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RESEARCH ARTICLE / ARAŞTIRMA MAKALESI

Mechanical Performance on Flax Fibre Epoxy Composites Filled with Montmorillonite Nanoclay for Lightweight Applications

Hafiflik Özelliği Gerektiren Uygulamalar İçin Keten Fiber Esaslı Montmorillonite Nanokil Katkılı Epoksi Kompozitlerin Mekanik Performanslarının Belirlenmesi

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Abstract

The objective of this study is to investigate the mechanical performance on develop flax/epoxy composite filled with montmorillonite nanoclay for lightweight applications. For this purpose, firstly, nanoclay at different weight percentages montmorillonite nanoclay such as 0.5, 1, 1.5 was dispersed homogeneously into epoxy resin with the help of ultrasanitization process. For better nanoclay distribution in composite, the montmorillonite nanoclay concentration higher than 1.5% was not analyzed. Secondly, using this mixture, flax fiber based composites were produced by vacuum bag molding process. Finally, the mechanical properties of flax/epoxy composites filled with different percentages montmorillonite nanoclay were determined with tensile, flexural, and in-plane shear test. From the experimental results obtained, the addition of montmorillonite nanoclay indicate positive effect on the performance of the composites compared with the neat composite samples, if the montmorillonite nanoclay distribute homogeneously in the epoxy. The composites added with 0.5 wt.% nanoclay showed the highest tensile modulus and tensile strength. Moreever, the elasticity modulus of composite samples with 0.5% nanoclay addition is approximately 87% higher than the pure composite. Also, the composite samples loaded with 1.5 wt.% of montmorillonite nanoclay performs better under flexural loading conditions.

Keywords: *Biocomposite, montmorillonite nanoclay, natural fiber, mechanical performance, in-plane shear characteristics*

Öz

Bu çalışmanın amacı, hafiflik özelliği gerektiren uygulamalar için, montmorillonit nanokil ilave edilmiş keten fiber esaslı-epoksi kompozit malzemelerin mekanik performansını araştırmaktır. Bu amaca yönelik olarak, ilk olarak, 0,5, 1, 1,5 gibi farklı ağırlık yüzdelerinde montmorillonit nanokil, epoksi reçine içerisine ultrasanikasyon işlemi yardımıyla homojen bir şekilde dağılması gerçekleştirilmiştir. Epoksi reçine içerisine ilave edilen nanokil miktarı,daha iyi homojen karışım olması için %1,5 sınır değer olarak belirlenmiştir. İkinci olarak, bu karışım kullanılarak keten fiber esaslı kompozitler vakum poşet kalıplama yöntemi ile üretimi gerçekleşmiştir. Son olarak farklı yüzdelerde montmorillonit nanokil ile doldurulmuş keten/epoksi kompozitlerin mekanik özellikleri çekme, eğilme ve düzlem içi kayma testleri ile belirlenmiştir. Elde edilen deney sonuçlarında, eğer montmorillonit nanokilin epoksi içinde homojen bir şekilde dağılması durumunda, keten fiberli/epoksi kompozit malzemenin mekanik peformansının nanokil katkısız kompozite göre iyileşme sağlandığı görülmüştür. Ağırlıkça %0,5 nanokil ilave edilen kompozitler en yüksek çekme modülünü ve çekme mukavemetini göstermişlerdir. Dahası, ağırlıkça %0,5 nanokil katkılı kompozit numunelerin elastisite modülü, katkısız kompozite göre yaklaşık %87 daha yüksek elde edilmiştir. Ayrıca, ağırlıkça %1,5 montmorillonit nanokil ile yüklenen kompozit numuneler, eğilme yükleme koşulları altında daha iyi performans göstermişlerdir.

Anahtar Kelimeler: *Biokompozit, montmorillonit nanokil, doğal fiber, mekanik özellik, düzlem-içi kayma karakteristikleri*

1. Introduction

In recent years, because of global warming and boosting environmental problems, it is important the use of environmentally friendly composites. The use of plant materials such as kenaf, hemp, flax, bamboo, ramie etc. like biodegradable materials has increased in industrial areas due to their lightness and lower traces of carbon attack compared to other reinforcement elements fibers like glass and carbon. Particularly flax fiber is an environmentally friendly and sustainable material stand out as alternatives and composite materials high strength, low density when used in production and offers advantages such as superior thermal properties.

