# Plastic/Fiber Composite Using Recycled Polypropylene and Fibers from *Sorghum halepense* L.

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Leaf and stem fibers of *Sorghum halepense* L. (SH) were investigated as fillers for recycled polypropylene (RPP) at 10, 20, and 30 wt%. The effects of SH in RPP on the thermal and mechanical properties of composites were investigated by tensile and bending tests, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC). It was found that mechanical properties of the RPP composites decreased but their elasticity modulus increased. The addition of SH fibers to the polymer matrix changed the thermal properties of RPP. A slight decrease in DSC crystallinity was observed with the addition of 10% SH leaf and stem fiber to the polymer matrix and then the percentages of crystallinity increased with the addition of 20% and 30% SH leaf and stem fiber. The SEM images showed that the SH fibers mixed well with the polymer matrix without agglomeration, and the fracture sections of the composites were less rough than that of the RPP by itself.

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Keywords: Sorghum halepense fiber; Leaf and stem fibers; Recycled polypropylene; Composite; Plant fiber

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# INTRODUCTION

The most widely produced composites contain large proportions of polypropylene (PP), poly(vinyl chloride) (PVC), and polyethylene (PE), etc., which are derived from petrochemicals and have environmental costs associated with their production. Limited oil resources and environmental concerns have led to significant research efforts in the search and evaluation of more sustainable options. Thus, biobased alternatives and biobased plastics have become of great importance. Although PP and PE are non-biodegradable materials, reports have shown that mechanical recycling of plastic waste is generally preferred over composting in terms of economic and environmental impact. Multiple rework cycles inevitably cause some degree of mechanical and thermal degradation of the polymer matrix as well as the fiber reinforcement, and repeated mechanical recycling can damage the mechanical properties of composites. However, several studies have shown that the mechanical performance of composites reinforced with natural fibers is maintained significantly better than those reinforced with glass fibers throughout rework cycles, due to the former's inherent flexibility and ability to withstand external mechanical forces. Therefore, these biocomposites exhibit better retention of properties through recycling, providing significant advantages in terms of life cycle impact and efficient waste management strategies (Uitterhaegen et al. 2018).

Natural fiber reinforced polymer composites, which are also known as natural fiber composites (NFC), have advanced significantly in recent decades to achieve sustainable manufacturing because of their relatively low cost and low density, acceptable specific properties, ease of separation, enhanced energy recovery, CO<sub>2</sub> neutrality, biodegradability, and recyclable properties (Kicinska-Jakubowska *et al.* 2012; Salit 2014). These remarkable characteristics are fueling growing demand for natural fibers in various industries such as the automotive and building sectors. Compared to synthetic fibers, such as carbon fibers and glass fibers, natural fibers have prominent advantages such as their abundance, low cost, biodegradability, flexibility during processing, low density, relatively high tensile and flexural modulus, and non-toxicity (Xie *et al.* 2010). Although biocomposites are environmentally friendly, they have disadvantages such as moisture absorption causing swelling of the fibers, susceptibility to photochemical degradation, low durability and lower impact strength (Uitterhaegen *et al.* 2018).

Considerable efforts have been made on investigating natural fiber reinforced polymer composites. These originate from plants grown in different climate zones, such as flax (More 2022) and hemp (in temperate climate zones) (Deshmukh 2022), *Sansevieria ehrenbergii* (mild to tropical climates) (Sathishkumar *et al.* 2013), palm (Khalili *et al.* 2016), kenaf (Arsad *et al.* 2013), *Cissus quadrangularis* (in tropical climate zones) (Sankarasubramanian and Pitchipoo 2021), sisal (Antich *et al.* 2006; Kim an Netravali 2010), rice straw fiber (Qin *et al.* 2011), and banana peel (tropical and subtropical climate zones) (Pereira *et al.* 2013). With adding certain amounts of these fibers from eco-friendly crops and plants, the tensile properties, flexural properties, impact resistance, and hardness of polymer composites were improved.

