



Research Article

Production and characterization of heat retardant fiber-reinforced geopolymer plates

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ABSTRACT

This paper presents an alternative environment-friendly thermal insulation material for the construction industry. This study aimed to produce this building material with superior heat resistance properties and comparable strength to the concrete produced with Ordinary Portland Cement. The primary purpose of the experimental studies was to produce a basic geopolymeric plate and to add cellulose and polypropylene fibers to the geopolymeric mortar. In the next stage, fiber-reinforced plates were prepared, thermal experiments were carried out, and discussions and conclusions were formed according to the results and findings. This study initially produced different types of fiber-based metakaolin plates with high heat resistance. Then, the flame test examined the heat resistance of the composite plates formed by the mixture of fibers consisting of cellulose, polypropylene, and cellulose + polypropylene fiber mixtures into geopolymeric mortars. It was found that the metakaolin plates containing approximately 6% by weight of Cellulose in the structure, besides their serious resistance to flame, their heat retardancy properties gave 72% better results than Kalekim (cementitious ceramic tile adhesive) plates and 55% better results than non-fiber metakaolin plates.

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1. INTRODUCTION

Concrete is the most used building material because of its mechanical properties, ease of handling, easy shaping, and availability of its raw materials. Producing and using that tremendous amount of concrete requires its typical constituent, ordinary Portland cement (OPC). OPC needs large amounts of natural resources for its production. 1.5–2.8 tons of raw materials are needed to produce 1

ton of OPC [1–3]. In addition, OPC production requires high temperatures of around 1500 °C, which causes large energy consumption. Thus, 12–15% of the total energy is used worldwide for all OPC production processes [4]. Energy consumption reaches about 40%, considering the construction industry. From the point of view of CO₂ emissions, 5–8% of all CO₂ emissions come from OPC production [5–7], and 1/3 of greenhouse gases emission are contributed by the construction industry [8]. Besides

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the CO₂ emission, more toxic gases such as Sulfur trioxide (SO₃) and nitrogen oxide (NO_x) are released into the atmosphere, which accelerates global warming and causes acid rain [9]. Recently, research has focused on developing alternative materials to OPC due to environmental and energy concerns [10–12]. One promising alternative to OPC is geopolymer material, which uses raw materials rich in aluminosilicate content and an alkaline solution to activate it [13–16]. Most of the geopolymer synthesis methods occur in this process. For example, aluminosilicate substances used in geopolymers may come from natural sources such as metakaolin (from kaolinite) or industrial wastes like fly ash and ground granulated blast furnace slag [2, 17–19]. These precursors are blended with an alkaline solution of silicate with the addition of a base, usually concentrated sodium or potassium hydroxide. The resultant material form looks similar to concrete paste but without Portland cement.

Geopolymers have many environmental and mechanical benefits over OPC [20, 21]. For example, fly ash-based geopolymers cause 80% less CO₂ in the atmosphere and consume 60% less energy during their production processes than OPC [22]. In addition, geopolymers can have better compressive strength and durability [23, 24], adjustable strength and workability [25–27], lower shrinkage and creep [28], better resistance against chlorides, acid attacks [29, 30], superior fire resistance [31], and improved thermal insulation properties [28]. Moreover, geopolymers can reach 90% of their top compressive strength in the first 72 hours [32]. This specialty makes it an ideal choice for early strength applications [33].

Using these alternative products has increased attention in construction due to the critical solution of decarbonization and energy-saving. Integration and formulation of thermal properties have been proven preferable by improving the energy efficiency and durability of the buildings [34]. One of the under research and promising applications of geopolymers are fireproof and fire retardant products [35–37]. In addition, some publications have shown their potential as thermal barriers [38, 39]. These studies proved that geopolymers could be used against fire and show high thermal stability.

One of the properties of the geopolymers, brittle behavior, puts a limitation on the application areas of geopolymer materials [40, 41]. Nowadays, researchers are trying to solve this problem to improve this mechanical property [42, 43]. Fiber-reinforcing is a well-known solution to overcome this problem. For this reason, researchers' studies with different types of fibers focused on reinforcing geopolymer [37], [44]. Polymer fibers are the first preferable for reinforcing the geopolymers [43]. Improving the mechanical properties, especially bending strength, is the main reason for adding fibers into geopolymer. The addition of some chemical fibers into geopolymers can reveal some benefits. Fiber reinforcing

can cause better properties, such as higher resistance to fire or a decrease in thermal conductivity, depending on their types [44, 45].