Natural fibers are recyclability, and easily disposable in nature and they also have high mechanical properties as well as light weight and minimal price [1]. It is given the examples for the natural fibers such as, flax, jute, kenaf [2]. However, the nature of plant fibers and matrix materials have drawbacks such as weak interfacial bonding moisture absorption. Natural fibers perform as hydrophobic by chemical treatments [3].

Recently, various studies have performed the composite properties by adding nanoparticles into the matrix [4-29].

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Homogeneous distribution of nanoparticles in matrix elements is important to make better properties of composites such mechanical and morphological. Flax in combination with nanoparticles serves for multifunctional composites.

It has been reported that nanoclays with plant materials have been provided benefits such as higher impact strength, fracture toughness, ballistics, impact properties [5-11].

Flax fiber has been used as filler in composites due to lightness. Flax fiber has average tensile strength ranging between 500 and 1500 MPa. Flax fiber is a lightweight fiber having a density of approximately 1.5 g/cm^3 and Young's modulus of 27.6 GPa. Due to its lightweight nature, flax fiber is used for various applications [3].

Plant materials in combination with nano particles have been used for multifunctional plant materials/matrix composites [11-15]. Due to its properties such as massive polymer nanoclay contact polymer interface and high aspect ratio, montmorillonite (MMT) is often added for dispersion in epoxy [1].

Numerous studies have shown that the addition of nanoclay and plant fiber can enhance the performance of composites. It is listed in Table 1 the latest studies on natural fiber and nano composites.

Table 1. Latest studies on natural fiber and nano composites

Year	Ref.	Type of Fiber/matrix/ Fillers	Remarks
2023	[16]	hemp fiber/flax fiber reinforced composites	The properties of composites have improved.
2023	[17]	alkali-activated concrete mixtures	The compressive strength of composite is increased.
2023	[18]	hybrid composites composed of synthetic and natural fibers	Composed of synthetic and natural fibers are used in longevity and durability practice.
2023	[19]	A extensive literature review of natural fiber reinforced polymer composites.	It was discussed the tribo mechanical characteristics of natural fiber reinforced polymer composites.
2023	[20]	kusha grass fibre, copper fibre and Emu fibre.	It has been observed that the bending strength of emu fiber reinforced composites decreases as the length and fiber load increases.
2022	[21]	jute and banana fiber as reinforcement	Tensile and bending strength were obtained in 0° fiber oriented composite material with jute fiber.
2022	[22]	woven kenaf, woven polyester and polylactic acid	The tensile, impact strength, and elongation was improved via woven kenaf.
2022	[23]	flax-basalt-glass woven fabrics	The highest tensile properties of composite was obtained for lightweight applications.
2022	[24]	silica aerogel fillers	The mechanical properties of flax composites increased addition of silica aerogel fillers.
2022	[25]	mechanical characteristics of an epoxy–clay nanocomposite.	The tensile strength of the nanocomposites improved adding treated nanoclay.
2021	[26]	banana fiber and polylactic acid as a resin	The thermal stability and mechanical properties of the polylactic acid were increased via banana fibers.
2021	[27]	sisal and palmyra palm fibers, polymers, nano-clays, and nano-clay	The mechanical properties were improved via the hybrid composites

In a previous study, we investigated the effect of nanoclay (Cloisite 30B), nano silica aerogel on the mechanical, thermal, impact and ballistic features of composites [5, 28].

The objective of this study was to improve natural flax epoxy composite with minimized weight. Montmorillonite nanoclay was chosen as nanofiller in this study for its good mechanical performance [7,9]. The change in tensile, flexural, and in-plane shear properties of flax/epoxy composites were analyzed at various weight percentages of nanoclay fillers. The mechanical performance of the composites with nanoclay was compared with pure(neat) flax epoxy composite [29]. The aim is to determine effect of nanoclay addition on mechanical performance of flax fiber/epoxy composites.

2. Materials and Methods

2.1. Materials

A flax fabric (Burs Silk Textile Company in Turkey) and resin system was purchased as the major reinforcement phase, while montmorillonite nanoclay (supplied by the Eczacıbaşı Company in Turkey) was used as the secondary reinforcement material. Figure 1 shows natural flax fiber in this study. No surface treatment has been applied to natural flax fabrics.

