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Performance of bio-ethylene propylene diene monomer (bio-EPDM) foam with mixed chemical and encapsulated blowing agents

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Abstract

Bio-EPDM, produced from sugarcane-derived ethylene, is an eco-friendly alternative material that can solve environmental pollution problems while showing performances similar to neoprene, a petroleum-based synthetic rubber. In this study, the optimum foaming conditions were determined for maximizing the properties of bio-EPDM foam for application in highly functional environment-friendly diving wetsuits. The bio-EPDM foam was prepared by mixing chemical and encapsulated blowing agents in different mixing ratios, and the mechanical properties, thermal stability, and salt-water resistance of the bio-EPDM foam were evaluated. The mechanical and elastic properties of the bio-EPDM foam decreased with increasing amount of encapsulated blowing agents, whereas the thermal stability and salt-water resistance remarkably improved with increasing mixing ratio of the encapsulated blowing agent. The mechanical properties and salt-water resistance of the bio-EPDM foam prepared using the mixed blowing agents were better than those of the foam prepared using a single blowing agent. At a mixing ratio of 2.5:2.5, a foam with excellent dimensional stability was achieved without a significant reduction in mechanical properties. In addition, the physical and mechanical properties of the bio-EPDM foams were not significantly affected by the curing system; however, the dimensional stability of the foam cured with the peroxide system was superior to that of the foam cured with sulfur system. Therefore, a proper control of the mixing ratio of chemical and encapsulated blowing agents can produce foams with optimal mechanical properties and salt-water resistance for application in watersport apparels.

Keywords: Blowing agent, Salt-water resistance, Bio-EPDM, Cross-linking agent, Microcapsule

Introduction

Recently, interest in sports and leisure activities has increased due to an improvement in the income level and greater need for leisure life. As a result, there has been rapid popularization of related sports as well as growth in the marine sports apparel market. Marine sports garments require high functionality such as shock absorption performance to safeguard the human body from extreme environments. Among the many materials that are used in these garments, neoprene, a class of synthetic rubber produced from chloroprene that accounts for more than 40% of diving wetsuit materials,

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has become a popular choice owing to its mechanical strength, and aging and thermal resistance. However, there is a growing need, domestically and globally, for the development of eco-friendly materials since the disposal of such wastes causes environmental pollution with a large CO_2 footprint (Patagonia 2016).

Therefore, studies on eco-friendly elastomers such as bio-based polyurethane and chlorine-free synthetic rubber are being actively conducted. Studies on polyurethane elastomers include studies on compatibility of bio-based and biodegradable polymer blends (Imer and Pukanszky 2013), improvement in the mechanical and thermal properties of bio-based polyurethane and its composites for expansion of various applications (Lee et al. 2018; Sebastian et al. 2018; Kuranska et al. 2016; Gama et al. 2015), and performance enhancement of bio-based polyurethane foam using nanoclay additives (Pauzi et al. 2014). In addition, there has been a wide range of studies on the synthesis and composites of bio-based polyurethane, such as the compatibility of bio-based polyol and conventional petroleum-based polyol for polyurethane production (Zhang and Kessler 2015; Park and Kim 2014) and the synthesis of bio-based polyurethane foam with organic materials (Li et al. 2018).

On the other hand, studies on chlorine-free synthetic rubber have focused on EPDM, an ethylene propylene rubber developed to address the low resistance to oxygen, ozone, heat, and gas of synthetic butadiene rubber, which is composed of a diene monomer (Allen 1983). EPDM is an ethylene-propylene copolymer that contains a double bond (diene), and is thus characterized with superb shock absorption property (Fig. 1). It not only has similar properties as that of neoprene, but is also an economical elastomer with ozone and thermal resistance as well as electrical insulation; because of these reasons, it has recently attracted attention as an industrial material in the fields of electricity, buildings, and automobiles (Lee and Bae 2018). Particularly, the development of bio-based EPDM from sugarcane-derived ethylene can reduce CO_2 footprint and fossil resource dependency (Eco-friendly rubber seal 2015; Bio-based rubber 2015), making it an environment-friendly alternative to neoprene. Therefore, an intensive study on the blowing agent and foaming method is required to maximize the properties of foam made of bio-EPDM.

