

Investigation of the Effect of Hexagonal Boron Nitride Addition on the Mechanical Properties of Flax Fiber-Reinforced Composite Materials

Ahmet ERYILMAZ¹ Hasan Yavuz ÜNAL¹ Yeliz PEKBEY^{1*}

¹ Ege University, Faculty of Engineering, Department of Mechanical Engineering, İzmir, Türkiye

Keywords	Abstract
Natural Fiber-	Hexagonal boron nitride (h-BN) has recently been utilized as a reinforcement in composite materials
Reinforced Composites	due to its properties such as hardness, thermal conductivity, electrical insulation, and strong chemical
Flax Fabric	stability. The aim of this study is to investigate the effect of nano-sized hexagonal boron nitride (h-BN) on the mechanical properties of flax fiber-reinforced composite material. For this purpose, initially,
Flax-Epoxy Composites	hexagonal boron nitride was added to epoxy resin in different weight ratios and homogenized without
Vacuum Bagging	agglomeration using ultrasonic treatment. Then, by employing the hand lay-up method, the mixture was applied to flax fiber fabrics and the flax fiber-epoxy composites were produced using the vacuum
Hexagonal Boron Nitride	bagging method. Mechanical performance of the composites, produced with 0.5%, 1%, and 1.5% by weight of hexagonal boron nitride, was determined through tensile, flexural, shear, and compression tests. Experimental results indicated that the addition of hexagonal boron nitride to flax fiber epoxy composite material increased the flexural strength and modulus compared to the unreinforced flax fiber epoxy composite material. The highest flexural strength and modulus were observed in the samples with 1.5% by weight of hexagonal boron nitride (h-BN). Consequently, it can be considered that flax fiber-epoxy composite material with hexagonal boron nitride (h-BN) addition holds potential, especially for applications subjected to bending moments.

Cite

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1. INTRODUCTION

In today's technology, studies on nanotechnology continue rapidly due to its use in all kinds of applications. Boron nitride, which is an advanced technology product and is not constitute in nature, is yielded by chemical reaction of boron oxide, carbon and nitrogen at high temperatures. Since about 1950, it has been produced and then synthesized. Corrosion resistant crucibles have frequently used in engineering practices such as refractory in casting walls in applications requiring high temperature resistance, dielectric parts in electronic fields and armor technologies (Haubner et al., 2002).

Due to their different physical properties and applications, they are available in forms such as hexagonal (h-BN), cubic BN (c-BN), turbostratic BN,(t-BN), wurtzite BN (w-BN), rhombohedral BN (r-BN), and amorphous BN (a-BN) (Chen et al., 2022). The hexagonal form of boron nitride is also known as white graphene in addition to its resistance to thermal shocks at high temperatures such as about 2000°C and its electrical insulation properties. Boron nitride in cubic form is the hardest material after diamond due to its short bond and is used in the processing and cutting of hard materials, especially at high temperatures and high speeds, and to reduce wear due to its lubricity. The fatigue life of parts made of boron nitride, especially in cubic form, is high due to their resistance to high temperature and hardness. Hexagonal form boron nitride is mostly used in applications.

^{*}Corresponding Author, e-mail: yeliz.pekbey@ege.edu.tr

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Because of its excellent thermal transmittance and electrical insulation properties, hexagonal boron nitride (h-BN) is regarded a promising filler in high-voltage insulation engineering applications for polymer-based composites (Meng et al., 2014). Kusunose and Sekino (2016) found significant improvement in the thermal conductivity of materials sintered with hexagonal boron nitride in the direction of hot pressing and perpendicular to this direction. Additionally, literature supports that thermal conductivity increases in composites produced by various methods with added hexagonal boron nitride. These studies suggest that incorporating h-BN into nanocomposites can form materials capable of withstanding extreme conditions and efficiently dissipating heat, offering a promising path for thermal management applications (Wang et al., 2023; Xu et al., 2023; Liu et al., 2024; Jeong et al., 2024; Jiang et al., 2024).

Some investigations have been carried out to reduce the agglomeration of h-BN at high ratios in the resin. Li et al. (2002) investigated the machinability of Al_2O_3/BN composite ceramics with nanosized boron nitride dispersions ranging from 0 to 30% by volume by hot pressing of turbostratic BN (t-BN) coated α -Al₂O₃ powders prepared by chemical processes using boric acid. They found that nanocomposite ceramics with more than 20% boron nitride content by volume exhibited excellent machinability compared to conventional hard materials that can be drilled using metal alloy drills.