Figure 1. Natural flax fiber in this study

The Epoxy matrix resin (Momentive MGS L160 and its hardener (Momentive MGS H160) was also purchased by Dost Chemical Company in Turkey. The matrix material is formed with an epoxy-to-hardener weight ratio of 10:1 as per the manufacturer's specification. At 25°C, epoxy and hardener have a density of 1.13–1.17 g/cm^3 and 0.96–1.0 g/cm^3, and a viscosity of 700–900 mPa.s, 10–50 mPa.s respectively.

Table 2. Fabricated flax fiber-epoxy composites

Specimen	% nanoclay	Flax-fiber composite type
1	0	Neat flax-fiber epoxy composite
2	0.5	Flax-fiber epoxy added nanoclay composite
3	1	Flax-fiber epoxy added nanoclay composite
4	1.5	Flax-fiber epoxy added nanoclay composite

In this study, as shown in Table 2, the composites were fabricated on 4 samples (Epoxy-Neat), (99.5 wt percent Epoxy-0.5wt. percent nanoclay), (99 wt percent Epoxy-1wt percent nanoclay), (98.5 wt percent Epoxy-1.5wt. percent nanoclay).

2.2. Preparation of flax fiber-epoxy composites

It is important the procedures in manufacturing of composites due to changing properties of composites.

The preperation of composites was performed in five different stages. In the first stage, the natural flax fibers were washed to remove dirt (Figure 2).

Figure 2. Cleaning the flax fabrics

During the second stage, cleaning the flax fabricsclay was added within the epoxy via ultrasonication procedure for better distribution in epoxy. If there is no proper nanoscale dispersion, the mechanical properties of composites get worse due to fracture propagation. For this reason, it is obviously clear that the nanoclay is dispersed in the epoxy. The montmorillonite nanoclay particles and epoxy resin are blended together quickly by ultrasonic stirrer (Hielscher UP400St) with the frequency of 24 kHz for 40 min [29]. The ice bucket was used to dissipate heat since the heat was occurred during ultrasonication. The basic epoxy was formed by blending 80% resin with 20% hardener. The first mixture consisted of 99.5 wt% epoxy and 0.5 wt% of montmorillonite nanoclay.

During the third stage, the same procedure was used to make the 1% and 1.5 wt% montmorillonite nanoclay. For better nanoclay distribution in composite, the montmorillonite nanoclay concentration higher than 1.5% was not analyzed. Figure 3 shows mixing of epoxy and nanoclay via ultrasonication process.

Figure 3. Mixing of epoxy and nanoclay via ultrasonication process

Following sonication process, the hand layup followed by vacuum bag molding process were fulfilled to manufacture composite. The flax epoxy composites with nanoclay were prepared by using hand layup stacking followed by vacuum bag molding process (Figure 4). Flax fabrics in 500 mm x 500 mm dimensions were cut. The resin system was distributed on 10 layers of flax fiber by rolling it evenly to each layer using a roller.

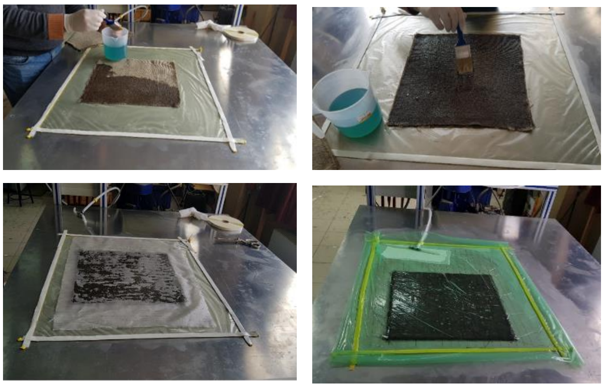

Figure 4. The fabrication of composite laminates

It was used a mould consisting of a top plate, bottom plate and two spacers. As shown in Figure4, it was used vacuum nylon and vacuum sealing tap for the mold system. The infiltration process lasted about 35 min for panels. At the end of this procedure, curing process was continued. The flow channel was surrounded by a silicon.

Finally, Tthe flax fiber-epoxy composite plates with various nanoclay were cut shape test coupons by using a CNC router accordance with ASTM guidelines for mechanical test. Figure 5 shows the cutting samples from composite plates with CNC router. There was no damage to the sample edges after CNC cutting. The thickness of specimen are 7 mm.