A blowing agent is a material that, when added into a polymer such as plastic or rubber, creates a foam through bubble generation. Among various blowing agents, chemical and encapsulated blowing agents are more commonly used. The former creates foam from CO_2 produced by the reaction of an isocyanate with a liquid such as water, whereas the latter creates foam by vaporization of liquefied hydrogen gas located at the core of an acrylic copolymer at high temperatures, which expands the capsule (Ha et al. 2014). The foam is used in vehicle interior, life jacket, shock absorber, flooring, and shoe materials owing to its properties such as high elasticity, thermal insulation, lightweight, and shock absorption after the formation of a micro foam structure.

Depending on the type of blowing agent, the decomposition temperature, amount of generated gas, and the gas discharge rate as well as the resulting property changes vary. Therefore, uniform cell formation, excellent quality, and stable productivity are important factors in foam development (Peyda et al. 2016). Unlike chemical blowing agents, which produce harmful gases upon thermal decomposition, encapsulated blowing agents do not generate gases and the surface of the produced foam is much smoother. It also creates uniform, independent foam that is easy to handle, and stable (Jonsson et al. 2010). In addition, since encapsulated blowing agents are noted for their ease of thin-foam processing and advantages in elasticity, thermal insulation, soundproofing, specific gravity degradation, and shock absorption, many attempts have been made to increase the use of microcapsules in some industries (Ha et al. 2014; Jonsson et al. 2006).

Most of the studies on chemical blowing agents have focused on determining the effect of blowing agent content in the formulation on the foaming and molding characteristics of foam and the decomposition temperature of composites (Kim and Youn 2009), or properties such as shock absorption and compression set by adjusting the additives or the mixing ratio of polymer. Such attempts were focused on maximizing the productivity through an optimal expansion ratio while obtaining properties suitable for application. However, efforts to prevent harmful gas generation or improve foam uniformity have not really been made. Meanwhile, research on microcapsule blowing agents include a basic study on the development of a lightweight foam for automobile interior materials using an expandable microcapsule (Ha et al. 2014) and a study on the characteristics of PU/MWNT foam films for electrostatic shielding (Park et al. 2012). Both the studies used a chemical blowing agent and a microcapsule blowing agent to compare the foam properties and cell morphology. However, no research has been conducted on foaming technology for the development of highly-elastic foams with improved foam uniformity and shock absorption performance that can replace neoprene.

The chemical composition of an elastomer as well as its properties such as chemical, ozone, flame, and oil resistance vary depending on the type of elastomer. In addition, a systemic research on elastomers is required for the development of eco-friendly high-elasticity materials for diving wetsuits that can protect the human body from accidents because the required performance depends on usage and use condition. Further, it is necessary to steer away from the existing productivity-oriented research and study various foaming technologies that use expandable microcapsules to obtain relatively uniform foam cells without generating harmful gases. That is, since a foam exhibits different properties depending on the type of blowing agent used for gas generation, we believe that an optimal foaming technology can be developed by appropriately adjusting the cell foaming gas generation method by mixing different blowing agents. Thus, in this study, we investigated the changes in mechanical properties, thermal stability, and the salt-water resistance of a bio-EPDM foam depending on the mixing ratio of blowing agents.

Methods

Materials

Keltan® Eco 6950C (LANXESS), a kind of bio-EPDM (ethylene propylene diene monomer), was used as the base monomer for this experiment. Keltan® Eco 6950C is produced from bio-based feedstock, wherein the ethylene used in the process is derived

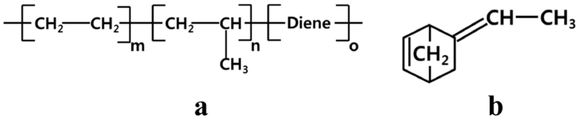

Fig. 1 Chemical structures of **a** EPDM and **b** ENB

Table 1 Specification of blowing agent

Chemical blowing agent	Trade name	Chemical composition	Decomposition temp. (°C)	Gas volume (ml/g)
	OBSH	4,4′-Oxybis (benzene-sulfonyl hydrazide)	158–163	130
Encapsulated blowing agent	**Trade name**	**Shell composition**	**Tstart (°C)**	**Tmax. (°C)**
	MSH-550	Acrylonitrile copolymer	120–135	175–185