Eichler and Lesniak (2008) reviewed boron nitride (BN) and BN composites for high-temperature applications, and Karim et al. (2023) provided a literature review on h-BN-based ceramics and their composites with oxides, nitrides, carbides, and metals based on different production methods.

Wen et al. (2000) produced a BN-SiO₂ composites for high-temperature dielectric material applications via a mixture of BN and glass powders. Similarly, BN-reinforced SiO₂ (BNW/SiO₂) ceramics, varying from 5% to 20% by mass, were produced by hot pressing at temperatures of 1250°C, 1300°C, 1350°C, and 1450°C for 10 minutes and 30 MPa pressure, and their microstructure, mechanical, and dielectric properties were investigated (Duan et al., 2023).

Yang et al. (2024) investigated the thermal conductivity of epoxy-based nanocomposites added boron nitridebased nanostructures using both experimental and atomistic numerical simulations (smoothed-particle hydrodynamics (SPH)). The numerical study examined effects of nano filler volume fraction, aspect ratio, and orientation on thermal conductivity.

Boron nitride is also used in the literature as an additive in composite materials. In recent years, it has been observed that nanomaterials enhance performance of composite materials when added to composite materials (Jia et al., 2011). Zou et al. (2024) investigated mechanical and dielectric properties of Si_3N_4 -BN composite to evaluate effect of hexagonal boron nitride (h-BN) content. The study found that as the BN content increased, the relative density of Si_3N_4 -BN-MAS composites decreased from 92.3% to 79.9%, flexural strength decreased from 1000 MPa to 225 MPa, and dielectric constant decreased from 7.0 to 4.6. However, the highest fracture toughness was observed with 20% by weight BN content.

Srikhar and Omprakash (2024) examined the mechanical properties of aluminum hybrid metal matrix composites reinforced with varying weight percentages (2%, 3%, 4%, 5%, and 6%) of boron nitride (BN) produced by sintering. Composites including 5% by weight BN displayed the highest tensile strength and hardness, while composites with 4% by weight BN exhibited the highest flexural strength. This improvement in mechanical properties achieved since the BN particles within the aluminum matrix are homogeneously distrubuted acting as an effective load-bearing and reinforcing agent.

Demircan and Kalaycı (2024) investigated the shear strength of glass and carbon fiber reinforced thermoset composites by using epoxy containing different (0, 1, 2 and 3 wt%) nano hexagonal boron nitride (nano-h-BN) by weight. The shear strengths of the joints bonded with epoxy adhesives containing 3 wt% nano hexagonal boron nitride (nano-h-BN) were the highest.

To the authors' knowledge, there is no publication in the literature in which boron nitride is added as a secondary reinforcement to natural fiber reinforced composites and the mechanical properties are characterized. In this study, three different weight ratios of boron nitride nanoparticles were mixed with epoxy

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resin to produce nano-reinforced composite materials. The main purpose of this research is to ascertain the impact of boron nitride, which exhibits favorable thermal and electrical insulation characteristics, on the mechanical properties of fiber-reinforced polymers. In order to gain this objective, boron nitride was incorporated into the resin at concentrations of 0.5, 1, and 1.5% by weight. The mechanical properties of the nano-reinforced samples as well as the composite sample without reinforcement were determined by tensile, three-point bending, compression and shear tests.

2. MATERIAL AND METHOD

Flax Fiber

The dmc linen (flax) woven fabrics used as fiber reinforcement material were purchased from Bursa İpek Tekstil San. Ve Tic. Ltd. Şti. The fabric is of plain weave type (1x1) and has a mass of 400 g/m². It has a weaving density of 13 ct, representing the number of stitches per inch (2.54 cm), which corresponds to 5.12 stitches. This equates to a stitch count of 5.12 pts/cm, indicating the number of stitches per centimeter. Figure 1a shows an image of the linen fabric used as fiber in the production of composite plates. Since the obtained fabrics may contain residues, dust, and other unwanted contaminants, they were pre-washed with tap water (Figure 1b). The purpose of this pre-treatment is to make the fabric surfaces cleaner, thereby improving the adhesion between the matrix and the linen fabric. The washed linen fabrics were left to dry at room temperature (Figure 1c).