The main objective of this study is to produce an alternative environment-friendly thermal insulation material for the market. Moreover, this study aimed to produce this building material with superior heat resistance properties and comparable strength to the concrete produced with OPC. For this purpose, commercially purchased metakaolin was used as precursor material. Alkaline solutions were prepared with NaOH, KOH, SiO₂, and Na₂O. The geopolymerization was obtained by mixing metakaolin and the alkaline solution at room temperature. Cellulor (CB) and polypropylene (PP) fibers were used as reinforced materials to improve the heat resistance properties of the geopolymer products. The study resulted in a thermal comparison of fiber-reinforced geopolymer plates and plates prepared with OPC. It was found that the heat resistance properties of metakaolin plates containing fibers gave 55% better results than metakaolin plates without fibers.

2. MATERIALS AND METHODS

2.1. Materials

The primary material used in this work, metakaolin, was purchased from AVS Mineral. 100 grams of metakaolin was used for each sample. The activator consists of water, NaOH, and commercial sodium silicate containing 28.7% SiO₂, 8.9% Na₂O, and 62.4% H₂O by weight (Sodel Chemistry, Module: 2). Module for silicate is defined as SiO₂/Na₂O ratio. NaOH was obtained from Interlab. All solutions were prepared with pure water.

This study used PP fibers (Beton Fiber BF06 Polypropylene fibers) obtained from Beton Fiber company. It is preferred to increase concrete strength, abrasion resistance, and toughness and prevent crack formation, manufactured from cold drawn wire according to ASTM A820 standard. Polypropylene fiber homogeneously dispersed in the mortar aims to prevent loading cracks by increasing the toughness of the mortar and its ability to absorb energy. Polypropylene fiber-reinforced concretes have high bending, fracture, and compressive strength. They also have high temperatures, high chemical resistance, and a relatively low-density structure. In addition, they are entirely electronic, heat, and sound insulation products [46].

Another type of fiber used in this study is cellulor fiber. Cellulor fiber, a boron-combined cellulosic insulation material, is a dark green material consisting of a combination of newspaper papers used as waste through various processes using a boron mine. It contains 70–75% by mass of waste paper and 23% boron compounds to increase its flame retardant capability. Since waste paper is used, it has an environmentally friendly structure. It can be used as a heat insulation material on ceilings, attics, floors, and walls [47].

Table 1. The contents of the produced plates

Sample	Metakaolin (g)	Kalekim (g)	Sodium silicate (g)	NaOH (g)	Water (g)	CB-fiber (g)	PP-fiber (g)	Fiber (wt. %)
Fiber free Kalekim	0	200	–	–	50	–	–	0
Fiber free	100	–	82.30	18.30	50	–	–	0
PP1	100	–	82.30	18.30	50	–	0.75	0.74
PP2	100	–	82.30	18.30	50	–	1.13	1.11
PP3	100	–	82.30	18.30	50	–	1.50	1.47
PP4	100	–	82.30	18.30	50	–	1.88	1.84
PP5	100	–	82.30	18.30	50	–	2.25	2.20
CB1	100	–	82.30	18.30	50	2	–	1.96
CB2	100	–	82.30	18.30	50	3	–	2.91
CB3	100	–	82.30	18.30	50	4	–	3.84
CB4	100	–	82.30	18.30	50	5	–	4.76
CB5	100	–	82.27	18.24	50	6	–	5.66
CBPP1	100	–	82.27	18.24	50	2	0.75	2.67
CBPP2	100	–	82.27	18.24	50	3	1.125	3.96
CBPP3	100	–	82.27	18.24	50	4	1.5	5.21
CBPP4	100	–	82.27	18.24	50	5	1.875	6.43
CBPP5	100	–	82.27	18.24	50	6	2.25	7.62

2.2. Methods

2.2.1. Formulation of Geopolymer Prescriptions

The formation and preparation of the geopolymeric plates generally consist of simple steps (Fig. 1). Therefore, the primary purpose of the experimental studies was to produce a basic geopolymeric plate and to add CB and PP fibers into the geopolymeric plate. In the next stage, fiber-reinforced boards were prepared, thermal experiments were carried out, and discussions and conclusions were formed according to results and findings.