*Sorghum halepense* (SH) is a perennial C4 plant from the Poaceae family, which has a high tolerance and competitive ability to live in different climatic and environmental conditions, and reduces crop yield in agricultural lands. At the same time, it has become a plant species that threatens biodiversity by ranking 6<sup>th</sup> in EPPO's (European Plant Protection Organization) invasive species list for 30 different agricultural crops in 50 different countries around the world. This plant originated from the Mediterranean zone and spreads widely to Asia, Europe, North America, South America, and New Zealand. Despite its strong invasiveness in ecosystems and damage to crops, its fibers show promising mechanical properties. The strength properties of SH fibers have been demonstrated to be comparable to other non-wood fibers, and the fibers of this plant are regarded as a potential raw material for pulp and paper production (Albert *et al.* 2011).

In this study, the potential to use *Sorghum halepense* fibers as an alternative lignocellulosic source in the production of biocomposites was investigated, and composites were produced by mixing recycled polypropylene (RPP) and SH fibers. Unlike the others, this study covers the investigation of various properties of RPP composites containing SH fibers. The RPP-based composites were characterized by tensile and flexural tests, Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and scanning electron microscopy (SEM). The produced materials are highly sustainable and can be used in the automotive and building sectors to replace synthetic polymer and different natural fiber-reinforced composite materials.

# EXPERIMENTAL

## Materials

## Polypropylene and recycled polypropylene

Polypropylene (PP) was obtained from Petkim (Izmir, Turkey), and recycled polypropylene (RPP) was obtained from Polipro (Kocaeli, Turkey).

## Preparation of SH leaf and stem flour

The Sorghum halepense (SH) plants were collected from İzmir city of Turkey in June, July, August, and September. The SH leaf and stem pieces were ground in a laboratory-type grinder (LOYKA LKD 100, Akyol, İstanbul, Turkey). Then, particles were passed through a 100-mesh sieve (particles smaller than 149  $\mu$ m) and oven-dried at 103  $\pm$  2 °C until constant weight to remove moisture (Fig. 1).



Fig. 1. A) Sorghum halepense leaf fibers, B) Sorghum halepense stem fibers

## Determination of ash ratio

The amount of inorganic matter was determined in accordance with TAPPI T211 om-02 standard (2007) using a muffle furnace (Protherm PLF 110/6, Protherm Furnaces, Ankara, Turkey).

## Solvent extraction

First, SH leaf and stem flours were extracted with a mixture of toluene-acetoneethanol (4/1/1, volume/volume) for 6 h. Then, the extracted flour was kept in an oven at  $103 \pm 2$  °C until it reached completely dry weight and then cooled in a desiccator. The amounts of extractive substances were determined according to the Eq. 1,

Solubility of 4-1-1 (toluene/acetone/ethanol) =  $[(W_1 - W_2) / W_1] \times 100$  (1)

where  $W_1$  is the weight (g) of dry SH flour prior to the process, and  $W_2$  is the weight (g) of dry SH flour the after the process.

# Determination of holocellulose and alpha-cellulose

The holocellulose amounts of fiber samples prepared in this study were determined according to Wise's chloride method (Libby 1962), and the percentage of holocellulose content was determined according to the Eq. 2 below,

Holocellulose (%) =  $(W_1 / W_2) \times 100$  (2)

where  $W_1$  is the weight (g) of dry holocellulose, and  $W_2$  is the weight (g) of the dry sample. TAPPI T203 (2008) standards were used to determine the alpha-cellulose content of leaf and stem fibers. The percentage of alpha-cellulose was determined according to the Eq. 3,

Alpha cellulose (%) = 
$$S_1 / S_2 \times 100$$
 (3)

where  $S_1$  is the weight (g) of dry alpha-cellulose, and  $S_2$  is the weight (g) of dry holocellulose.

#### Production of composite materials

The SH flour at addition levels of 0, 10, 20, and 30% was mixed with the RPP polymer. The SH-filled RPP composites were produced using a high-speed thermokinetic mixer (Gelimat Mixer, Gülnar Machinery, Istanbul, Turkey). The produced sheets were coded as RPP, Leaf-10, Leaf-20, Leaf-30, Stem-10, Stem-20, and Stem-30 (Table 1). In addition, virgin PP sheets are produced with the code PP in order to compare with RPP.