Figure 5. The cutting samples from composite plates with CNC router

2.3. Mechanical Performance Tests

ASTM D3039-17 and ASTM D7264-21 were used for tensile and flexural testing, respectively. The tensile and 3-point flexural tests were conducted using SHIMADZU Autograph AG-IS tensile test instrument having a load cell capacity of 100 kN. All tests have been fulfilled at room temperature.

Figure 6. Tensile, bending and shear set up for testing flax/epoxy composites

For the tensile and 3-point flexural test, four identical samples of each composite were tested, and the average value was noted. Load-displacement curves were recorded for every sample.

The tensile tests were conducted at a crosshead speed of 2 mm/min, while flexural tests were conducted at 1 mm/min.

The flexural strength (σf) was claculated bu following equation:

$$\sigma_f = \frac{3FL}{2wt^2} \quad (1)$$

where F, L,w and t indicate maximum load, span length, width and thickness of the samples, respectively. The sample size was 136 mm x 13 mm x 7 mm and the span length was 13 mm.

The in-plane shear characteristics of the fabricated flax/epoxy composites were determined using Iosipescu shear test. According to ASTM D7078-20, the dimensions of test specimens are 76 mm length and 56 mm width. A 90°double V-notches were machined at specimen mid-length through its thickness. It was cut into strips with 250 mm long and 25 mm width for tensile test specimens while it was 136 mm long and 13 mm width for flexural test specimens [29].

Figure 6 shows tensile, flexural and shear test apparatus for determining mechanical properties.

2.4. SEM analysis

The SEM analysis was fulfilled on the tested specimens to determine the distribution of montmorillonite nanoclay in composite using Thermo Scientific Apreo S scanning electron microscope with an accelerated voltage of 10 kV. All the surfaces of the samples were coated with a thin layer of gold. Figure 7 illustrates the SEM images at the 50000 mag target. As illustrated in Figure 7, it was observed that the nanoclay additive was distributed homogeneously in the composite.

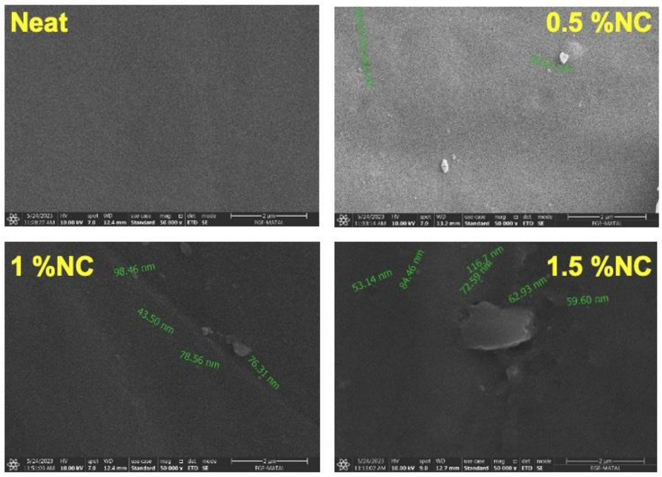

Figure 7. SEM images of nanoclay flax/epoxy composites

3. Results and Discussion

In this section the mechanical properties of montmorillonite nanoclay infused flax fiber epoxy composite have been discussed the obtained experimental results from tensile, flexural and in-plane shear tests.

In general, the mechanical properties of flax reinforced composite have been greatly enhanced by adding the montmorillonite nanoclay. It is obvious that the flax/epoxy nanoclay with 0.5 wt% has a remarkable effect on the tensile, flexural and in-plane shear strength of the composites.

3.1. Tensile properties of flax fibre epoxy composites

The tensile properties of different compositions of flax-epoxy composites are shown in Figure 8 and Table 3.

Table 3. Tensile properties of different compositions of flax-epoxy composites

	Neat	0.5 % Nanoclay	1 % Nanoclay	1.5 % Nanoclay
Tensile Modulus [GPa]	4.21 ± 0.69	5.37 ± 0.62	3.85 ± 0.22	4.03 ± 0.48
Tensile Strength [MPa]	26.81 ± 3.78	50.22 ± 3.31	40.71 ± 3.87	32.14 ± 5.29
Elongation at break [%]	1.04 ± 0.31	1.41 ± 0.21	1.65 ± 0.28	0.97 ± 0.13

It is observed that adding 0.5 wt% of nanoclay filler to composite formulation enhances flax-fiber epoxy resin composite properties including tensile and flexural. As fiber–matrix adhesion and interface bonding between the nanoclay filler increases fiber–matrix adhesion, the composites' stiffness properties is get better compare to pure composites.