Fig. 2 Production of bio-EPDM foam

from ethanol produced from sugarcane. Its viscosity is 65 g/10 min at 125 °C, and it is composed of 44 wt% ethylene and 9.0 wt % ENB (ethylene norbornene). ZnO and stearic acid were added to the compounds as accelerators. DCP (dicumyl peroxide) and sulfur were used as the crosslinking agents in a peroxide (PO) curing system and a sulfur curing system, respectively. OBSH (4, 4′-oxybis-(benzene sulfonyl hydrazide)-); Kumyang Co., Ltd.) was used as a chemical blowing agent, which is widely used for rubber foams for chloroprene series of wetsuits because of its relatively low decomposition temperature, excellent resistance to contamination, and non-toxicity. MSH-550 (Matsumoto, Japan) was selected as the encapsulated blowing agent. The specification of blowing agent is presented in Table 1.

Preparation of bio-EPDM foam

The chemical blowing agent and the encapsulated blowing agent were added to the compounds in the ratio of 5:1, 4:1, 3:2, 2.5:2.5, 2:3, 1:4, and 0:5, respectively. Bio-EPDM and the additives (ZnO and stearic acid) were mixed well in a kneader, a closed mixer, at 100-110 °C for 20 min in a specified mixing ratio. They were then transferred to a roll mill, which is an open mixer. Then, single and mixed blowing agents in various mixing ratios and a crosslinking agent were gradually added to the bio-EPDM mixture to form a sheet at 60-80 °C for 5-10 min. The sheet was placed in a mold of 10 mm thickness and molded at 155 °C under a high-pressure (150 kgf/cm^2) for 20 min (Fig. 2).

Characterization

Field emission scanning electron microscopy (FE-SEM, JSM6701, JEOL, Japan) was performed to evaluate the effect of blowing conditions on the microstructure of the foam. The specific gravity and compression recovery were measured according to the Korean Standard KS M 6660: (2016). Resilience was measured by ball rebound according to the Korean Standard KS M ISO 8307: (2008). Hardness test (Asker C) was performed using a digital durometer according to American Society for Testing Materials ASTM standard D2240-05: (2010). Tensile strength and elongation were measured using a UTM (MTDI UT-100F) according to the Korean Standard KS M ISO 1798: (2012) to investigate the mechanical properties of the bio-EPDM foam. The tear strength was measured by the unnicked angle tear strength method according to the Korean Standard KS M ISO 34-1: (2014). Thermal shrinkage was evaluated from the volume change of the bio-EPDM foam after heating in an oven at 70 °C for 40 min, and cooling it to room temperature for 30 min. The salt-water resistance of the bio-EPDM foam was measured by an immersion test according to Korean Standard KS M 6518: (2016). It was tested on an accelerated basis. The bio-EPDM foam was immersed in 7% and 14% NaCl solutions at 4 °C for 24 h, and the change in volume before and after immersion was recorded.

Results and discussion

Cell morphology

Figure 3 shows the cross-sectional cell shapes of the bio-EPDM foams prepared with single and mixed blowing agents with various mixing ratios using two different crosslinking systems. Regardless of the curing system, the size of the cell differs depending on the type of blowing agent. When only the encapsulated blowing agent was used, larger and uniform cells were obtained, whereas much smaller cells were obtained when only the chemical blowing agent was used. In the case of chemical blowing agent, bubbles generated irregularly after the foaming reaction started and the bubble size reduced due to the instability of bubbles. However, in the case of encapsulated blowing agent, it is inferred that uniform-sized cells are formed since the single capsules individually expanded during the thermal expansion reaction, thereby maintaining the shape of the capsules. When the mixed blowing agent was used, bubbles with different sizes were formed, and the number of individual large cells increased as the amount of encapsulated blowing agent increased.