The thermokinetic mixer was operated at a shaft speed of 2000 rpm until the temperature of the material reached 180 °C. About 80 g of material was put into the thermokinetic mixer for each batch, and the resulting mixing time in the melt state was about 20 s. The composite plates were obtained by a hydraulic hot (at 180 °C and under 40 to 120 bar, through 180 s) and cold (at 10 °C and under 120 bar, through 120 s) pressing, respectively.

Samples	RPP (%)	SH Fiber (%)	PP(%)
PP	-	-	100
RPP	100	-	-
Stem-10	90	10	-
Stem-20	80	20	-
Stem-30	70	30	-
Leaf-10	90	10	-
Leaf-20	80	20	-
Leaf-30	70	30	-

**Table 1.** PP, RPP Sheets, and SH/RPP Composite Formulation and Contents

#### Determination of mechanical properties

Tensile tests were conducted according to ASTM D638-14 (2014) standard with a crosshead speed of 5 mm/min. To determine the flexural properties of the samples, three-point bending tests were performed at a deformation rate of 1 mm/min according to the ASTM D790-17 (2017) standard. The tests were performed using a universal testing machine (MTS CRITERION-C45.305, MTS Systems Corporation, Eden Prairie, MN, USA).

#### Determination of thermal properties

Thermogravimetric analysis (TGA) was performed using a thermogravimetric analyzer (TA-Q600, TA Instruments Inc., New Castle, DE, USA) to determine the thermal properties of the SH filled RPP composites. The TGA was performed at a heating rate of 10 °C/min from 25 to 800 °C under nitrogen atmosphere with a flow rate of 50 mL/min.

The DSC analyses of RPP and SH filled RPP composites were performed using a DSC analyzer (TA instrument Q2000, TA Instruments Inc., New Castle, DE, USA). The DSC analysis was performed under an inert atmosphere with a nitrogen flow of 50 mL/min at a temperature range of 25 to 300 °C and a heating rate of 10 °C/min. The degree of

crystallinity (X<sub>c</sub> [%]) of the samples was calculated by using the following Eq. 4,

$$X_{\rm c} = \Delta H_m / \Delta H_m^{\circ} (1 - f_m) \times 100 \tag{4}$$

where  $\Delta H_m$  is the enthalpy of melting,  $\Delta H_m^{\circ}$  is the enthalpy of melting for RPP(190 J/g) and PP (209 J/g), and  $f_m$  is the mass fraction of feathers incorporated in the blend.

#### FTIR analysis of SH leaf and stem flour

The FTIR analyses were performed using FTIR spectrometry (Shimadzu FTIR 8400s, Shimadzu Corporation, Kyoto, Japan) to determine the chemical composition change of extracted SH fibers. The analyses were performed in the wavenumber range of 400 to 4000 cm<sup>-1</sup>.

#### Morphological characterization of SH composites

The breaking surfaces of SH filled RPP composites were coated with a thin layer of gold *via* plasma sputter coater (Quorum Q150R, Quorum Technologies, Laughton, UK). Then, the breaking surface morphology of SH filled composites was examined by using a Scanning Electron Microscope (SEM) (Carl Zeiss 300VP, Carl Zeiss Microscopy, LLC, White Plains, NY, USA).

## **RESULTS AND DISCUSSION**

#### Chemical Composition of the Sorghum halepense L.

The chemical contents of *Sorghum halepense* (SH) fibers are given in Table 2. The highest cellulose ratio was found in the samples collected in July with 34.0% for the leaf part of the plant and 38.0% for the stem part. While the highest hemicellulose ratio was detected in the leaf samples collected in August with 36.6%, the highest lignin ratio was determined in the stem samples collected in September with 31.2%. The highest ash ratio was found in the leaf samples of the plant in July with 13.7%. These differences are attributed to the anatomical structure difference arising from plant physiology. The stem and leaf of the plant are anatomically different from each other. According to the chemical analysis results, SH fibers can be an excellent alternative reinforcement/filling material for various polymers.

#### **Mechanical Properties of Composites**

Figure 2 shows the tensile strength and Young's modulus variations of PP, RPP, and RPP with the addition of SH, respectively. As shown in Fig. 2, it is clear that the tensile strength of the composite decreased as the SH content gradually increased. The addition of 30 wt% of SH fiber reduced the tensile strength of the RPP approximately 29%. This may be due to the weak interfacial bonding between the hydrophilic fiber and the hydrophobic matrix polymer. It is known that as the fiber volume used in the polymer matrix is increased, the fiber-fiber interactions and the porosity increase, which causes an ineffective stress transfer between the fiber and the matrix. This ineffective stress transfer causes deterioration failure of the composite due to increased shear stresses along the planes parallel to the fiber axis (Ashori and Nourbakhsh 2008; Ku *et al.* 2011; Rahman 2021).

