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Ablation Properties of Glass Fiber Reinforced Geopolymer Composite Incorporation of Nanoclay and MWCNTs using Response surface Methodology (RSM) Approach

Izzat Najmi Mohd Yaacob[1,2], Norkhairunnisa Mazlan[1,2,3,*], Nur Amalin Huda Rozuki[1,2], Siti Madiha Muhammad Amir[4], Natesan Kapilan[5]

1 Department of Aerospace Engineering, Faculty of Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
2 Aerospace Malaysia Research Center (AMRC), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
3 Institute of Nanoscience and Nanotechnology (ION2), Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia
4 Malaysia Nuclear Agency, 43000, Kajang, Selangor, Malaysia
5 Department of Mechanical Engineering, Nitte Meenakshi Institute of Technology, Bengaluru, India

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ABSTRACT

Glass fiber reinforced geopolymer composites (GFRGCs) materials are widely used in several industry especially in the aerospace industry due to their high strength to weight ratio, tremendous ablative properties, and high thermal stability. In the context of rocket insulation, the ablation properties of GFRGCs have attracted growing research interest. The extreme thermal stresses experienced during rocket launches and atmospheric re-entry demand advanced materials with high ablation resistance. Furthermore, recent studies were reported that incorporation of nanofiller such as nanoclay and multiwall carbon nanotubes (MWCNTs) can improve the ablation properties of GFRFCs. Nevertheless, whereas MWCNTs can increase mechanical strength and thermal conductivity and nanoclay is recognised for its capacity to increase thermal stability, their combined impact on the ablation performance of GFRGCs has not been well investigated. This study aims to elucidate the effect of incorporation of both nanoclay and MWCNT to the ablation properties of GFRGC. A systematic approach, such as Response Surface Methodology (RSM), is utilized to optimize the composite formulation in identifying the optimal combination of these additives and their interaction with the glass fibers and geopolymer matrix. GFRGC samples were fabricated using hand lay-up technique and the ablation properties were evaluated through fire test. Through RSM, both nanoclay and MWCNTs has signifanct effect on the back temperature of the sample. Besides, the experimental result shows that GFRGC has the lowest temperature at the back of the sample with 3% nanoclay and 7% MWCNTs with value of 306.741°C. The thermal conductivity value of this sample also resulted in the lowest value of 0.069 W/mK compared to the other sample. This finding can contribute to the development of promising insulation materials for high thermal application.

* *Corresponding author.*
E-mail address: norkhairunnisa@upm.edu.my

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1. Introduction

Fiber-reinforced geopolymer composite (FRGC) is emerging as an alternative material to cement in the construction sector. FRGC is regarded as an environmentally sustainable material because of its contribution to the global decrease in CO_2 emissions. Concurrently, the composite exhibits substantial mechanical strength, including flexural modulus, loss modulus, post-impact strength, and durability for a specified duration at both ambient and increased temperatures. A beneficial characteristic of the geopolymer matrix utilised in this composite is its capacity to offer a limited duration of durability at high temperatures without emitting harmful gases into the environment [1-3]. Besides, the geopolymer matrix is deemed capable of enduring high temperatures during the production of composite products, in contrast to plastic matrices [4]. The operational temperature exceeding 200 ◦C facilitates the thermal exposure of geopolymer composites, resulting in their significant heat resistance feature [5,6]. FRGC is suggested as an alternative building material in the cement business and as a repair material in the construction sector, emerging as a hybrid solution for the restoration of damaged cementitious materials. The geopolymer composite may serve as a cohesive material with thermoplastic and polyamide components in diverse applications [7]. The geopolymer matrix fulfils a primary criterion for structural safety, specifically its fire-resistant qualities, which denote its capacity to withstand elevated temperatures caused by fires.

