



Original Article

The Impact of Mixed NaOH and KOH Mole Fraction on Mechanical Performance of Metakaolin Based Geopolymer Material

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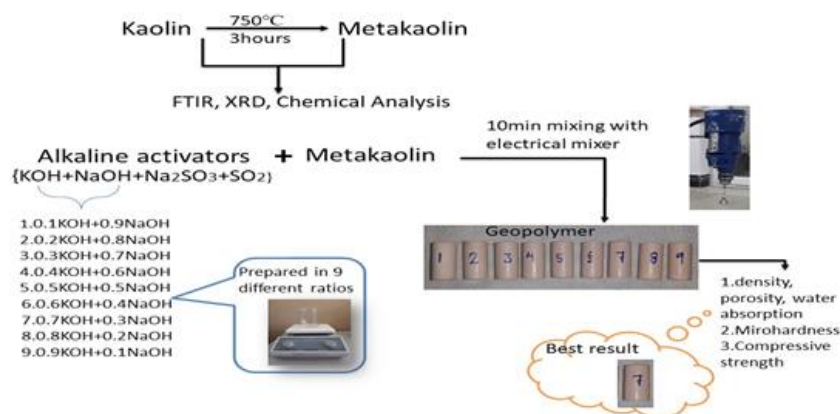
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ABSTRACT

Geopolymer is one of the newly promising materials with potential comprehensive implementation. Its parameters can affect its synthesis and mechanical performance, and accordingly influence the area of geopolymer applications. The type and concentration of alkaline activators is a powerful parameter that activate geopolymerization. In the following study, metakaolin was used as a source for aluminum and silicate. The aim of this study is to optimize the mole fraction effect of NaOH to KOH as mixed alkaline activators on the hardness and compressive strength of the geopolymer paste. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) were used as a mixed alkaline activator in 9 mole ratios from 0.1 up to 0.9, while keeping the ratio of NaOH and KOH to aluminum content is 1:1 of the final geopolymer formula. The other parameters were fixed like the silica/alumina ratio is equal to 3.8, the alkaline hydroxide to alkaline silicate is 3.02. The results showed that the best mole ratio was 0.3NaOH + 0.7KOH which gave the highest compressive strength and optimum hardness around 62.85MPs and 78.21VHN, respectively. It can be inferred that the metakaolin based geopolymer compressive strength can be modified by the type of used alkaline elements. The strength of the geopolymer can increase by increasing the amount of KOH related to NaOH up to 0.7 KOH + 0.3 NaOH. After this ratio, the compressive strength decreased. The effect of the little amount of Na element has vanished from the effect of the other K element.

GRAPHICAL ABSTRACT



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Introduction

Geopolymer is an inorganic polymer resulting from a reaction of aluminum silicate which provided from natural or synthetic sources and alkaline activated solution [1]. This reaction named a geopolymerization as it forms a polymeric Si-O-Al-O framework which becomes the binder with other elements [1]. The geopolymer term was firstly emerged in 1978 by Davidovats who found it can replace Portland cement because of its mechanical properties, durability, thermal stability, and more resistance to acid attack [2].

Geopolymer material has attractive properties like its preferable mechanical properties, not corroded, long lasting and can withstand high temperature, economy, and cost-effective as many raw materials can provide aluminum silicate, and less energy for its production. Therefore, geopolymer has become a fertile ground for research area [3, 4]. Embolden researchers to use this material in many study fields such as industrial, construction in civil engineering and even in biological studies. Some authors suggested the ability of using geopolymer as an implant or bone graft material [5, 6]. The potential application of geopolymer as a bone implant related to the ability of metakaolin based geopolymer to form a layer of hydroxy apatite on its surface after soaking in simulated body fluid [5].

The preparation conditions, type of raw source materials, and type of alkaline activated elements all can affect the mechanical properties of resulted geopolymer which can determine its specific application [4].

Raw materials obtained from the nature to produce aluminum and silicate can provide an economical way in industry. One of the best aluminum silicate sources is the metakaolin, which positively reacts with alkaline material and can be obtained easily from nature by calcining kaolin at a temperature from 750-800 °C to form amorphous to semicrystalline material [7].