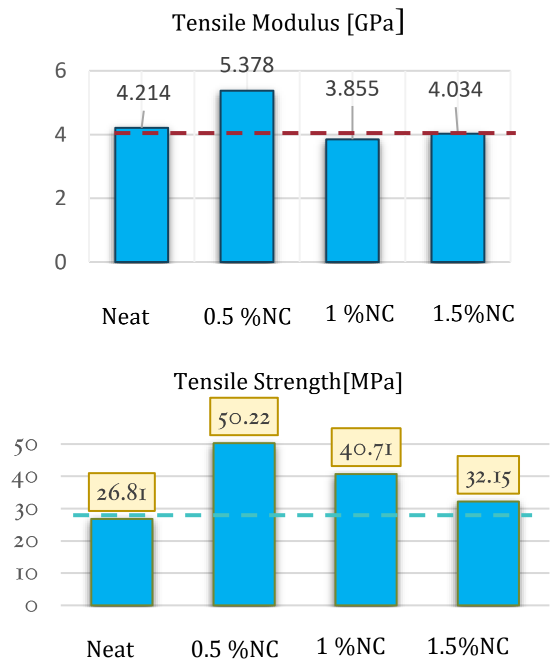

Figure 8. Tensile modulus and strength of neat and nanoclay treated flax fiber epoxy composites

As illustrated in Figure 8, the flax/epoxy nanoclay with 0.5 wt% displayed highest tensile modulus and strength properties compared to other samples. The 0.5 % nanoclay treatment yielded the highest tensile modulus and strength of 5.378 GPa and 50.22 MPa, respectively. As a result, the tensile strength of the composites increased from 26.81 MPa to 50.22 MPa. It can be seen that the concentration of montmorillonite nanoclay strongly affected tensile strength characteristics. The tensile test also showed that tensile modulus slightly increased from neat composites with 0.5 wt% nanoclay but tensile modulus decreased beyond 1 wt% which resulting in a more brittle composite. Also, the tensile modulus of flax epoxy composites with 1.5 wt% nanoclay decreased because of weak interfacial bonding.

3.2. Flexural Properties of flax fiber-epoxy composites

The flexural properties of different compositions of flax-epoxy composites are shown in Table 4. Compared to the three-point bending test results, a different trend of results for flexural modulus can be observed. The 3-point flexural test showed that flexural modulus slightly increased from neat composites to 1.5 wt% nanoclay. The Flexural Strength of neat composites increased from 69.49 MPa to 86.92 MPa when reinforced with 1.5 wt% nanoclay. The value decreased further to 72.32 MPa and 75.17 MPa when nanoclay with 0.5 wt% and 1 wt% respectively. The increasing nanoclay loading makes the flexural strength value higher, suggesting that higher concentrations of nanoclay leads to increased strength. A decrease in bending strength occurred due to the formation of voids in the matrix due to the increasing nanoclay content.

Table 4. Flexural properties of different compositions of flax-epoxy composites

	Neat	0.5 % Nanoclay	1 % Nanoclay	1.5 % Nanoclay
Flexural Modulus [MPa]	3074.10 ± 449.84	2945.73 ± 247.80	3340.55 ± 130.00	3590.17 ± 195.43
Flexural Strength [MPa]	69.49 ± 12.13	72.31 ± 4.98	75.16 ± 12	86.92 ± 6.37
Elongation at break [%]	3.91 ± 0.37	4.30 ± 0.31	3.28 ±0.29	3.31 ± 0.29

3.3. In-plane shear strength properties of flax fiber-epoxy composites

In-plane shear strength for neat and nanoclay treated composites are illustrated in Figure 9.

When comparing the results, the in-plane shear stress of neat composites increased from 17.63 MPa to 20.68 MPa when reinforced with 0.5 wt% nanoclay. The value decreased 16.77 MPa when nanoclay with 1.5 wt%.