Fibres in various forms, such as threads, filaments, whiskers, and nanoparticles, have been utilised as reinforcement in geopolymer composites to enhance their flexural strength and energy absorption capabilities. When selecting fibres for reinforcement in cementitious and geopolymer composites, three primary criteria must be considered: (i) alignment of material properties with the intended application [8], (ii) adequate fibre-matrix interaction to facilitate stress transfer [8,9], and (iii) an optimal aspect ratio to ensure effective post-cracking performance [10]. These fibres improve the brittle nature of the geopolymer matrix and change it into a ductile form, thereby enhancing its mechanical strength and residual impact resistance [11]. Glass fibre-reinforced geopolymer composites (GFRGCs) represent an innovative category of materials that combine the advantageous properties of geopolymers and glass fibres. The inclusion of glass fibres significantly improves the mechanical characteristics of the composites, including tensile strength and hardness [10-13]. Furthermore, the presence of glass fibres enhances the geopolymer matrix`s flexural properties and crack resistance, making GFRGCs suitable for construction, infrastructure, and aerospace applications. In aerospace applications, fire-resistant panels for aircraft cabin interiors are essential components that facilitate passenger evacuation during emergencies. The E-glass FRGC composite provides survivors with valuable extra time for escape [14]. The flashover phenomena defines the total time before complete material incineration, indicating the window for avoiding a fire hazard. Notably, geopolymer matrices will not combust or produce smoke until reaching temperatures above 1000 °C [15]. In this context, E-glass fibres are the most appropriate reinforcements within the geopolymer matrix to preserve material strength [16].

Recently, GFRGCs have become essential materials for insulating layers in solid rocket motors, significantly enhancing their safety and performance. The primary function of the geopolymer insulation is to serve as a heat barrier between the internal rocket casing and the propellant [17]. This barrier is crucial for preventing the casing from reaching temperatures that could compromise its structural integrity, thereby ensuring the rocket's reliability during operation. Besides, numerous researchers in [17-20] have explored the incorporation of nanofillers, such as nanoclay and multiwall carbon nanotubes (MWCNTs), to improve the insulation properties of GFRGCs by modifying their ablation characteristics. The addition of either nanoclay and MWCNTs individually has been shown to improve thermal insulation and provide mechanical reinforcement, leading to superior ablation resistance in high-temperature environments [18]. While nanoclay is recognised for its potential to enhance thermal stability, and MWCNTs are known to improve thermal conductivity and mechanical strength, the synergistic effects of these nanofillers on the ablation performance of GFRGCs still need to be explored. A significant research gap exists in understanding the synergetic effect of incorporating nanoclay and multi-walled carbon nanotubes (MWCNTs) into glass fiber-reinforced geopolymer composites, particularly regarding their combined composition percentage influence on thermal insulation properties. Furthermore, data are scarce regarding the interactions between nanoclay, MWCNTs, and the glass fibre-reinforced geopolymer matrix under extreme thermal fluxes, complicating accurate predictions of material behaviour in high-temperature applications such as aerospace, fire protection, and thermal barriers [19,20]. Therefore, targeted research is necessary to systematically assess the role of these nanofillers in enhancing the ablation resistance of GFRGCs.

Recent experimental studies have investigated the thermal ablation properties of geopolymer composites reinforced with nanoclay and MWCNTs respectively. A study by Shauqi *et al.,* [20] examined the ablative behavior of glass fiber-reinforced epoxy and geopolymer composites with varying nanoclay concentrations. Notably, the geopolymer nanocomposite exhibited superior ablative behavior compared to the epoxy nanocomposite, with a back surface temperature of 51.34 °C compared to 176.86 °C for 7 wt% nanoclay in the epoxy nanocomposite. Besides, another study by Senthil Assaedi *et al.,* [19] investigated the effect of nanoclay on the mechanical and thermal properties of glass fiber-reinforced polymer nanocomposites. The findings showed that the addition of 2.0 wt% nanoclay enhanced the tensile strength and thermal stability of the composites. A study by Khater *et al.,* [23] examined the effect of MWCNTs on the thermal stability and mechanical properties of geopolymer composites. The researchers found that adding MWCNTs improved the thermal stability of the composites, as evidenced by thermogravimetric analysis (TGA) and differential thermal analysis (DTA). There is no research data reported on the synergetic ablative performance of the GFRGCs incorporation of nanoclay and MWCNTs as nanofiller. This paper presents an experimental study on the incorporation of nanoclay and MWCNTs as nanofillers in GFRGCs to enhance their ablation properties, employing Response Surface Methodology (RSM). RSM is a statistical tool particularly suited for optimizing and modelling complex interactions between variables, making it ideal for this investigation. Utilizing RSM facilitates an investigation into how variations in the concentrations of nanoclay and MWCNTs influence ablation performance. Moreover, this method enables the identification of optimal nanofillers and processing conditions to maximize the composite's ablation resistance. The findings from this research contribute to the development of optimized nanofillers formulations to enhance the ablation properties of for aerospace applications