The amount of compressive strength of geopolymer cement can be influenced by its chemical composition ($\text{SiO}_2/\text{Al}_2\text{O}_3$, $\text{R}_2\text{O}/\text{Al}_2\text{O}_3$, $\text{H}_2\text{O}/\text{M}_2\text{O}$ (R denotes Na or K) [8].

Geopolymer's alkaline oxide (R) ratio to Al_2O_3 ($\text{R}_2\text{O}/\text{Al}_2\text{O}_3$) were suggested to be around (1-1.2). This value seemed to be reasonable because the value of R^+ cations provide the positive charge that stabilizes the negative charge came from Al^{3+} in tetrahedral sites, leading to geopolymer formation [9, 10].

The effect of mixed alkali geopolymer on compressive strength instead of one type pure alkaline was studied by researchers like Duxson et al. (2007) who observed that the use of mixed alkali of Na and K in geopolymer synthesis with $\text{Si}/\text{Al} \geq 1.90$ can increase the compressive strengths compared with geopolymer specimens contain Na or K, the later specimens decrease strength notably [11, 12]. The compressive strength of geopolymer is time dependent property due to it increased with time increased [12]. The geopolymer made of mixed-alkali (Na and K) with high Si/Al ratio showed rise in its compressive strength after one to four weeks [10, 11].

This study attempts to evaluate the highest compressive strength and hardness of metakaolin based geopolymer through modifying the amount of NaOH to KOH moles, while keeping the sum of the mole fraction ratio to the Al is 1:1.

Materials and Methods

Metakaolin preparation and characterization

Kaolin (Dwaikhla western Iraqi desert) was calcined to transform to Metakaolin at 750 °C for 3 hours in non-vacuumed digital muffle furnace (Model (MF-12) 1200°C HYSC/ Korea) [12].

The structural functional groups of the raw kaolin and thermally treated metakaolin were identified by FTIR-8300 spectrometer (Shimadzu, Japan). All the samples were measured within the wave range of $4000\text{--}400\text{cm}^{-1}$ using KBr pellets. Geometrical state of kaolin and metakaolin powder was carried out by X-ray Diffractometer (XRD 6000, Shimadzu, Japan). The XRD patterns from Bragg's angle (2θ) of 5° to 60° , the scanning speed is 0.3 second per 1° step, generator settings of 30 mA and 40 kV and $\text{CuK}\alpha$ node material Cu (1.54 \AA).

A quantitative analysis of the chemical compounds that present in metakaolin

composition is revealed in Table 1 using Atomic Absorption devise (Varian, America). A 5 g of metakaolin powder was transformed to solution infused in a mixture of sodium carbonate and potassium carbonate was kept on sand for 2 days. Then, hydrochloric acid was added, after that filtration with the aid of filter paper, and washed with hot water. Silica sustained in filter paper determined by weight method, the remaining solution containing the rest of the elements were measured by atomic absorption of the optical light by free metallic ions

The optimum amount of (NaOH+KOH) mole fractions that form geopolymer formula

Geopolymer formula was used in this study (1(KOH+NaOH). 1Al₂O₃. 3.8 SiO₂. 9 mL H₂O), while keeping alkaline silicate to alkaline hydroxide ratio 3.02 following stoichiometric method. The amount of metakaolin used about 10.73 g [11].

A nine NaOH to KOH mole fraction was used from 0.1 up to 0.9, at the same time keeping this ratio equal to 1 to balance the negative charge of tetrahedral aluminum in general geopolymer formula. The required weights of alkaline activators constituents that achieved in the final geopolymer formula are provided in Table 2 for each NaOH to KOH mole fraction.