In the first stage, studies were carried out using different recipes on the ratios of compounds ($\text{Na}_2\text{O}/\text{NaOH}$, $\text{H}_2\text{O}/\text{Na}_2\text{O}$) and fibers in different ratios to find the primary geopolymeric plate with the most perfect thermal and structural properties. Initially, sixteen main geopolymeric plates and one Kalekim (Cementitious ceramic tile adhesive) plate sample were produced. Then, the mechanical properties and heat resistance tests of the geopolymeric plates were carried out at the end of seven days. The ratios of substances and fibers that made up the content of the seventeen samples investigated are shown in Table 1.

2.2.2. Preparation of Geopolymers

It is based on the combination of metakaolin used in the production phase of the geopolymeric plate with alumina silicate in an alkaline environment. As the first step, the NaOH compound in the solid phase was measured on a precision balance based on predetermined calculations and transferred to the beaker. In the next step, sodium silicate and distilled water were added to the calculated amount.

After, the solution was mixed for 10 minutes until the sodium hydroxide particles melted and became homogeneous in the mixer. After mixing, the solution was left to cool at room temperature and rested until it reached room temperature. If the resting phase is not performed, lumps occur in the mortar, making it more challenging to obtain a homogeneous mortar.

No heat was needed for the solution during the mixing phase because the geopolymerization reaction was exothermic. During the mixing stage, the mixing was covered to ensure minimum water loss so that the water from the solution was not evaporate. After the alkaline solution reached 25 °C, it was added and mixed gently and slowly on the metakaolin, which was prescribed and weighed. At this stage, fibers were added to the paste gradually and mixed in a mechanical mixer to have a homogenous structure. The mixing phase should last for a minimum of 10 minutes. Homogenizing the resulting mortar was an essential factor for the thermal and mechanical quantities of the geopolymer. Mixed and homogenized geopolymer mortars were poured into metal molds measuring 15 cm x 15 cm x 5 cm. After curing for 24 hours at room temperature, they were removed from the molds.

2.2.3. Testing

The flame gun used in the experiments is Integra Flameboy brand. It is a portable, hand-held, and battery-free Bunsen burner that provides piezo-electric ignition at the push of a button. In addition, the size and temperature of the flame can be easily adjusted with an air

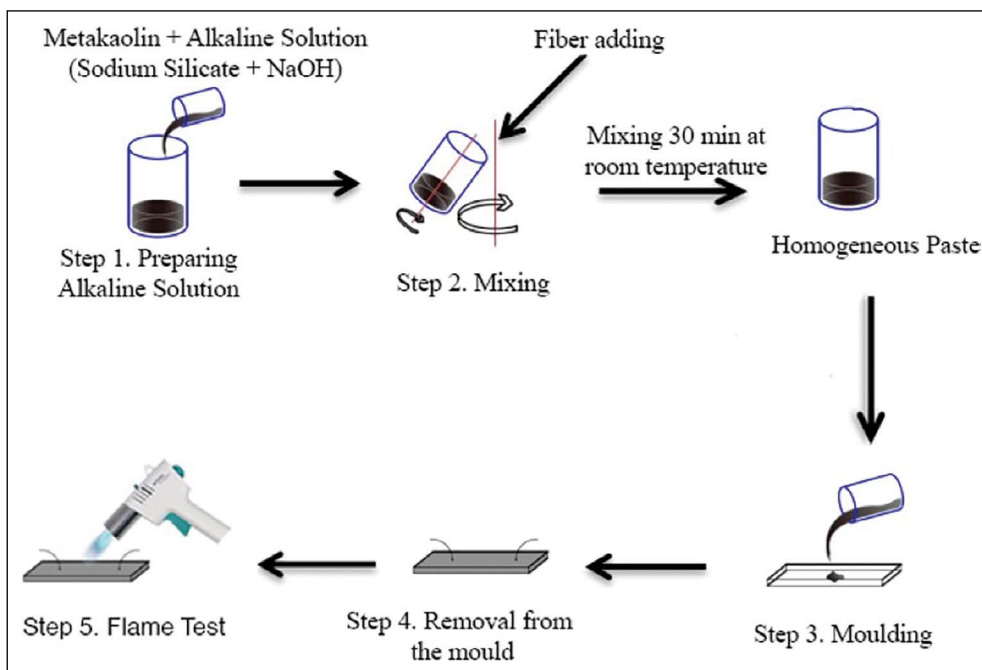


Figure 1. Geopolymer production process.