As shown in Fig. 2, Young's modulus of RPP increased with increasing SH ratio. Adding 30% by weight SH to RPP increased Young's modulus approximately 84%. The compression state of the fiber in the composite material increases the fiber density and increases the elastic modulus of the material. While fibers with smaller diameter width have higher tensile modulus values than fibers with larger diameters ensures high modulus of elasticity values (Thomason 2002; Shibata *et al.* 2006). Zulkifli *et al.* (2015) revealed that Young's modulus of RPP increased with the addition of 40 wt% microcrystalline cellulose. The increase in Young's modulus can be attributed to the high stiffness of the cellulose fibers.

Months	Holocellulose (%)	α-cellulose (%)	Hemicellulose (%)	Lignin (%)	Extractives (%)	Ash (%)
LEAF	(,	(14)	(	(/-//	(14)	(,,,,
June	62.71	32.02	30.69	24.44	1.31	11.54
July	63.27	33.96	29.31	21.54	1.45	13.74
August	69.60	33.05	36.55	16.57	1.43	12.40
September	63.06	31.87	31.19	22.25	1.65	13.04
STEM						
June	63.82	35.80	28.02	29.07	1.08	6.03
July	66.40	38.05	28.35	26.06	1.28	6.26
August	68.37	33.74	34.63	24.96	1.20	5.47
September	62.11	35.09	27.02	31.15	1.47	5.27

## Table 2. Chemical Analysis Results of SH Fibers



**Fig. 2.** A) Tensile strength of PP, RPP and composites, B) Young's Modulus of PP, RPP and composites

The flexural strengths and modulus values of PP, RPP, and composite samples are depicted in Fig. 3. The addition of 10 wt% of SH fiber increased the flexural strength of the RPP. However, the addition of more than 10 wt% SH fiber to the RPP resulted in a decrease in flexural strength. Composites containing 10 wt% SH fiber showed higher flexural strength than RPP and other composites. In addition, composite samples containing 30% SH fiber exhibited the lowest bending value. In the present study, it has been observed that the flexural strength of the composite samples with added SH fiber (leaf fiber or stem fiber) decreases in general when the fiber ratio is increased more than 10%. Similarly, Mohanty *et al.* (2004) found decreases nearly 26, 13.2, and 30%. The flexural strengths of the composite samples containing 20% SH fiber and the RPP without fiber addition showed close values. Mohanty *et al.* (2004) claimed that strength decrease is usually due to the formation of matrix fiber interfacial microcracks by fiber mismatch in the matrix and non-uniform stress transfer by fiber aggregation in the matrix.

An increase in flexural modulus was observed as a result of adding up to 30% SH stem and leaf fiber to the RPP. The addition of 30% SH fiber into the RPP increased the flexural modulus from 1355 to 2205 MPa. It can be clearly seen that among SH fiber-added RPP composites, 30 wt% SH stem fiber-added RPP composite achieved the greatest improvement in terms of flexural modulus. It is known that there is an increased fiber ratio allowing higher stress transfer at the matrix and fiber interface (Mohanty *et al.* 2004).



Fig. 3. A) Flexural strength of PP, RPP and composites, B) Flexural modulus of PP, RPP and composites

## Thermal properties of samples

The TGA curves for PP, RPP, SH fiber filled RPP composites, SH leaf, and stem fibers are shown in Fig. 4. The thermal decomposition temperatures and weight loss values of samples are given in Table 3.