Table1: Chemical analysis of metakaolin

Compounds	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	LOI
Values (%)	54.84	38.73	2.58	0.89	0.56	0.31	<0.07	0.01	0.33	0.73

Table 2: KOH to NaOH mole fractions and their corresponding weights that form alkaline activators

Groups	Elements mole fraction		The required weight of alkaline activators (g)			
	KOH	NaOH	KOH	NaOH	Na ₂ SO ₃	SiO ₂
1	0.1	0.9	0.401	1.387	3.637	1.767
2	0.2	0.8	0.833	1.347	2.924	1.967
3	0.3	0.7	1.267	1.082	2.809	1.997
4	0.4	0.6	1.700	0.755	2.857	1.981
5	0.5	0.5	2.134	0.350	3.115	1.904
6	0.6	0.4	2.568	0.025	3.156	1.889
7	0.7	0.3	3.000	0.112	2.108	2.185
8	0.8	0.2	3.433	0.115	1.282	2.418
9	0.9	0.1	3.865	0.045	0.650	2.595

Geopolymer preparation

A stoichiometric weight of potassium hydroxide and sodium hydroxide (Avonchem, UK) dissolved in 9 mL of distilled water at room temperature. Then, water was added to about double the initial amount to facilitate the reaction. With the aid of magnetic stirring the solution is heated to 70 °C to dissolve the added sodium silicate (Thomas Baker, India). Finally, the amorphous silica (Thomas Baker, India) was added. After that, the mixture was kept on the stirrer at 10 rpm, till the excess water evaporated about one hour to ensure dissolving of silica. Next, the mixture solution was waited to cool gradually to room temperature. After cooling of the alkaline activator mixture was completed, the metakaolin 10.73 g is added and mixed for 10 min by

electrical mixer (700-1000) rpm. The mixture was then poured into cylindrical plastic molds (18, 36) mm diameter and length respectively. The specimens were de-molded after 2 days and kept in ambient temperature till testing procedure which was done after 28 days [11].

Testing geopolymer paste

The geopolymer density, porosity, and water absorption

The geopolymer samples were followed Archimedes method to obtain density, porosity, and water absorption percentage. After 28 days, three cylindrical samples of each test group were dried in oven at 150 °C for 6 hours, and then their weights were determined which represented the dry weight (D). After that, these samples were

submerged in distal water for 24 hours, to get the samples suspended weight (S). The saturated weight (M) was got in air after wiping the sample to get rid of the extra surface water. The equations 1, 2, and 3 determined the density, apparent porosity, and water adsorption.

$$\rho = D / (M - S) \quad (1)$$

$$Pa = (M - D / M - S) \times 100 \quad (2)$$

$$Wa (\%) = (M - D / D) \times 100 \quad (3)$$

Where, ρ = bulk density g/cm³, Pa= apparent porosity (%), and Wa= Water adsorption.

Microhardness test

Digital Vickers micro-hardness tester was used to record the micro hardness of the geopolymer specimens according to (ASTM E92-82, 1997), for 5 seconds 300 g load was applied to the surface of the geopolymer cylinder using (Digital display microhardness tester model MHV-2000S). An average of 3 different readings was measured for 7 samples of each test group.

Compressive strength

The compression test was done according to ASTM (E9-89a) at room temperature for 7 cylindrical shape specimens with length/diameter equal 2, for each group. The universal testing machine (JIANQIAO TESTING EQUIPMENT) model (CZL203- 2000 Kg) was used at the crosshead speed of 0.1mm/min and load of 2000 kg. The compressive strength was determined as the maximum stress at or before fracture by dividing the maximum load on the cross-sectional area, as demonstrated in following equation.

$$\sigma_c = P / A$$

Where, σ_c = compression strength (MPa), P = load that used (KN), and A= sample's area (mm²).

Results and Discussion

The kaolin FT-IR curve displayed in Figure 1 showed the remarkable peaks in the bands at 3697.66 cm⁻¹, 3622.44 cm⁻¹, and 3448.84 cm⁻¹ is representing O-H stretching vibrations of the internal surface [14]. The area of these peaks be a

straight curve for metakaolin indicating the loss of surface hydroxyl groups due to dehydroxylation of kaolin after calcination at 750 °C for 3 hours, as illustrated in Figure 1 black curve. The kaolin bands with the maximum peak at 1114.89 cm⁻¹ represent the Si-O stretching vibrations and the bands at 1037.74 cm⁻¹ and 1004.95 cm⁻¹ represent the Si-O stretching vibrations (quartz interference) is replaced by a single one band at 1068.6 cm⁻¹ in metakaolin [15]. The bands with the maximum peak at 788 cm⁻¹ and 751 cm⁻¹ in kaolin curve were replaced by a band with maximum curve at 794.7 cm⁻¹ in metakaolin which is the characteristics of the amorphous phase of kaolin. The disappearance of the 912.36 cm⁻¹, and 540.09 cm⁻¹ band from the IR spectrum for kaolin ensures that no trace of the kaolinite structure is retained in metakaolin. These results are in agreement with the previous work [14].