2. Methodology
2.1 Materials and Apparatus

Kaolin, an aluminosilicate material sourced from Kaolin (Malaysia) Sdn. Bhd., is used in the fabrication of geopolymer matrices. Potassium hydroxide (KOH) and sodium hydroxide (NaOH) were procured from R&M Chemicals, serve as the alkaline solution in the geopolymer matrix. Acetone, with a purity of 99%, is utilised as the dispersing agent for the nanoclay. The hydrophilic bentonite nanoclay was acquired from Sigma Aldrich (USA). Two components of epoxy type 103, provided by Smooth-On, were employed as the polymer matrix. Additionally, woven roving glass fibre was used as the reinforcing material in this research.

2.2 Glass Fiber Reinforced Geopolymer Composites (GFRGCs) Preparation

KOH and NaOH were diluted with distilled water and then combined to form a highly alkaline solution. The alkaline solution was prepared one day in advance to ensure the mixture's homogeneity. The aluminosilicate precursor, kaolin, was subsequently added to the solution and thoroughly mixed. Following this, a specified quantity of nanoclay and multiwall carbon nanotubes (MWCNTs) was incorporated into the mixture and stirred to enhance its properties. Both nanoclay and MWCNTs served as nanofiller. The geopolymer solution mixture was then prepared using glass fibre for the hand layup procedure. Figure1 shows the fabrication process of GFRGCs using hand lay-up method same as experimental works conducted by Rao *et al.,* [21]. The size of the sample prepared is 100 x 100 mm glass fibres for all the samples.

Fig. 1. Glass fiber reinforced geopolymer composites (GFRGCs) fabrication process

First, the geopolymer matrix is prepared by mixing an aluminosilicate precursor with an activator solution made of NaOH) and KOH. Nanoclay and MWCNTs are pre-dispersed in deionized water using ultrasonication to achieve homogeneity before being added to the activator solution. The glass fiber sheets are then layered with the geopolymer slurry, ensuring even distribution using techniques such as rolling to remove trapped air. The composite is initially cured at room temperature for 24 hours, followed by heat treatment at 60°C for 24 hours to complete geopolymerization. Post-curing, the composite is trimmed, and polished for fire testing.

The total number of GFRGSc samples prepared was determined based on the Response Surface Methodology (RSM), which serves as the experimental design tool for this study. In this experiment, the nanoclay percentage varied from 1% to 9% (1%, 3%, 5%, 7%, 9%), while the MWCNTs percentage ranged from 0% to 8% (0%, 2%, 4%, 6%, 8%). Using RSM, 26 experimental runs were created for each unique combination of nanocomposite percentages. The proportions of each nanofillers are based on the experimental works conducted by Agnihotri *et al.,* [22].

2.3 Response Surface Methodology and Sample Size

Response surface methodology (RSM) combines regression analysis with statistical techniques used to visualize data through plots, enabling the prediction of correlations between dependent and independent variables. At the same time, it can forecast the optimal conditions for a given process. This research focuses on elucidating the synergetic effects of the two nanofillers on the ablation properties of GFRGC. Through RSM, the sample size was generated and the sample size illustrated in Table 1.