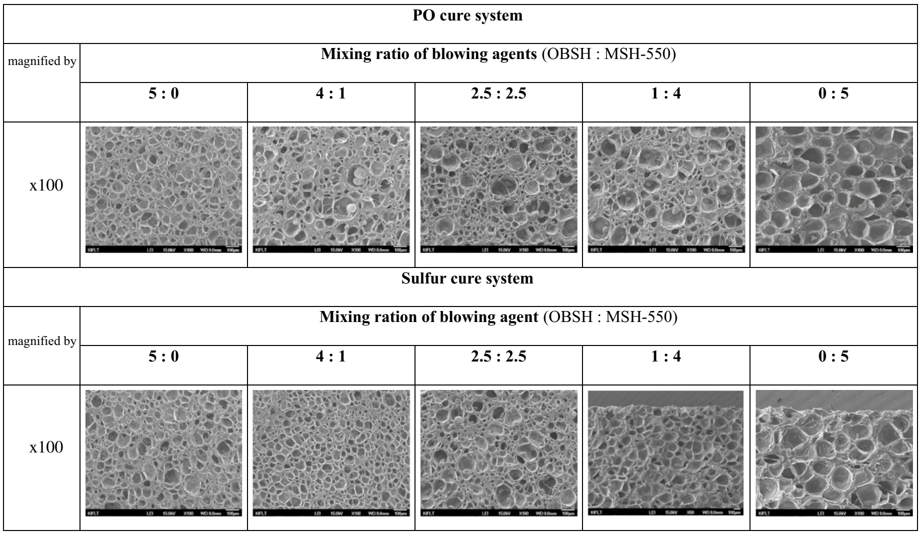

Fig. 3 SEM images of bio-EPDM foam depending on the mixing ratio of blowing agents

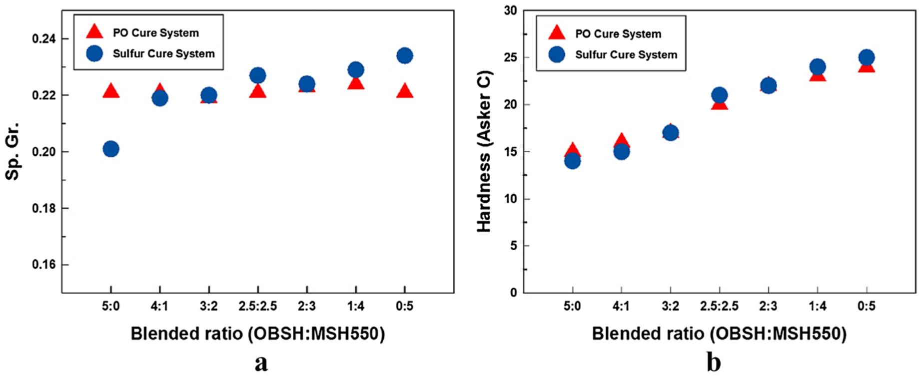

Fig. 4 Specific gravity (**a**) and hardness (**b**) of bio-EPDM foam depending on the mixing ratio of blowing agents

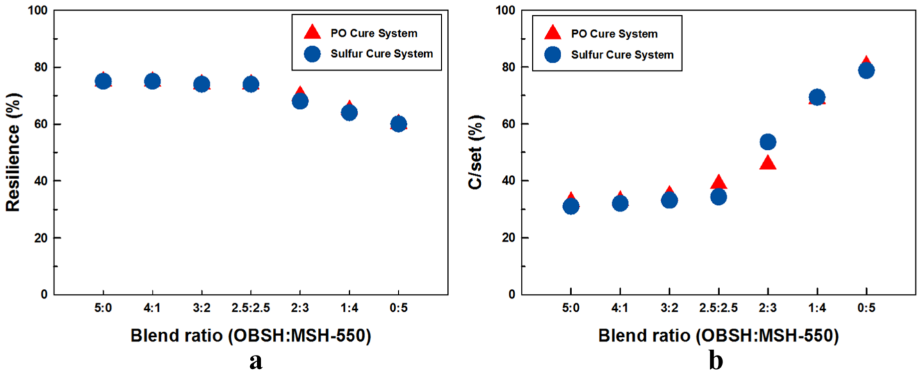

Fig. 5 Resilience (**a**) and compression recovery (**b**) of bio-EPDM foam depending on the mixing ratio of blowing agents

Similar results were obtained for the specific gravity measured depending on the mixing ratio of blowing agents. As can be seen in Fig. 4, the specific gravity of the bio-EPDM foam used in this experiment is not significantly related to the cell shape, whereas, the hardness increased as the amount of encapsulated blowing agent increased. This seems to be related to the presence of individual cell walls, which cause hardening of foam, as reported in previous studies (Park et al. 2012).