X-Ray diffraction analysis

The XRD pattern (red curve) in Figure 2 showed the narrow peaks with high intensities in the kaolin curve represented a highly crystalline powder. The strongest peaks pattern at 12.4722, 25.0854, and 26.7878 correspond with the intensity counts 538, 434, and 208, respectively. These peaks were comparable with the common characteristic peaks of kaolinite peaks were observed according to the International Center for Diffraction Data (ICDD = 00-001-0527) and (ICDD = 00-033-1161) for kaolinite and SiO₂ minerals, respectively. The sharp peak at 26.7878 refers to quartz [11]. The XRD pattern (black curve) in Figure 2 revealed a broad peak with low intensities observed that indicated obtaining an amorphous structure of metakaolin powder. Accordingly, this result reflected the mineral phase transformation of kaolin due to the heat treatment.

Density, porosity, and water absorption of the tested samples

The results in Table 3 revealed that the values of geopolymer density increase with the lowering of sodium concentration content replaced by

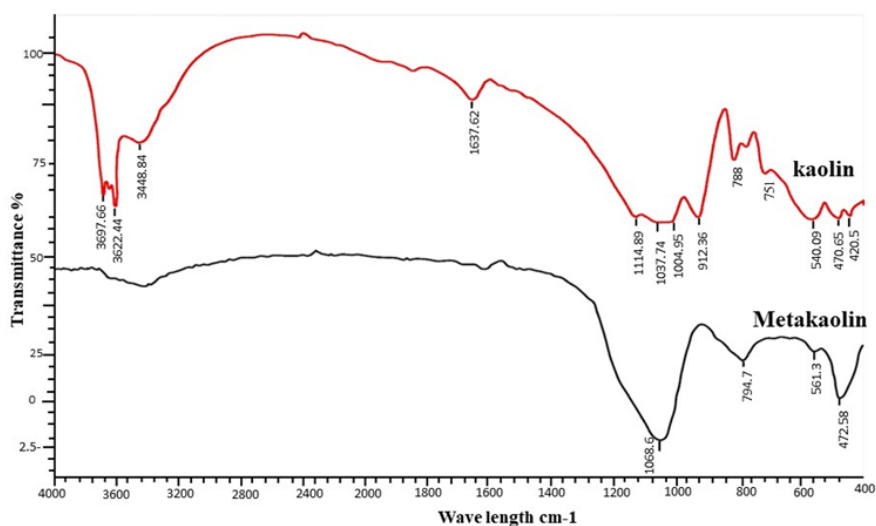


Figure 1: FT-IR curves for kaolin (red curve) for metakaolin (black curve)

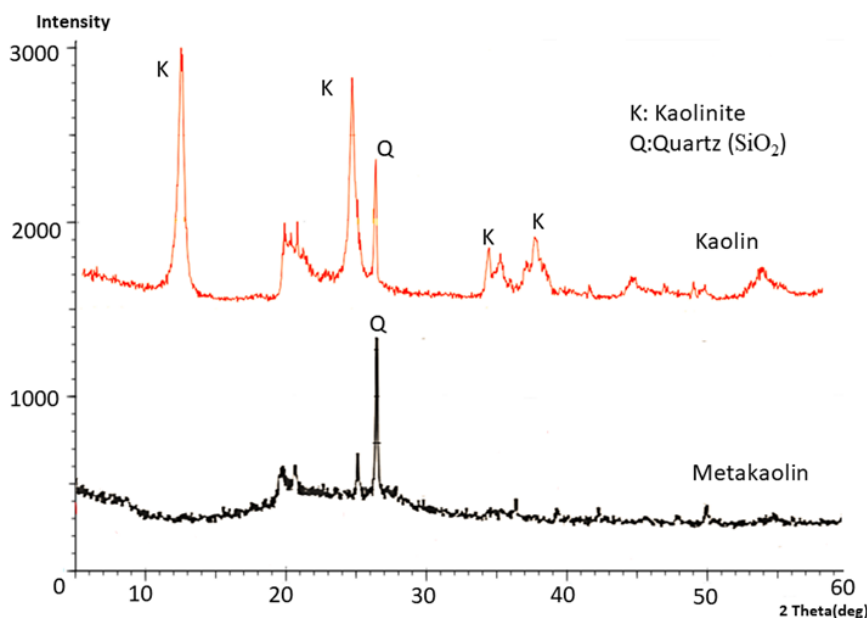


Figure 2: The XRD pattern for kaolin (black curve) and metakaolin (red curve)