Table 1
Run order of all the samples generated by RSM

RunOrder	Percentage of Nanoclay, %	Percentage of MWCNTs, %
1	5	1
2	5	5
3	7	7
4	7	3
5	1	5
6	5	5
7	5	9
8	7	3
9	5	5
10	3	7
11	5	5
12	5	5
13	5	9
14	5	5
15	3	3
16	1	5
17	9	5
18	5	5
19	7	7
20	5	5
21	5	5
22	5	1
23	3	7
24	5	5
25	9	5
26	3	3

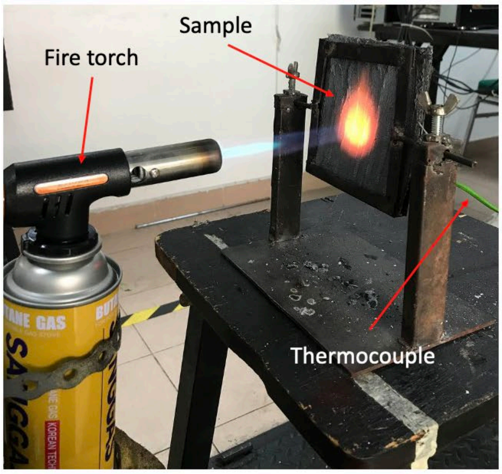
All 26 samples were tested, and the response, specifically the back temperature, was measured and calculated to obtain the thermal conductivity of the samples, which was subsequently discussed. Besides, RSM was employed to generate contour and surface plots for data presentation, allowing for a comparison and investigation of the relationship between the percentages of both nanofillers to thermal conductivity. The glass fibre consisted of five layers with a total mass of 13.2 g. Given a fibre-to-resin ratio of 60:40, the mass distribution was determined for various percentages of additional components.

$$\frac{40}{60} = \frac{R}{13.2} \tag{1}$$

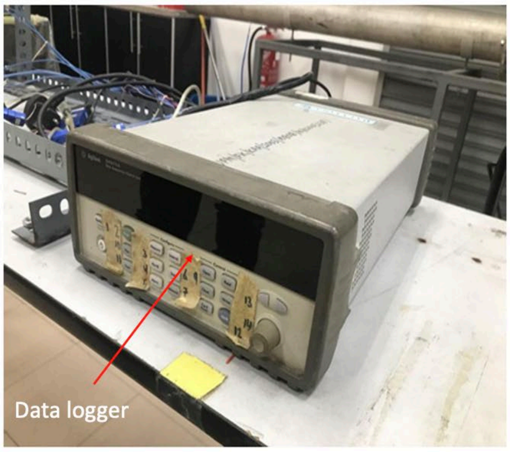
Eq. (1) yields a value of *R* to 8.8 g, which then utilized to calculate the masses corresponding to various percentages of nanoclay and MWCNTs. This process ensured that the mass distribution for each sample was accurate. In addition, this sample mass was instrumental in calculating the thermal conductivity.

2.4 Experimental Testing
2.4.1 Ablation test

The ablation test was performed on samples containing varying percentages of nanoclay and MWCNT. Each sample was affixed to a steel plate and a thermocouple was positioned on the back of the steel plate to monitor temperature. The opposite side of the insulation material was subjected to direct flame from a high-temperature torch set at 1000°C. During the 60-second ablation test, the temperature at the back of the steel plate was recorded using a data logger. Figure 2 illustrates the data logger, which captures and stores data from various sensors and equipment over time. When employing a thermocouple, the data logger effectively recorded temperature measurements along with other relevant data. Temperature readings were taken using a fibreglass-insulated type-K thermocouple. The test specimens were placed 10 cm away from the blowtorch to begin the fire retardants test. This specific distance ensured that the blue flame made direct contact with the specimens' surfaces, thereby simulating real-life fire exposure conditions and enhancing the applicability of the test.



(a)



(b)

Fig. 2. (a) Fire test setup using blow torch and (b) data logger system

Thermal conductivity (K) was calculated using the measured temperature data and the following formula derived from Fourier's law of steady-state heat conduction [24]:

$$k = \frac{q \times L}{A \times \Delta T} \quad (2)$$

where *k* is the thermal conductivity, *q* is the heat flux, *L* is the thickness of the samples, *A* is. The cross-sectional area through which the heat is transferred, and *ΔT* is the temperature difference across the sample. Then, the heat flux, q, is calculated as [25,26]:

$$q = \frac{Q}{A \times t} \quad (3)$$

where *q* is the heat flux, *Q* is the total heat transfer, *A* is the cross-sectional area through which the heat is transferred, and *t* is time over which heat transfer occurs. For this experiment, the heat transfer parameter, *Q*, is based on this formula [23]:

$$Q = mc\Delta T \quad (4)$$

where *m* is mass of the sample, *c* is the specific heat capacity of the samples, and *ΔT* is the temperature difference across the sample.