Resilience and compression set

Resilience is the ability of a material to absorb energy when deformed and release that energy upon unloading. The compression set of a material is the permanent deformation remaining when the compression force is removed. Both properties are normally tested for elastomers, and an improvement in the compression set means that the resilience is reduced. Comparing the resilience and compression set showing the elastic properties of the foam (Fig. 5), the resilience of the foam decreases slightly, while the compression set increases significantly as the amount of encapsulated blowing agent increases, especially beyond a mixing ratio of 2.5:2.5. This seems to be due to the increase of hardness and the decrease of resilience as the content of encapsulated blowing agent increases. However, there are no significant changes of resilience and compression set of EPDM were found when encapsulated blowing agent were used less than 50% of blowing agent. A similar tendency for the resilience and compression set changes is observed for two different cure systems.

The main factors determining compressive behavior are the porosity, size and shape of the inner cell, and the porosity of the porous material. The compressive behavior is sensitive to change in the internal cell with the compression load; moreover, as the porosity of the porous material increases, the compressive modulus decreases. As the size of the inner cell increases, the flow resistance of the uncompressed material decreases (Lee et al. 2017). Furthermore, as the cell shape is irregular, the degree of deformation of the cell increases with increasing porosity. Therefore, the compression set of foam increased with increasing amount of encapsulated blowing agent, thereby generating cells of a larger size. However, there are small size of pores generated by chemical blowing agent were evenly distributed in a foam when encapsulated blowing agent were used less than 50% of blowing agent. That maintains resilience and compression set of EPDM foam.

Mechanical properties

The mechanical properties of the bio-EPDM foam prepared by mixing blowing agents under different cure systems are shown in Fig. 6. Regardless of the cure system, the mechanical properties of the bio-EPDM foam are significantly affected by the mixing ratio of the blowing agent. The tensile strength, elongation, and tear strength of the bio-EPDM foam decrease with increasing amount of encapsulated blowing agent. For more than 50% of encapsulated blowing agents, the mechanical properties are significantly deteriorated. This is consistent with previous studies (Lee et al. 2017), wherein deformation or collapse occurred primarily from large-sized cells, thereby causing destruction due to the concentration of the load. Also, it seems that the foam with irregular bubble shape and mixed with various sizes influences the mechanical properties by affecting the progress speed of the crack when the bubble is impacted from the outside.

Dimensional stability (thermal shrinkage and salt-water resistance)

In order to investigate the dimensional stability of the bio-EPDM foam deformed by heat during the bonding process for wetsuit production, the size change was measured after leaving the foam in a 70 °C oven for 40 min. As shown in Fig. 7, the thermal shrinkage of the bio-EPDM foam decreases as the amount of encapsulated blowing agent increases, thereby leading to improved dimensional stability. In addition, regardless of the blowing agent, the thermal stability of EPDM foam prepared in the PO curing system is better than that in the Sulfur cure system. This result coincides with the previous study (Kim et al. 1998) in that the aging proceeds more slowly than the cross-linked EPDM by sulfur. It assumed due to the differences in binding energy of -C–C- vs -C–S- of the crosslinking structure of EPDM (Ciesielski 2000).

The salt-water resistance of the bio-EPDM foam was also tested, because it is an important factor for the performance of the wetsuit. As shown in Fig. 8, the volume change of the bio-EPDM foam with a higher mixing ratio of encapsulated blowing agent shows good dimensional stability. Particularly when the encapsulated blowing agent alone is used, the volume change is approximately 80% lower than that with the