potassium content, these results are in agreement with the previous studies [11, 16].

In this study, during the geopolymer preparation, it was observed the initial alkaline activator solutions based on more Na⁺ than K⁺ content be more viscous and induce faster polymerization under the same condition and water content, compared with geopolymer samples with more K⁺ content, which gave thinner initial mixture and slower polymerization. This observation is consistent with previous study [16]. Therefore, the geopolymer with more K⁺ content exhibited the slow initial hardening and more geopolymer

formation was obtained increasingly after long time. Subsequently increasing K⁺/ Na⁺ ratio in geopolymer samples gave more dense structure after 28 days [17].

This may be explained by that the larger particle size of K⁺ ion than Na⁺ ion. Therefore, it was suggested that the small Na⁺ cation binds to small silicate monomers and dimers, while the K⁺ cations bind with larger silicate oligomers. Since the difference in the size between K⁺ ion as it be larger than silicate oligomer, the bond will be strong which increases the geopolymer bulk density by increasing the amount of the K⁺ ion

[18]. The apparent porosity and water absorption are proportional properties since the apparent porosity reduces whenever the water absorption decreases. The apparent porosity and water absorption are inversely proportion to the bulk density, whenever the density increased the porosity and water absorption decrease, as presented in Table 3.

Microhardness

Table 4 and Figure 4 indicate the microhardness values of the means geopolymer test specimens with their standard deviation (SD). The increase in microhardness value with increasing the amount of KOH related to NaOH in the alkaline activator may be due to denser geopolymer with more K⁺ ion than Na⁺ ion as proved in

geopolymer density calculation in our study. Furthermore, it is known that the hardness property is proportional with the compressive strength [19]. Since the geopolymer compressive strength increased with more K⁺ ion to Na⁺ ratio [11], the tested geopolymer hardness increased accordingly.

Compressive strength

Sodium and potassium alkali activating solutions are usually used to synthesize geopolymeric materials both of them are of high significance for geopolymerization because they are able to provide an aqueous media to release the large amounts of hard silicate and aluminate from the source material that leads to polymerization [12].

Table 3: Density, porosity, and water absorption of the made geopolymers samples

Sample No.	Alkaline element Mole fraction		Density (g/cm ³)	Apparent Porosity (%)	Water absorption (%)
	K	Na			
1	0.1	0.9	1.286±0.071	40.8±0.034	31.9±0.045
2	0.2	0.8	1.281±0.032	40.2±0.038	31.4±0.037
3	0.3	0.7	1.317±0.044	39.9±0.056	28.9±0.019
4	0.4	0.6	1.314±0.077	39.8±0.030	30.2±0.006
5	0.5	0.5	1.395±0.163	36.8±0.051	27.1±0.048
6	0.6	0.4	1.414±0.181	39.2±0.038	27.9±0.031
7	0.7	0.3	1.444±0.038	33.7±0.056	23.4±0.039
8	0.8	0.2	1.359±0.04	36.8±0.043	27.2±0.039
9	0.9	0.1	1.305±0.074	41.0±0.020	28.9±0.048

Table 4: Geopolymer microhardness values at different K to Na ratios

Sample No.	1	2	3	4	5	6	7	8	9
Mean± SD	62.43±2.16	65.26±2.67	65.95±2.60	72.24±1.93	75.99±1.11	75.96±1.27	79.21±1.10	81.87±1.26	75.50±3.51