The distance between the samples and the torch was fixed at 10 cm. The thickness of each sample was 0.13 cm, and the duration of exposure was set to 60 seconds. Temperature gradients across the sample were crucial for evaluating thermal conductivity, as they illustrated how temperature fluctuated with distance throughout the material under applied heat flux. This technique detailed on how ablation testing was employed to gather temperature data and how thermal conductivity was determined using basic heat transfer principles. This method ensured the reliability and validity of the thermal conductivity results presented in this study. Additionally, contour and surface plots were included to observe the effects of both nanofillers on the ablation properties of the samples.

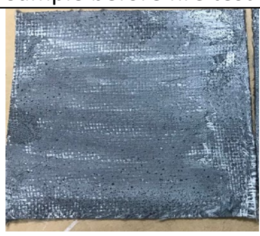
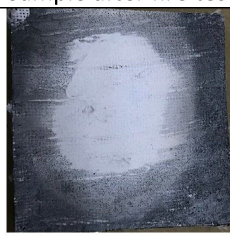
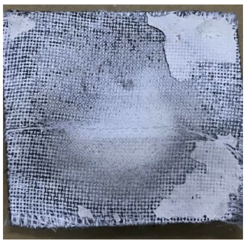
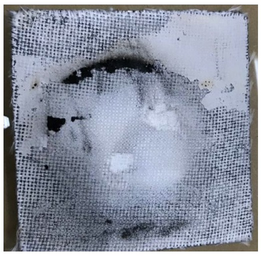
3. Results and discussions

3.1 Ablation test

Based on the fire data reported in Table 2, along with direct observations obtained during the 1000°C fire test, the geopolymer composite samples exhibited a substantial difference. The table presented geopolymer samples containing nanoclay and MWCNTs before and after exposure to fire from a direct blowtorch. In Table 2, the results illustrated only those samples with varying percentages of nanoclay and MWCNTs, excluding some of the 29 samples due to repetitions. Visually, the samples before the fire displayed a smooth, intact surface, while those after exposure to fire showed significant discrepancies. Various colour changes and circular burning patterns indicated areas of intense heat exposure. Following the fire test, the samples' surface appearances changed significantly, particularly on the front and back surfaces.

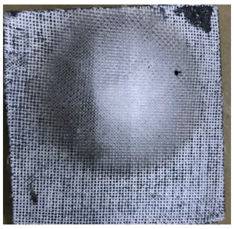
Significant discoloration and damage were visible on the front surface, which had been directly exposed to heat. A large white patch in the centre, surrounded by darker areas, indicated prolonged exposure to high temperatures. This centre discoloration most likely implied material degradation, such as charring and a loss of structural integrity. In contrast, the back side exhibited less severe discoloration, suggesting that the material provided some thermal insulation. These findings contradicted the data presented in the experiments conducted by Sharma *et al.,* [27] in which the authors elucidated the ablation properties of glass fibre-reinforced epoxy coated with graphene and MWCNTs.

Table 2
Result of the samples before and after fire test

Sample before fire test	Sample after fire test	Sample before fire test	Sample after fire test
 GFRGC 1% Nanoclay : 5% MWCNTs	 Front	 GFRGC 3% Nanoclay : 3% MWCNTs	 Front
	 Back		 Back

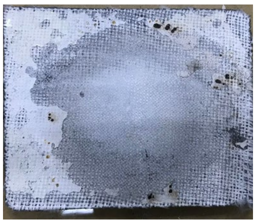

GFRGC 3% Nanoclay : 7% MWCNTs


Front

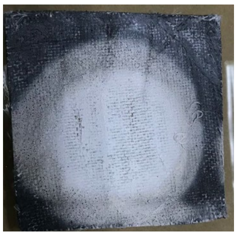

Back


GFRGC 5% Nanoclay : 1% MWCNTs


Front


Back


GFRGC 5% Nanoclay : 5% MWCNTs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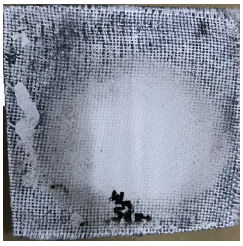
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GFRGC 5% Nanoclay : 9% MWCNTs