Samples	Weight Loss to 600 °C (%)	1st Max. Decomposition Temp. (°C)	Initial Decomposition Temp. (°C)	Final Decomposition Temp. (°C)	2nd Max. Decomposition Temp. (°C)
PP	99.00	412.16	433.60	462.56	478.97
RPP	93.55	378.88	366.58	440.15	476.05
Leaf-Fiber	84.22	257.51	226.29	309.18	358.57
Stem-Fiber	75.46	253.09	216.01	328.01	361.07
Leaf-10	84.80	327.79	318.27	446.99	480.06
Leaf-20	76.83	340.03	320.15	447.05	478.10
Leaf-30	68.41	342.65	321.91	450.17	482.17
Stem-10	84.57	329.54	319.96	446.64	479.39
Stem-20	75.77	344.23	328.68	447.28	479.29
Stem-30	68.65	351.67	329.30	447.80	480.22

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Table 5. IGA	values of PP,	RPP, C	SI FIDEIS,	and Sh/KPP	Composites

The chemical components of cellulosic fibers decompose below 400 °C. It is known that cellulose decomposes at 275 to 350 °C, hemicellulose at 150 to 350 °C, and lignin at 350 to 500 °C (Narlıoğlu 2018). According to the thermogram curves given in Fig. 4, it was seen that the addition of SH fiber to the RPP generally reduced the initial and 1<sup>st</sup> stage

maximum decomposition temperatures. In addition, it was determined that the addition of SH fiber to the RPP increased the final and 2<sup>nd</sup> stage decomposition temperatures of the samples except for the 20% SH leaf fiber added sample. According to the comparison between the composite samples, it was determined that TGA temperature values increased with the increase in the SH fiber ratio, except for the 20% SH leaf fiber added sample.

In addition, it was observed that with the addition of SH fiber to the RPP, the weight losses at the maximum temperature (600  $^{\circ}$ C) decreased in parallel with the increase in the addition rate. This has been attributed to the lower thermal stability of lignocellulosic fibers compared to plastic materials.



**Fig. 4.** TGA curves of A) SH leaf fiber, RPP composites, PP and RPP sheets and B) SH stem fiber, RPP composites, PP and RPP sheets



**Fig. 5.** DSC curves of A) SH leaf fiber, RPP composites, PP and RPP sheets and B) SH stem fiber, RPP composites, PP and RPP sheets

The  $T_m$  (melting temperature),  $X_c$  (crystallinity; %) and  $\Delta H_m$  (melting enthalpy) values of RPP and composites are presented in Table 4. As shown, a decrease in the melting enthalpy of composite materials was observed as the fiber was added to the RPP. This is because less energy is needed to melt the material with the decrease in the amount of polymer matrix (Lee *et al.* 2009). In Table 4, it can be seen that the melting enthalpies of the stem fiber added composites were slightly higher than the leaf fiber added composites. The highest melting enthalpy of 46.47 J/g was determined in RPP composite with 10% stem fiber addition. The melting peak temperatures ( $T_m$ ) of RPP in composites decreased slightly with increasing fiber content. The low melting peak was attributed to strong nucleation on the fiber surfaces, which shortens the crystallization time of RPP, and leads to the formation of smaller crystals (Pereira *et al.* 2013).

When the crystallinity percentages of the samples were examined, it was determined that as the SH fiber addition increased, the crystallinity percentages of the composite materials increased and the highest percentage of crystallinity was observed in the RPP composite with 30% stem fiber addition. This increase in crystallization percentages may be due to the natural fibers tending to act as good nucleating agents for crystallization in recycled polypropylene. Large fiber clusters formed as a result of aggregation tendencies of fiber flours with small particle sizes (0 to 420  $\mu$ m) mingled with polymer chains and supported crystallization, particle size, fiber distribution, and interfacial adhesion of the fiber to the polymer matrix significantly affect the crystallinity values (Usman *et al.* 2020).

Samples	Melting Enthalpy (J/g)	<i>T<sub>m</sub></i> (°C)	X <sub>Cr</sub> (%)
PP	60.23	163.00	28.81
RPP	52.56	162.74	25.39
Leaf-10	45.84	161.39	24.60
Leaf-20	43.14	160.57	26.05
Leaf-30	41.53	160.05	28.66
Stem-10	46.47	162.59	24.94
Stem-20	44.90	161.39	27.11
Stem-30	42.15	161.43	29.08