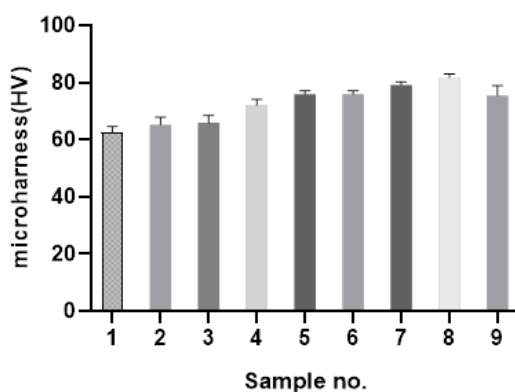


Figure 3: Effect of KOH+ NaOH mole fraction on the hardness of the studied geopolymers

The effect of mixed alkaline activators (Na⁺, K⁺) gave better geopolymer strength than using single alkaline activator due to the synergistic effect of the sodium and potassium elements [11, 12].

Table 5 and Figure 4 demonstrate the effect of increasing the K⁺ ratio to Na⁺ in geopolymer formula on the compressive strength of geopolymer after 28 days aging. It was found that the increase in the amount of compressive strength with the increase in the amount of KOH. The best compressive strength was obtained is equal to 62.8 MPa when K to Na ratio is (0.7 and 0.3), respectively.

These results agreed with the previous studies [11, 12]. It was noticed from the laboratory preparation of the 9 geopolymer ratios, before

specimens setting the geopolymer samples paste with more KOH than NaOH is more flowable. This may be explained by the larger size and less reactivity of K⁺ ion than Na⁺ ion led to slow polymerization and more compact material after long time (28 days), resulting in geopolymer with higher compressive strength [12], while the smaller Na⁺ ion leads to higher geopolymer strength at early time (7 days) [21].

After the 0.7K+0.3NaO ratio the amount of compressive strength decreased this may be due to the increasing the amount of KOH related to NaOH. Therefore, the effect of single compound (KOH) is more prominent than the effect of the other compound (NaOH). Thus, the synergistic effect of the mixed alkaline element will disappear.

Table 5: Compressive strength values of geopolymer at different KOH to NaOH ratio

Sample No.	1	2	3	4	5	6	7	8	9
Mean/MPa	35.94±2.122	39.92±1.374	41.50±2.396	45.83±0.9159	49.46±2.207	52.96±1.374	62.85±2.615	37.18±2.545	36.5±3.741

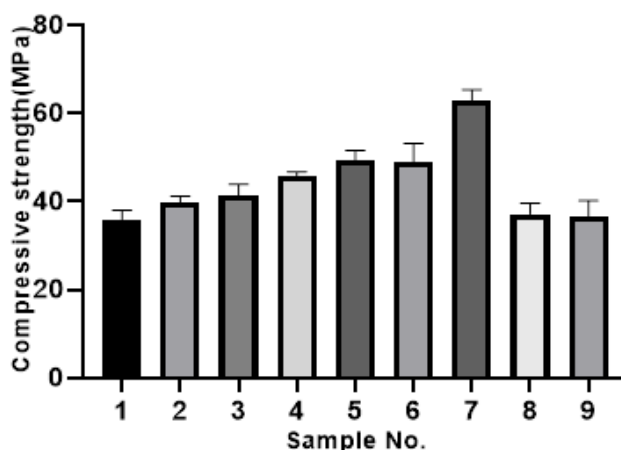


Figure 4: Effect of KOH-NaOH ratio on the compressive strength of geopolymers studied

Conclusion

Increasing the amount of potassium hydroxide related to sodium hydroxide in the alkaline activator while keeping this ratio equal to one, for geopolymer synthesis has a positive impact on its mechanical properties regarding the compressive strength and hardness. The value of geopolymer compressive strength and microhardness increased with increase KOH to the NaOH concentration. Regarding the compressive strength the (0.7KOH+0.3NaOH) ratio is the best

among other nine ratios used in this study. After this ratio, the effect of the little amount of Na element has vanished from the effect of the other K element. Type of alkaline activator is an effective parameter in geopolymer synthesis and if used in more than one type, the value determination of each type has an impact on the produced geopolymer. For future consideration, several trails to improve the mechanical properties of geopolymer paste material can act as a step for a wide range of employments even in the biomedical field and implant material.

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Authors' contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

Conflict of Interest

The author declared that they have no conflict of interest.

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