Figure 2. Flame test.

and gas regulator. The thermocouple thermometer used in this study is UDL 100 brand. It measures the front and back side temperature of the plates. The flame test was applied directly on the panel from a 25 cm distance clamped between the aluminum panel, as seen in Figure 2. The plates' front and back surfaces temperature are recorded at a speed of one-tenth of a second with J-type temperature meters connected to the computer. At this time, the flame-prone plate surface was also measured using an infrared

Table 2. Fiber-free metakaolin and Kalekim plate flame test results

Sample	$T_{Front-Max}$ (°C)	T_{Back} (°C)	t_{Back} (s)	t_{Back} (s) (@50 °C)	Δt_{Back} (s)
Metakaolin plate	909.8	27.9	39	60	21.0
Kalekim plate	880.2	32.2	16	28	12.0

thermometer. Thus, the temperature monitoring of the front surface is measured simultaneously with different measurement techniques.

3. RESULTS AND DISCUSSIONS

Fiber-containing geopolymer plates, fiber-free geopolymer, and Kalekim plates were fabricated. The study consists of 2 stages. The first step was to perform flame tests of fiber-free metakaolin and Kalekim plates and to determine the time it takes for the back side temperature to reach 50 °C. The second stage of the study was the flame tests of the fiber plates and the determination of flame retardancy.

A procedure has been developed to compare the study flame test results. Accordingly, the plate's maximum temperature reached by the front side was $T_{Front-Max}$. When the $T_{Front-Max}$ temperature was determined, the back temperature of the plate was recorded as back-plate temperature " T_{Back} " (24 ± 0.5 °C). In addition, the time elapsed from the start of the flame test to the occurrence of these temperatures ($T_{Front-Max}$ and T_{Back}) was recorded as the " t_{Back} " time. T_{Back} (°C) temperature and

Table 3. Flame test results of metakaolin plates containing different ratios of CB fiber

Sample	Fiber (Wt.%)	T _{Front} -Max (°C)	T _{Back} (°C)	t _{Back} (s)	t _{Back} (s) @50 °C	Δt _{Back} (s)
CB1	1.96	903	24.6	22	52	30.0
CB2	2.91	860.2	20.3	14	44	30.0
CB3	3.84	890.6	24.1	18	48	30.0
CB4	4.76	873.2	24.4	15	50	35.0
CB5	5.66	851.1	24	17	60	43.0

Table 4. Flame test results of metakaolin plates containing PP fiber in different ratios

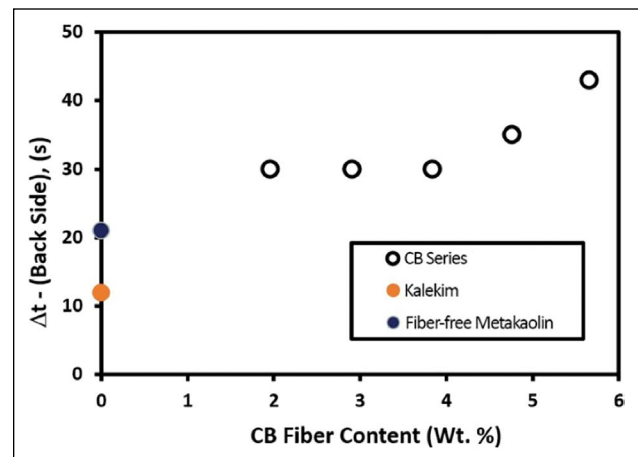
Sample	Fiber (Wt.%)	T _{Front} -Max (°C)	T _{Back} (°C)	t _{Back} (s)	t _{Back} (s) @50 °C	Δt _{Back} (s)
PP1	0.74	852.7	22.8	20	50	30.0
PP2	1.11	986.7	22.5	15	47	32.0
PP3	1.47	772.2	24.4	20	51	31.0
PP4	1.84	872.2	24.1	19	52	33.0
PP5	2.20	828.5	24.6	18	46	28.0

the t_{Back} (s) time were defined as the initial temperature and time of the back plate. The first time the back plate reaches 50 °C, it was reported as " t_{Back} (s) @ 50 °C" in the test records. This value measures how long after the start of the experiment the backplate reaches 50 °C. The difference between the time of the front and back of the plate to reach the same temperature was expressed as " Δt_{Back} (s)." This period shows how long the plate transmits the heat from the front side to the backside.