Figure 1. Flax Woven Fabric and Its Cleaning Process a) Purchased Linen Fabric; b) Washing and c) Dried Linen Fabrics

Epoxy and Hardener

Epoxy resin with a viscosity of 700-900 mPas and a density of 1.13-1.17 g/cm³, MGS Laminating Epoxy Resin L160, and a hardener with a viscosity of 10-50 mPas, MGS Laminating Epoxy Hardener H160, were used. The epoxy resin and hardener were bought from Dost Kimya.

Hexagonal Boron Nitride Powder

Hexagonal boron nitride powder with a purity greater than 99% and a particle size of 65-75 microns was obtained from Nanografi. The properties of the hexagonal boron nitride used, as detailed in Table 1, were provided by the manufacturer and supplemented by literature (Srikhar & Omprakash, 2024).

Figure 2 schematically illustrates the preparation of composite materials with epoxy resin reinforced with flax fibers and hexagonal boron nitride (h-BN) for determining their mechanical performance (Eryılmaz, 2024).

Element Anglesis (0/)	Fe ₂ O ₃	CaO	MgO	B_2O_3
Element Analysis (%)	0,03	0,002	0,04	0,1
Purity (%)	99,85			
Average Particle Size (nm)		65-7	5	
Melting Point (°C)	3000			
Boiling Point (°C)	3927			
Hardness (Knoop 100 g) (kg.mm ²)	400–3000			
Density (g/cc)	2,3			
Fracture Toughness (MPa.m ^{-1/2})	2,5–6,5			
Young Modulus (GPa)	20–103			
Electrical Conductivity (at 25°C) (S)	140			
Thermal Conductivity (at 25°C) (W/m-K)	20–27			

Table 1. Mechanical Properties of Hexagonal Boron Nitride



Figure 2. Production Stages of Flax Fiber- (h-BN) Reinforced Epoxy Resin Composites

Figure 3 shows supplementation of hexagonal boron nitride (h-BN) nanopowders in various weight ratios into epoxy resin.



Figure 3. Addition of Hexagonal Boron Nitride (h-BN) Nanopowders in Different Weight Ratios into Epoxy Resin

Ultrasonication

The process of homogeneously mixing hexagonal boron nitride was carried out using a Hielscher UP400St ultrasonic mixer. The probe of the mixer was positioned 2 cm below the surface of the resin, and mixing was performed. Ultrasonic sound waves facilitated the dispersion of hexagonal boron nitride within the resin, resulting in a homogeneous mixture. To prevent the resin from curing due to the temperature increase during the mixing process, the container was cooled with another vessel filled with water and ice (Figure 4).



Figure 4. Mixing of Hexagonal Boron Nitride into Epoxy Using an Ultrasonic Mixer

Production of Composite Plates

Flax fabric measuring $38 \times 38 \text{ cm}^2$ was placed on a release film, and the prepared resin mixture was applied with a brush until all fibers were saturated. A second layer of fabric was then placed, and the process was continued until a total of four layers were achieved. The application of the epoxy matrix prepared with a brush on the flax fabrics is shown in Figure 5.



Figure 5. Application of (h-BN) Nanopowder Reinforced Epoxy Resin onto Flax Fabrics by Hand Lay-Up

To facilitate the production process and ensure higher quality parts, a 45 x 55 cm peel ply was used (Figure 6). The peel ply placed on the top surface of the composite piece was used to remove air bubbles, enhance the even distribution of the matrix in a vacuum environment, and facilitate the easier separation of the produced plate from the system. The use of vacuum bags improved the production process by aiding in the absorption of excess resin. The vacuum blanket absorbed excess resin within the vacuum bag and reduced the amount of resin that could escape into the vacuum hose, allowing the vacuum pump to operate more safely.