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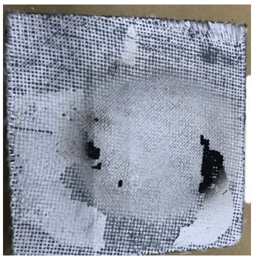
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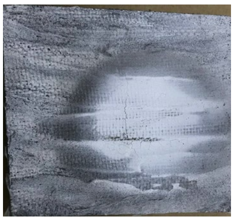
GFRGC 7% Nanoclay : 3% MWCNTs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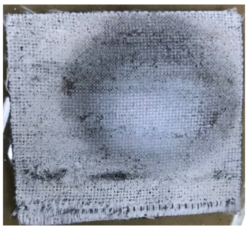
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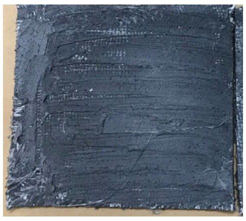
GFRGC 7% Nanoclay : 7% MWCNTs


Front


Back

 GFRGC 9% Nanoclay : 5% MWCNTs	 Front  Back

Referring to Table 2, the damage observed in each sample with varying ratios of nanoclay and MWCNTs were distinct. This variation was attributed to the roles and synergetic effects of nanoclay and MWCNTs after the composite surface was exposed to fire. The combination of nanoclay and MWCNTs within the geopolymer matrix significantly enhanced its fire resistance due to several synergistic interactions. Presence of nanoclay improved thermal stability by serving as an insulating barrier that slowed heat transmission, while MWCNTs facilitated heat dissipation and provided structural reinforcement. Furthermore, the addition of MWCNTs increased the mechanical strength of the geopolymer, enhancing its resistance to structural degradation under fire conditions [28]. This combination of effects ensured that the material remained intact even after prolonged exposure to fire. Both nanoclay and MWCNTs being inorganic and non-combustible meant that the materials did not add to the fire load. These properties made the geopolymer nanocomposite suitable for applications requiring substantial fire protection. Table 3 illustrated interesting patterns regarding how different percentages of nanoclay and MWCNTs influenced the fire resistance of the geopolymer nanocomposite, as evidenced by the average back temperature.

Table 3
Average back temperature of all the samples

Percentage of Nanoclay, %	Percentage of MWCNTs, %	Average Back Temperature, ℃
3	7	306.741
7	3	335.258
1	5	333.912
5	1	337.542
5	9	344.166
9	5	334.128
3	3	342.916
5	5	410.558
7	7	313.057

From Table 3, the sample containing 3% nanoclay and 7% MWCNTs exhibited the lowest back temperature of 306.741℃, indicating a synergistic action that significantly improved fire resistance. In contrast, samples with 5% nanoclay and 5% MWCNTs recorded higher back temperatures of 410.558℃, reflecting a less effective fire resistance. This difference was attributed to particle aggregation in which the particles were not evenly distributed throughout the matrix at these concentrations, leading to poor heat dissipation and higher temperatures. The lower back temperature of the combination with 3% nancolay and 7% MWCNTs likely resulted from a more advantageous interaction between the particles, which facilitated more excellent heat dissipation and mitigated the issues of particle agglomeration observed in some other combinations. Additionally, the synergetic effect of nanoclay as thermal insulation, which reduced heat penetration, alongside the role of MWCNTs in enhancing structural integrity and thermal stability, contributed to this sample achieving the lowest back temperature.

The combined examination of the contour and surface plots using response surface methodology (RSM) provided comprehensive insights into the interaction between the percentages of nanoclay and MWCNTs, and their impact on the back temperature of the GFRGC samples. The contour plot in Figure 3 highlighted regions with different temperature ranges.

3.2 Thermal Conductivity Analysis

Table 4 presented the thermal conductivity of several samples with varying ratios of nanoclay and MWCNTs, offering valuable insights into how these additions influenced the thermal characteristics of the geopolymer nanocomposite. The temperature differences were calculated using the difference between the temperature in front of the sample produced by the blow torch at 1000°C and the back temperature of the samples based on the Table 4.