Firstly, the flame test of the fiber-free Kalekim plate was performed. The heat delay time of the Kalekim plate was found to be 12 seconds. In the same experiment, when it is made with a metakaolin plate that does not contain any fiber, it is seen that the back side of the plate reaches 50 °C in the 21st second. Therefore, the metakaolin plate provided heat retardancy 9 seconds longer than the Kalekim plate (Table 2).

The back side of the fiber-free metakaolin plates was observed to reach 50 °C 9 seconds later than the Kalekim plate. This heat transfer delay was due to the metakaolin structure [48]. The back plate temperature of metakaolin reached 27.9 °C after 39 s of the start of the experiment at the 909.8 °C of the front plate. Regarding the Kalekim plate, the T_{Back} reached 32.2 °C at 16 s after the experiment at the 880 °C of the front plate. Therefore, when the duration of reaching 50 °C is compared to both plates, the Metakaolin plate was reached after 32 s than the Kalekim plates, which was essential delaying of heat transfer.

Regarding fiber-reinforced plates, CB, PP, and CB+PP mixed fibers were used to obtain fiber-containing metakaolin plates. Flame test analysis results of CB series metakaolin plates containing cellubor fiber in different ratios are shown in Table 3.

**Figure 3.** Metakaolin plates flame retardant time variation due to increased CB fiber content.

The t_{back} @50 °C of CB fiber-reinforced metakaolin increased depending on increasing CB fiber content after 3% wt in the metakaolin mortar. The variation of flame retardancy times according to the fiber ratio of metakaolin plates containing different ratios of CB fiber is given in Figure 3. When the CB-fiber content was up to 3% wt, no relation was observed between CB and the t_{back} @50 °C, and heat transfer delaying remained constant. However, a linear trend was observed after 3% wt of fiber content due to the CB fiber heat resistance property. In addition, even with 1.96% wt of CB, the heat transfer delay was observed comparing the fiber-free metakaolin and Kalekim plates (Fig. 3).

Regarding the PP fiber-reinforced metakaolin plates, no relation was observed between the heat transfer delay and fiber content, as shown in Table 4.

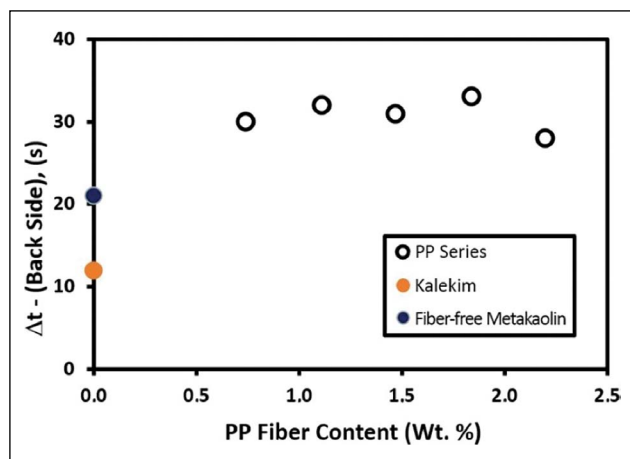


Figure 4. Metakaolin plate flame retardant time change due to increased PP fiber content.