Table 4. DSC Values of PP, RPP, and SH/RPP Composites

#### FTIR analysis

Figure 6 shows the FTIR spectra of leaf and stem fibers of SH. The peak detected at a wavelength of  $3406 \text{ cm}^{-1}$  in the FTIR spectrum shows the presence of O-H stretching and H-bond vibration in the hydroxyl clusters of cellulose. These O-H stresses observed between  $3100 \text{ and } 3500 \text{ cm}^{-1}$  band are due to the presence of moisture and functional alcohol groups in the structure of the natural fiber (Manimekalai *et al.* 2021). The peaks of 2923 cm<sup>-1</sup> for leaf fiber and 2924 cm<sup>-1</sup> for stem fiber indicate CH (CH<sub>2</sub> and CH<sub>3</sub>) vibrations in cellulose and hemicellulose and periodic movements of functional alkyl groups (Ilaiya and Sarala 2020). The FTIR spectrum exhibited strong peaks detected at a wavelength of 1630 cm<sup>-1</sup> for leaf fiber and 1634 cm<sup>-1</sup> for stem fiber and can be attributed to the C=O stretching vibration of alpha keto carboxylic acid in lignin or the presence of ester groups in hemicellulose (Sathishkumar *et al.* 2013). When the FTIR spectrum analysis of SH fiber is evaluated in general, it is concluded that basic groups, such as ester, alcohol, carbonyl, and acid, are present in the structure of this fiber.



Fig. 6. FTIR spectra for the SH A) leaf and B) stem fibers

## Morphological properties of composites

The SEM images of 10% SH stem fiber RPP composites showing the best results in tensile and flexural strength are given in Fig. 7c-d. In the SEM images it is seen that SH fibers did not show much agglomeration. Rather, the polymer exhibited elongated structures in the form of threads, and the absence of void structure is evidence that the fiber and polymer matrix were homogeneously mixed.

The SEM images of RPP composites containing 30% stem SH fiber correspond to the best results in flexural modulus of elasticity. These are given in Figs. 7e-f. In the SEM images, it can be seen that the SH fiber and RPP polymer matrix had good mixing and that the void structures in the material decreased as the SH fiber ratio increased.



**Fig. 7.** SEM images of samples: RPP (A, B); 10 wt% SH stem fiber added composites (C, D); and 30 wt% SH stem fiber added composites (E, F)

# CONCLUSIONS

- 1. It was determined that the cellulose content of the *Sorghum halepense* L. (SH) stem was higher than that of the SH leaf. The month with the highest cellulose content was determined as July with 38.0% for the leaf and stem part of the plant. The highest hemicellulose ratio was determined in the leaf samples collected in August with 36.6% and the highest lignin ratio was determined in the stem samples collected in September with 31.2%.
- 2. The composite sample with 10% stem SH added showed the highest tensile properties compared to other composites with SH fiber addition (20% and 30% SH stem and leaf fiber added). The highest Young's and flexural modulus values of RPP composites were obtained in the sample with 30 wt% added SH.

- 3. Although slightly lower values were observed when compared to RPP, no significant effect was observed on the melting temperature with the addition of SH. The first degradation temperatures of SH fiber added composites showed up to 13% decreases compared to the control composites; this resulted in a 2% increase in the 2<sup>nd</sup> degradation temperatures (excluding the 20% leaf fiber added sample).
- 4. Except for the 10% stem and leaf fiber added RPP composites, the crystallinity values of all composites were higher than the RPP. In addition, as the fiber addition increased, it was determined that the crystallinity values of the leaf and stem groups also increased.
- 5. *Sorghum halepense* is easy to grow and is an invasive species. It has been determined that it can be a fiber source that producers can choose and benefit from for industrial branches that need mass production. Due to its high cellulose ratios, it can be used in the paper industry, and environmental and economic benefits can be provided by adding value to this plant, which is currently regarded by farmers as a "weed" or "waste" when it is present on agricultural lands.
- 6. Biocomposites produced as a result of this study, in accordance with their ideal mechanical, morphological, physical, and thermal properties, can be used in construction, automotive, furniture-decoration, various household appliances, *etc*. The composites can be an alternative for other plastics or other composite materials in current usage.
- 7. In future studies, physical and chemical modifications can be made to the fiber and polymer matrix surface and the effect of these modifications on the properties of the composite material can be investigated.

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# **CONFLICTS OF INTEREST**

The authors declare no conflict of interest.

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