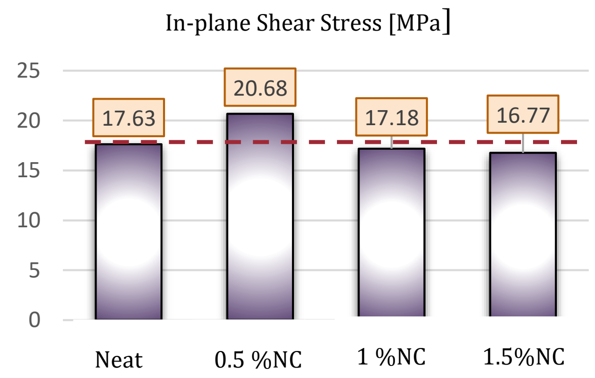

Figure 9. In-plane shear characteristics of neat and nanoclay treated flax fiber epoxy composites

Based on the obtained experimental results, Table 5 sums up different specific properties of the flax fiber epoxy composite with regard to their experimental densities.

Table 5. The percent change results obtained from the experiments

	Montmorillonite nanoclay addition (wt.%)		
	0.5 %	1%	1.5%
Tensile Strength (MPa)	87.32 ↑	51.85 ↑	19.92 ↑
Tensile Modulus (GPa)	27,62 ↑	8.32 ↓	4.28 ↓
Flexural Strength (MPa)	4.06 ↑	8.18 ↑	25.09 ↑
Flexural Modulus (GPa)	4.20 ↓	8.67 ↑	16.78 ↑
In-plane shear test (MPa)	17.30 ↑	2.56 ↓	4.88 ↓

4. Conclusions

In this study, flax-reinforced epoxy composites were infused with montmorillonite nanoclay, and the effect of nanoclay on the tensile, flexural and in-plane shear characteristics was elaborately studied. The following results were obtained:

- ✓ The tensile strength of flax-reinforced epoxy composite increased with addition of montmorillonite nanoclay. The raise in the tensile strength increased with increasing montmorillonite nanoclay in the composites. A maximum increase of 87.32% in the tensile strength was gained with addition of 0.5 wt% montmorillonite nanoclay in comparison with neat composite.
- ✓ Similar trend observed tensile modulus. At the slightly higher montmorillonite nanoclay concentration of 1.5 wt%, the tensile modulus was reduced by almost 4% compared with that of the neat composites without montmorillonite nanoclay. It is thought that reduction in tensile modulus properties of 1 and 1.5 % by weight of montmorillonite nanoclay are caused by this inappropriate dispersion.
- ✓ Reinforced with montmorillonite nanoclay flax- epoxy composites affected the flexural strength of the composites as well. The growth in the fexural modulus for the composites were about 4.06, 8.18, and 25.09% with increasing montmorillonite nanoclay 0 wt% to 0.5, 1, and 1.5 wt%, respectively.
- ✓ Despite the improvements achieved in the tensile and flexural strength with all three concentrations of montmorillonite nanoclay in composites, the reinforcing effect on tensile modulus and in-plane shear test started to deteriorate at 1 and 1.5 wt% nanoclay in flax-epoxy reinforced composites. This decrease can be improper distribution of nanoclay in the composites, their agglomeration, and inefective adhesion [28].

It is observed that an about 4% increase in the flexural strength was obtained with an addition of 0.5 wt% montmorillonite nanoclay. However, an obvious relationship between the flexural modulus and content of montmorillonite nanoclay were not observed. Trends of reduction in the plane shear tests with addition of montmorillonite nanoclay were also noticed in previous studies [7,9].

The montmorillonite nanoclay concentration of 0.5 wt% showed optimum improvement in the tensile and flexural properties because of improved fiber matrix adhesion. So the above observations, this study displayed that the addition of montmorillonite nanoclay improved the mechanical properties flax natural fibers of the composite. Thus, flax epoxy composite can also be used composites environment friendly products and the healthy living.

Ethics committee approval and conflict of interest statement

Ethics committee approval and conflict of interest statement This article does not require ethics committee approval. This article has no conflicts of interest with any individual or institution.

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Author Contribution Statement

Aybike Çelik did material preparation and tests.

Yeliz Pekbey did literature investigation, contributed to methodology, drafting of article and critical review of content and final approval and financial support.

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