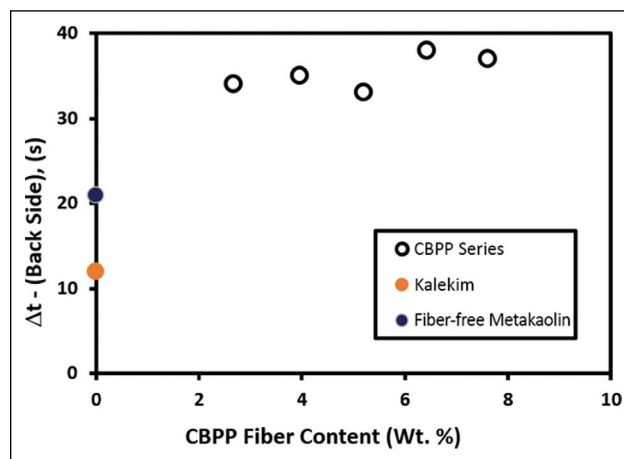


Figure 5. Change of flame-retardant time of metakaolin plate due to increasing CB+PP fiber mixture.

Table 5. Flame test results of metakaolin plates containing different ratios of CB+PP fiber mixture

Sample	Fiber (Wt.%)	T _{Front} -Max (°C)	T _{Back} (°C)	t _{Back} (s)	t _{Back} (s) @50 °C	Δt _{Back} (s)
CBPP1	2.67	878.2	23.1	15	49	34.0
CBPP2	3.96	842.7	23.9	12	47	35.0
CBPP3	5.21	815.8	23.9	10	43	33.0
CBPP4	6.43	907.1	23.9	14	52	38.0
CBPP5	7.62	808.2	24.0	25	62	37.0

The variation in flame retardancy times according to PP fiber content is given in Figure 4. This figure indicated that constant flame retardancy times were observed despite increasing the fiber ratio. It is due to the lower PP fiber melting properties. The PP fiber melts after 120 °C and fills the porous structure of the metakaolin. Thus, the heat-delaying properties are reduced due to the lowering space of the metakaolin. However, PP fiber-reinforced metakaolin plate has better heat retardancy than the fiber-free metakaolin and Kalekim plates.

The CB and PP mixed fibers were also investigated for the thermal performance of metakaolin plates. The flame test results of CBPP series metakaolin plates containing CB+PP fiber mixture in different ratios are given in Table 5.

The variation of flame retardancy times according to the fiber ratio of metakaolin plates containing CBPP fiber in different ratios is seen in Figure 5. This figure indicated that constant flame retardancy times were observed despite increasing the fiber ratio similarly to PP-containing metakaolin plates (Fig. 4). This similarity is due to the melting properties of PP fibers. However, the introduction of CB fiber in PP fiber-reinforced metakaolin plates showed more heat insulation (Fig. 5). In addition, CBPP fibers brought approximately three times stronger fire retardancy to the Kalekim plate and 1.5 times to the metakaolin fiber-free plate (Fig. 5).

4. CONCLUSIONS

This paper produced CB, PP, and CBPP fiber-reinforced geopolymer plates. The flame tests of the plates were carried out. First, according to the flame test results, all the mortars reinforced with fibers showed higher heat retardancy than the fiber-free metakaolin and Kalekim mortars. Depending on the increased CB ratio, it was observed that the temperatures of the back parts of the plates reached 50 °C in between 20 and 43 seconds. Furthermore, it was found that the metakaolin plate containing 5.66% by weight of CB showed approximately 55% more heat retardancy than the fiber-free metakaolin plate and 72% more than the fiber-free Kalekim plate.

On the other hand, the performance of PP-containing metakaolin plates did not change seriously depending on the variation of PP content, and the time to reach 50 °C for the back plate was around 31±2 seconds. This result indicated that a small amount of PP in the building is enough to supply heat resistance. However, heat resistance performance remained stable despite the increasing PP content. It is due to the filling of the gaps with melting PP. Although the flame test results of CBPP series metakaolin plates containing a certain amount of PP and CB blended fibers are relatively good compared to plates containing only PP fibers but not good as CB fibers. The melting PP reduced the gaps between

the CB fibers, so the heat retardancy performance decreased compared with the CB fiber-reinforced metakaolin plates.

As a result of the experiments, it was found that the metakaolin boards containing approximately 6% by weight of CB in the structure showed 72% better heat resistance results than Kalekim boards and 55% better results than non-fiber metakaolin boards. This study is a pioneering study in producing fiber-containing plates, and it is expected that the produced plates will be used as building materials in the construction industry in future studies, as they have both heat resistance and fire retardant properties.

ETHICS

There are no ethical issues with the publication of this manuscript.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

FINANCIAL DISCLOSURE

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PEER-REVIEW

Externally peer-reviewed.

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