Table 4
Temperature different and thermal conductivity on each sample

Percentage of Nanoclay, %	Percentage of MWCNTs, %	Temperature difference, ΔT, K	Thermal Conductivity, W/mK
3	7	966.41	0.0660
7	3	937.892	0.0661
1	5	939.238	0.0662
5	1	935.609	0.0661
5	9	928.984	0.0664
9	5	939.022	0.0661
3	3	930.234	0.0663
5	5	862.592	0.0665
7	7	960.094	0.0661

The thermal conductivity values across all samples are rather similar, ranging from 0.0661 W/mK to 0.0665 W/mK. This suggests that variations in the quantities of nanoclay and MWCNTs lead to only minimal changes in the material's heat transfer capabilities, consistent with the data reported in references [29,30]. As shown on Table 4, GFRGCs with 3% nanoclay and 7% MWCNTs resulted the highest temperature different. This significant temperature difference indicates that the heat does not transfer easily through the material’s thickness, resulting the front surface (fire-exposed side) to reach much higher temperatures than the back surface. This could be attributed to the low conductivity properties of this sample, which recorded the lowest thermal conductivity value of 0.0660 W/mK. Conversely, the GFRGCs sample containing 5% nanoclay and 5% MWCNTs demonstrated the lowest temperature difference of 862.592 K, correlating with the highest thermal conductivity value of 0.0665 W/mK. These findings highlight the synergistic effect of both nanoclay and MWCNTs in modifying the thermal conductivity of the composite, with nanoclay acting as a thermal insulator and MWCNTs providing thermal stability to prevent deformation at high temperatures. Besides, Figure 3 illustrates the contour and surface plot of nanoclay and MWCNTs percentage on the temperature difference in the GFRGCs sample.

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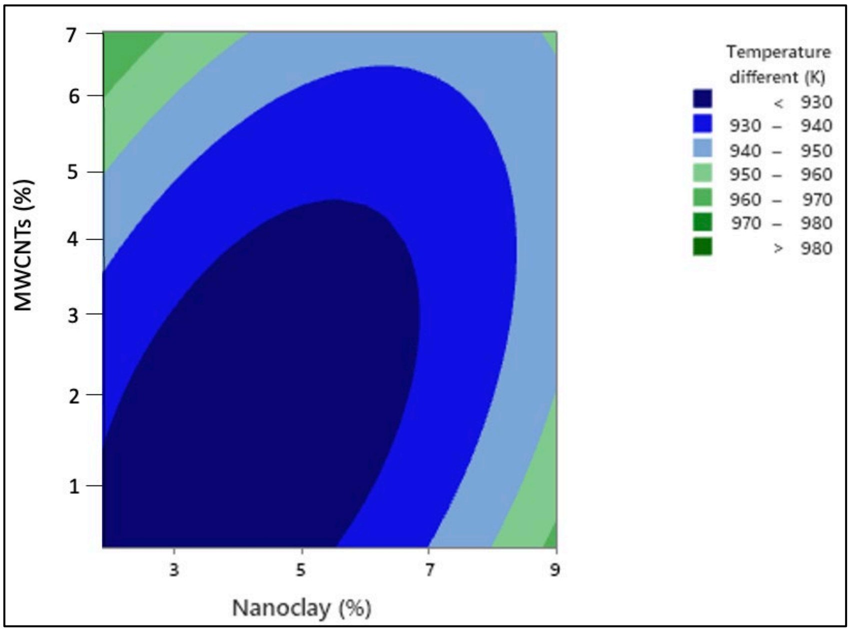

Fig. 3. Contour plot of nanoclay and MWCNTs percentage effect on GFRGCs sample's temperature different

Based contour plot in Figure 3, the highest temperature difference is observed at lower percentages of both nanoclay and MWCNTs, represented by the dark blue colour, which indicates a temperature difference of approximately 930 K. Besides, the lowest temperature difference occurs in the region characterized by lower percentages of nanoclay combined with higher percentages of MWCNTs, which contradicts the results presented in Table 4. The relationship between percentage of nanoclay and MWCNTs with the resulting temperature difference is further illustrated in the surface plot in Figure 4.

