between 320 and 380 °C, which represents the degradation of cellulose and the epoxy matrix in the composites. The degradation in the A3 sample is more pronounced compared to the A2 sample. The presence of three layers of areca fiber showed higher thermal stability compared to the two layers of areca with one layer of banana in the core. This is further evidenced by



Fig. 8 Thermogravimetric analysis of A2 and A3 composites, a TGA, b DTG



Fig. 9 a-d Banana-areca-banana stacked composite

the degradation rate of the A2 fiber, which was higher compared to that of A3. The residue for the A3 sample was 17.08%, while it was 14.30% for the A2 sample. The presence of areca in all layers resulted in higher thermal stability, which can be attributed to better interfacial

adhesion between the fibers. Thus, the presence of areca in all layers led to better thermal stability than other composites manufactured. The DTG curves of the A2 and A3 samples are shown in Fig. 9(b). Both curves initially show a flat line in the range of 0-250 °C, indicating no



Fig. 10 a-d Areca-banana-areca stacked composite

significant mass loss in this region. Both materials are stable and do not decompose at these temperatures. The major peak is observed in the 350 to 450 $^{\circ}$ C range, indicating the main decomposition phases of samples A2 and

A3. For the A2 sample, there is a sharp peak, suggesting a maximum mass loss rate due to thermal degradation. In contrast, the A3 sample shows a broader peak, indicating a slower degradation rate. This also represents multi-step



Fig. 11 a-b Banana stacked composite. c-d Areca stacked composite

degradation, which is a key factor in the increased thermal resistance of the A3 sample. Thus, the A2 sample exhibits a higher degradation rate compared to the A3 sample. The obtained results also correlate with the findings of the flammability test.

3.8 Morphological characteristics of different stacked areca/banana fiber

The tensile fracture morphology of the developed composites is presented in Figs. 9, 10 and 11. The fracture



Fig. 12 Card holder box made up of A2 composite

morphology of the A4 composite is shown in Fig. 9(a–d). It was noted from the fracture morphology of the A4 composite that the silane-treated areca fibers established good interfacial adhesion between the fiber and matrix, resulting in less fiber pullout compared to the banana fibers [43]. The banana fiber layer showed fiber pullout and fiber debonding from the matrix due to lower interfacial adhesion.

The tensile fracture morphology of the developed composites is presented in Fig. 10(a-d). The composite had banana fibers as the core layer. When the tensile load was applied, fiber pullout occurred at the core layer due to the lower adhesion of banana fibers compared to the silanetreated areca fibers. The areca fibers revealed fiber tearing due to the enhanced adhesion between the fiber surface and matrix due to silane treatment [44]. This resulted in higher tensile strength compared to the A4 composite.

The tensile fracture morphology of the pure banana composite A1 is presented in Fig. 11(a and b). The fractured surface revealed a higher level of fiber-matrix debonding during the tensile fracture. This was due to higher impurities and hydrophobic substances on the banana fibers that hindered fiber-matrix adhesion. Consequently, this resulted in easy fiber pullout and debonding during the application of tensile load [45]. Figure 11(c and d) depicts the fracture morphology of the silanetreated areca-reinforced A3 composite. From the morphological analysis, it was observed that there was increased fiber-matrix interaction, and the silane-treated areca fibers were able to establish good bonding with the matrix [46]. This resulted in lower fiber pullout and debonding. Thus, when the tensile load was applied, the A3 composite was able to withstand more load.

4 Application development

The results revealed that the developed composites are highly suitable for creating lightweight materials. Typically, household and office utilities such as chairs, desks, filing cabinets, and storage bins are made from synthetic polymeric materials. To promote environmentally friendly alternatives and support conservation efforts, a visiting card holder was developed using the best-performing A2 composite, as shown in Fig. 12. This eco-friendly composite not only reduces the reliance on non-renewable resources but also minimizes the environmental footprint of the products. Although the current thickness of the composite is 3 mm, it is possible to reduce the thickness through various manufacturing techniques. This reduction would further decrease the weight of the final product, enhancing its suitability for lightweight applications. Additionally, the material's robustness and durability ensure that even at reduced thicknesses, the composites maintain their structural integrity, making them ideal for various applications beyond office utilities, such as automotive components and consumer electronics. Moreover, adopting such ecofriendly composites in manufacturing can lead to significant cost savings over time due to reduced material usage and potential tax incentives for using sustainable materials. This advancement not only benefits the environment but also offers economic advantages, making it a compelling choice for businesses aiming for sustainability and efficiency.

5 Conclusions and future directions

The current work investigated the mechanical, flammability, and morphological properties of hybrid composites influenced by stacking sequence. The mechanical results concluded that incorporating silane-treated areca fibers in a hybrid composite significantly enhanced mechanical performance due to their higher cellulose content and improved surface bonding with the matrix. The impact results showed that the A1 composite, made entirely of banana fiber, exhibited the highest impact resistance at 4.1 J/mm. This was attributed to the lower fiber-matrix adhesion and lower stiffness of the composite. The flammability study concluded that fiber-matrix adhesion plays a crucial role in the flammability behavior of the composite. The A3 composite, made entirely of silane-treated fibers, exhibited the lowest flammability rate of 18.14 mm/min due to increased interfacial adhesion and decreased amorphous constituents in the fiber. The morphological analysis revealed that the silane-treated areca fiber exhibits good interfacial adhesion with the matrix, thus enhancing the mechanical properties.

The findings indicate that epoxy hybrid composites incorporating silane-treated areca fibers exhibit higher performance and are suitable for lightweight applications. Future work will analyze the thermal properties of the developed composites using TGA (thermogravimetric analysis), TMA (thermomechanical analysis), and DMA (dynamic mechanical analysis), along with their tribological and biodegradation characteristics.

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Data availability The data supporting this study's findings are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Declarations

Ethical approval Not applicable.

Competing interests The authors declare no competing interests.

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