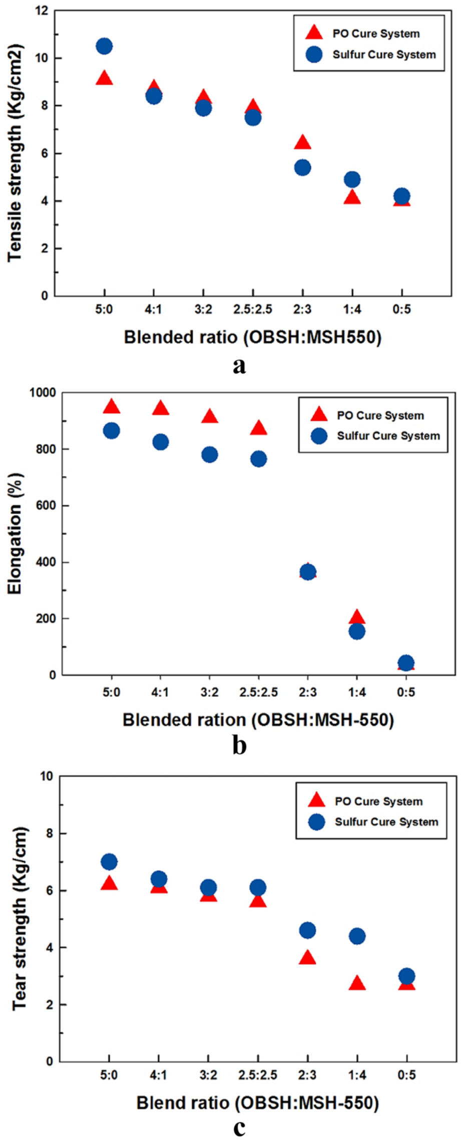

Fig. 6 Mechanical properties of bio-EPDM foam depending on the mixing ratio of blowing agents: **a** tensile strength, **b** elongation, **c** tear strength

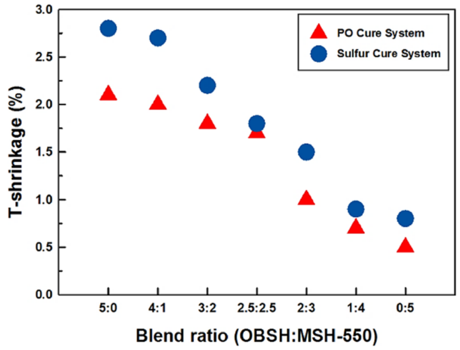

Fig. 7 Thermal shrinkage of bio-EPDM foam depending on the mixing ratio of blowing agents

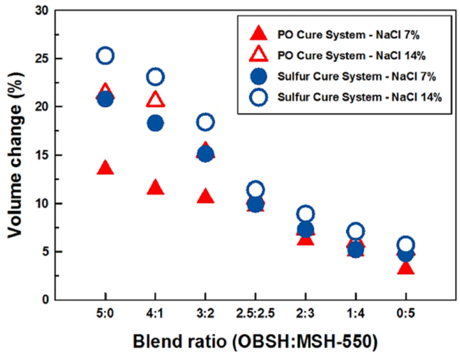

Fig. 8 Salt-water resistance of bio-EPDM foam depending on the mixing ratio of blowing agents

chemical blowing agent alone, indicating excellent dimensional stability. In addition, the salt-water resistance in the PO cure system is somewhat better than that in the sulfur cure system. Moreover, the volume change of the bio-EPDM foam increases with the concentration of the NaCl solution. The salt-water resistance of the bio-EPDM foam significantly improves when more than 50% (2.5:2.5 mixing ratio) of encapsulated blowing agent is added.

Conclusion

Bio-EPDM foam was prepared by mixing chemical blowing agents and encapsulated blowing agents at different mixing ratios, then their mechanical properties, thermal stability, and salt-water resistance were compared. The mechanical and elastic properties of the bio-EPDM foam deteriorated as the amount of encapsulated blowing agent increased over 50%. However, the thermal stability and salt-water resistance of the foam remarkably improved with increasing mixing ratio of the encapsulated blowing agents. The mechanical properties and salt-water resistance of the bio-EPDM foam produced by mixed blowing agents were better than those for the foam employing a single blowing agent. At the optimum mixing ratio of 2.5:2.5, superior dimensional stabilities of the foam were achieved without significant reduction of the mechanical properties and resilience. In addition, the physical and mechanical properties of the bio-EPDM foam were not significantly different among different cure systems; however, the dimensional stabilities of the foams cured with PO were better than those cured with sulfur. Therefore, if the mixing ratio of the chemical blowing agent and encapsulated blowing agent is controlled properly, it will be possible to produce a foam with optimal resilience mechanical properties, thermal stability, and salt-water resistance to suit the application.

Authors' contributions

JJS and KWO designed and carried out the experiments, analyzed the data, and drafted the manuscript. EYP, SWL and SJP guided and helped in the experimental work, and provided feedback and helped to prepare the final paper. All authors read and approved the final manuscript.

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Competing interests
The authors declare that they have no competing interests.

Availability of data and materials
The data sets used and analyzed during the current study are available from the corresponding author on reasonable request.

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