After cutting the vacuum cover (nylon) to the required dimensions, spiral hoses were attached to the Tconnector fittings on the vacuum hose. Sealing tapes were placed around these hoses to prevent air leaks from the corners. Finally, the vacuum cover was sealed with sealing tape to ensure that air ingress and egress were blocked. Once the system was prepared for vacuuming, the vacuum process was initiated (Figure 6). During this process, the composite plate underwent a thermal treatment from the surface where the vacuum bagging was performed. On the recommendation of the supplier, the initial temperature was set at 60°C, maintained for eight hours. Immediately afterwards, it was allowed to cool to room temperature. The cooled composite plate was carefully removed from the vacuum bag. As a result of the production, four composite plates measuring 38×38 cm were obtained, with one being unreinforced and the other three containing hexagonal boron nitride at weight percentages of 0.5%, 1.0%, and 1.5%, respectively.



Figure 6. Production of Composite Plates Using the Vacuum Bagging Method

Standard Cutting of Plates

The plates were cut using a water jet (Figure 7). The plates were cut with high precision using a mixture of diamond and sand in addition to pure water at 3000 bar pressure, and each test specimen was cut to standard dimensions.



Figure 7. Cutting of Composite Plates to Testing Standards Using Water Jet

3. EXPERIMENTAL METHODS

To determine the mechanical performance of the produced composite plates, tensile, compressive, three-point bending, and shear tests were conducted (Figures 8-9). For each mechanical test, at least three samples were tested.

Mechanical tests were conducted according to standards using the Shimadzu Autograph AG-IS uniaxial 100 kN testing machine located at the Biomechanics Laboratory of the Department of Mechanical Engineering, Ege University. The tensile test was performed according to ASTM D3039-17. Tensile tests were performed at a rate of 2 mm/min on three samples per plate, with each sample measuring 250 mm in length and 25 mm in width. The elongation of two parallel lines drawn on the sample was monitored with a dual-camera system to obtain the unit strain value. The tensile test results determined the maximum normal stress, the unit strain at this stress, the unit strain at fracture, and the modulus of elasticity.

ASTM D7264-21 at a speed of 1 mm/min was carried out for the three-point bending test. The sample used in the tests had a width of 13 mm and a span distance between supports that was 16 times the thickness. The bending strength and unit strain values were computed using Equation 1 and Equation 2. The bending modulus was figured out slope of the stress-strain curve.

$$\sigma_{bending} = \frac{3.F.L}{2.w.t^2} \tag{1}$$

$$\varepsilon = \frac{6.\delta.t}{L^2} \tag{2}$$

In Equations (1) and (2), σ_{bending} : represents bending stress [MPa], *F*: the applied load [N], *L*: the span distance [mm], *w*: width [mm], *t*: thickness [mm] and δ : the vertical displacement [mm].



Figure 8. Tensile and Three-Point Bending Test Apparatus

The shear test was controlled according to ASTM D7078-20. At a constant speed of 2 mm/min, shear stress was applied by moving one half of the sample upward. Shear stress was calculated using the formulas in Equation 3 and Equation 4. Since strain gauges were not used in the tests, unit strain was not calculated.

$$\tau_{1} = \frac{F_{i}}{A} \tag{3}$$

$$A = w.t \tag{4}$$

In Equation 3 and Equation 4, τ_i represents shear stress at point i [MPa], F_i represents shear force at point *i* [N], *A* represents cross-sectional area [mm²], *w* represents the width [mm], and *t* represents the thickness [mm].

The compressive test was conducted according to ASTM D6641-16. Using the apparatus shown in Figure 9, only compressive stress was obtained without causing buckling. The sample dimensions were set to 140 mm in length and 13 mm in width, with a measurement distance of 13 mm. The test speed was applied at 1.3 mm/min. Compressive stress was calculated using the formulas in Equation 5 and Equation 6. Since strain gauges were not used in the tests, unit strain was not calculated.

$$\sigma_{compressive} = \frac{F_i}{A} \tag{5}$$

$$A = w.t \tag{6}$$

In Equation 5 and Equation 6, $\sigma_{\text{compression}}$ represents compressive stress at point *i* [MPa], F_i represents compressive force at point i [N], A represents cross-sectional area [mm²], w represents the width [mm], and t represents the thickness [mm].



Figure 9. Shear and Compression Test Apparatus

4. RESULTS AND DISCUSSION

Tensile Test Results

Typical results from the tensile tests of epoxy resin composites with flax fibers and hexagonal boron nitride (h-BN) nanopowder additions under constant load are depicted in Figure 10 and Table 2. For composites without hexagonal boron nitride (h-BN), the modulus of elasticity and tensile strength were 3.71 GPa and 53.02 MPa, respectively, while the addition of hexagonal boron nitride reduced the tensile properties. The modulus of elasticity showed the greatest decrease in the sample with 1% h-BN by weight. The tensile strength decreased by 36% in the sample with 0.5% h-BN by weight, and by 28% and 24% in the samples with 1% and 1.5% h-BN by weight, respectively.