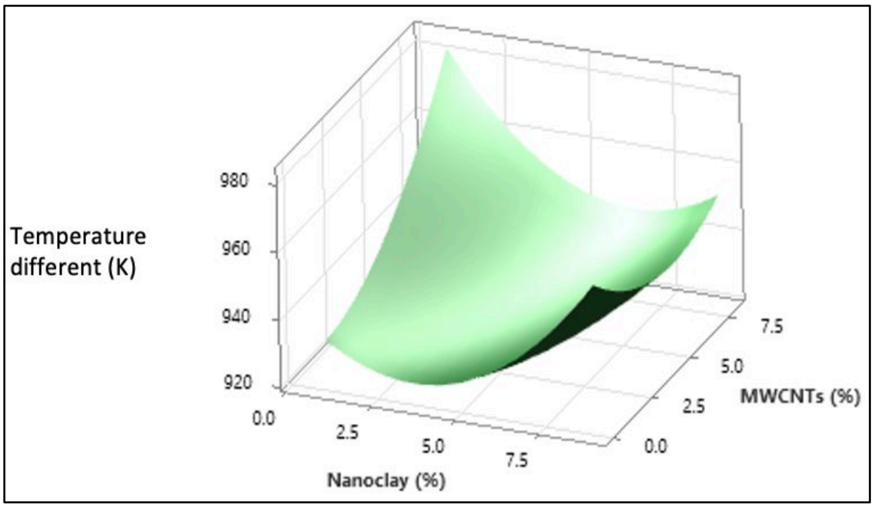

Fig. 4. Surface plot of nanoclay and MWCNTs percentage effect on GFRGCs sample's temperature different

The surface plot in Figure 4 illustrates that the relationship between the percentages of nanoclay and MWCNTs contradicts the findings presented in the contour plot in Figure 3, where the highest temperature difference is observed at lower nanoclay and MWCNTs. Besides, the downward dome-shaped surface suggests that there may be an optimal combination of nancolay and MWCNTs percentages for achieving the lowest temperature difference. Significant research has been conducted to explore the thermal conductivity of glass fiber-reinforced geopolymer composites incorporating nanoclay and multi-walled carbon nanotubes (MWCNTs) for thermal insulation applications. Studies reveal that the addition of nanoclay enhances the composite's thermal insulation by forming a dense, layered microstructure that creates a barrier effect, effectively reducing heat transfer. Meanwhile, MWCNTs contribute by improving thermal conductivity within the matrix, which aids in dissipating localized heat and enhancing thermal stability. The synergistic effect of combining nanoclay and MWCNTs has shown promising results, as nanoclay minimizes heat penetration while MWCNTs provide structural reinforcement and manage heat distribution. This dual functionality not only enhances thermal insulation but also ensures the material's durability under thermal stress, making it suitable for applications in high-temperature environments such as aerospace and construction. Further research is ongoing to optimize the composition and dispersion techniques for these hybrid reinforcements to maximize performance.

4. Conclusions

A comprehensive investigation into the effects of nanoclay and MWCNTs on the fire resistance of GFRGCs reveals several significant findings. The results indicate that a specific combination of 3% nanoclay and 7% MWCNTs within a geopolymer matrix significantly enhances thermal insulation properties. This optimised mixture demonstrates a synergistic effect, leading to the lowest back temperature recorded during fire tests. Furthermore, the thermal conductivity of the GFRGC sample with 3% nanoclay and 7% MWCNTs reached a value of 0.0660 W/mK, while those 5% nanoclay and 5% MWCNTs exhibited an even higher value of 0.0665 W/mK. These results suggest that a lower percentage of nanoclay combined with a higher percentage of MWCNTs improves the composite’s thermal insulation properties. In contrast, equal percentages of both nanofillers yield favourable thermal conductivity. For aerospace applications, this implies that the insulation layer can effectively maintain lower temperatures, even under the extreme heat generated by rocket propulsion. Besides, these findings provide valuable insights into the ongoing research and development of thermal insulation layers for GFRGC using nanofillers.

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