Figure 10. Comparing to Modulus of Elasticity and Tensile Strengths of Epoxy Composites with Varying Weight Percentages of (h-BN)

When examining area under the typical stress-strain curve for hexagonal boron nitride (h-BN) changes in flax fiber-epoxy composites, i.e., toughness, it is observed that the highest toughness values were found in the unreinforced samples, with the 1.5% h-BN reinforced composites showing the best performance. Table 2 compares the mechanical properties of flax fiber-epoxy composites with different weight percentages of hexagonal boron nitride (h-BN).

Samples	Modulus of Elasticity (GPa)	Tensile Strength (MPa)	Max. Load (kN)	Strain (%)	Toughness (kJ/m ³)
Neat	3.71 ± 0.16	53.02 ± 1.48	6.22 ± 0.21	1.66 ± 0.04	549.89 ± 37.70
0.5% h-BN	3.42 ± 0.08	33.62 ± 3.00	4.64 ± 0.18	1.06 ± 0.14	208.40 ± 56.50
1% h-BN	3.26 ± 0.15	37.65 ± 0.91	5.33 ± 0.06	1.13 ± 0.07	249.78 ± 23.26
1.5% h-BN	3.65 ± 0.11	39.78 ± 3.61	5.43 ± 0.29	1.27 ± 0.13	303.87 ± 64.09

 Table 2. Comparison of Mechanical Properties with Variation in Weight Percentages of (h-BN) in Flax Fiber-Epoxy Composites

Three-Point Bending Test Results

The results of the three-point bending tests are presented in Figure 11 and Table 3. The addition of hexagonal boron nitride (h-BN) caused an increase the bending modulus of the flax fiber-epoxy composites, with the greatest increase observed in samples with 1.5% h-BN by weight. A similar trend was seen in bending strength. The bending modulus and bending strength increased by 24.4% and 14.7%, respectively, compared to unreinforced flax fiber-epoxy composites. The addition of hexagonal boron nitride (h-BN) reduced unit strain.

 Table 3. Comparison of Bending Properties with Variation in Weight Percentages of (h-BN) in Flax Fiber-Epoxy Composites

Samples	Flexural Modulus (GPa)	Flexural Strength (MPa)	Strain (%)
Neat	3.28 ± 0.05	73.04 ± 3.19	3.79 ± 0.42
0.5% h-BN	3.65 ± 0.20	80.36 ± 4.12	2.95 ± 0.16
1% h-BN	3.49 ± 0.14	61.37 ± 4.84	2.35 ± 0.36
1.5% h-BN	4.08 ± 0.26	83.77 ± 3.65	3.24 ± 0.22

The increase in bending strength with the addition of hexagonal boron nitride (h-BN) improved the rigidity of the epoxy while also enhancing its ductility. However, compared to unreinforced samples, tensile strengths were reduced. This reduction may be attributed to poor adhesion of hexagonal boron nitride (h-BN) within the epoxy resin and the occurrence of agglomeration during ultrasonication.

Similar results have shown in literature. In 2019, Kartal and Boztoprak produced boron nitride particle reinforced vinylester composite materials using vinyl ester resin as the matrix material and hexagonal boron nitride particles at different ratios as reinforcing material and investigated tensile strength, flexural strength, impact strength, abrasion resistance. The addition of boron nitride into vinyl ester resin started at 0.5 wt% and continued up to 2 wt%. A decrease in tensile, % elongation and bending properties of the composite material obtained was observed (Kartal & Boztoprak, 2019).

The % elongation decreases along with the tensile strength, which indicates that ductility decreases and brittleness increases with the increase in the amount of boron nitride, so the 1.5 wt% boron nitride reinforced composite material breaks more quickly due to being more brittle. In addition, the formation of agglomeration also contributed to the decrease in tensile strength. Agglomeration formation also has an effect on the decrease in tensile strength.

Although mixing was done manually and in an ultrasonic homogenizer to prevent agglomeration, agglomeration still occurred in some areas.



Figure 11. Comparison of Bending Modulus and Bending Strength with Variation in Weight Percentages of (h-BN)

Shear and Compressive Test Results

Figure 12 shows typical stress-displacement curves from shear tests. The shear strength for unreinforced flax fiber-epoxy composites was 35.66 MPa. For specimen with 0.5%, 1%, and 1.5% h-BN by weight, the shear strengths were 33.91 MPa, 32.97 MPa, and 33.44 MPa, respectively. Shear strengths decreased by approximately 4.91%, 8.05%, and 6.23% compared to unreinforced samples.



Figure 12. Shear Test Stress-Strain Curves for Flax Fiber- (h-BN) Reinforced Epoxy Resin Composites

Table 4 shows the results from the compressive tests. The compressive strength for unreinforced flax fiberepoxy composites was 75.73 MPa. The highest increase in compressive strength was 15.2% in specimen with 1% h-BN by weight. Additionally, specimen with 0.5% and 1.5% h-BN showed increases in compressive strength of 12.9% and 8.4%, respectively.

In terms of the atomic structure/array characteristics of hexagonal boron nitride, it is in the form of layers (laminar) like the structure of graphite and made up of hexagonal rings formed by boron and nitrogen atoms. There are strong covalent bonds between the atoms forming the rings (Watanabe & Taiguchi, 2011). There are Van der Waals bonds between the layers consisting of rings formed by strong covalent bonds. As shown in Table 4, in compressive strength tests, in specimens with more h-BN reinforcement by weight, more compressive strength is necessitated to break the strong covalent bonds between these layers. Therefore, compressive strength is considered to increase in compressive strength tests.

Samples	Shear Strength (MPa)	Compressive Strength (MPa)
Neat	35.66 ± 3.53	75.73 ± 4.07
0.5% h-BN	33.91 ± 3.58	85.53 ± 5.14
1% h-BN	32.97 ± 1.92	87.24 ± 3.94
1.5% h-BN	33.44 ± 0.35	82.11 ± 6.40

 Table 4. Comparison of Shear and Compression Test Properties with Variation in Weight Percentages of (h-BN) in Flax Fiber-Epoxy Composites

5. CONCLUSION

This study investigated the effects of hexagonal boron nitride (h-BN) addition on the mechanical performance of flax fiber-epoxy composite materials. For this purpose, composites containing 0.5%, 1%, and 1.5% by weight of hexagonal boron nitride (h-BN) were produced and changes in tensile, bending, shear, and compressive tests were examined. Adding hexagonal boron nitride (h-BN) at specific levels caused enhancements in bending strength and bending modulus of the composites. Composites containing 1.5% h-BN by weight exhibited the highest bending strength and bending modulus. This improvement in mechanical properties can be attributed to the homogeneous dispensation of h-BN particles within the epoxy matrix and good adhesion with the flax fibers.

However, when the h-BN content exceeded a certain weight, no further improvement in mechanical properties was observed. This is likely due to the agglomeration of h-BN nanopowders within the epoxy and their non-uniform distribution. The experimental results suggest that flax fiber-hexagonal boron nitride (h-BN) epoxy composites could meet needs for high-strength applications.

AUTHOR CONTRIBUTIONS

Conceptualization, A.E., H.Y.Ü. and Y.P.; methodology, A.E., H.Y.Ü. and Y.P.; fieldwork, A.E., H.Y.Ü. and Y.P.; software, A.E., H.Y.Ü. and Y.P.; title, A.E. and Y.P.; validation, A.E., H.Y.Ü. and Y.P.; laboratory work, A.E. and H.Y.Ü.; formal analysis, H.Y.Ü. and Y.P.; research, A.E. and Y.P.; sources, A.E., H.Y.Ü. and Y.P.; data curation, A.E. and H.Y.Ü.; manuscript-original draft, A.E. and Y.P.; manuscript-review and editing, A.E., H.Y.Ü. and Y.P.; U. and Y.P.; validation, A.E. and Y.P.; validation, A.E. and H.Y.Ü.; manuscript-original draft, A.E. and Y.P.; manuscript-review and editing, A.E., H.Y.Ü. and Y.P.; validation, H.Y.Ü. and Y.P.; validation, Y.P.; project management, Y.P.; funding, A.E., H.Y.Ü. and Y.P.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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