Metakaolin-red mud/carbon nanotubes geopolymer nanocomposite: mechanical properties and structural studies

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Abstract
In this research, the mechanical properties and structural studies of the geopolymer nanocomposite of metakaolin-red mud/carbon nanotubes were investigated. The geopolymer was designed by using metakaolin and red mud as aluminosilicate sources. Red mud replaced between 10 to 30% of the metakaolin. The mass ratio of the solid material and activator liquid was optimized in 1.5:1. Afterward, 1, 2, and 3 wt% multi-wall carbon nanotubes (MWCNTs) were dispersed in a polycarboxylate-based superplasticizer and were added to the geopolymer, which was mixed homogeneously. Geopolymer nanocomposites were characterized by XRD, FTIR, SEM, compressive and flexural strength measurements. The results indicated that the compressive and flexural strengths of the samples increased with curing time. The addition of red mud decreased the compressive and flexural strength of the geopolymer samples due to less reactivity and presence of a non-reactive impurity in red mud. By adding 2% MWCNTs, the compressive and flexural strengths increased to 37.05% and 36.06%, respectively, owing to the crack-bridging mechanism and filling of the cavities and porosity. FTIR spectra demonstrated the growth of the asymmetric stretching vibrations of T–O–Si (T: Si or Al) at approximately 995.85–1083.55 cm⁻¹, confirming the realization of the geopolymerization process in the structure.

1. Introduction
Geopolymers or inorganic polymers were successfully synthesized using aluminosilicate raw materials (including, fly ash, metakaolin, blast furnace slag, red mud and etc) at low temperature, which were activated by alkali. One of the main advantages of geopolymers against ordinary Portland cement (OPC) form is the much lower CO₂ emission rate during production and use. These materials display many excellent performances such as higher early-age strength, better volume stability, and durability, chemical resistance in addition to other features that are potentially better [1–3].

Recent studies were directed to the incorporation of the red mud in construction materials, traditional ceramics, clinker, mortar and concrete, cement, lightweight aggregates, and geopolymer [1, 4]. Red mud is the major industrial waste produced by the Bayer process for the extraction of alumina from bauxite ores [5]. Red mud contains an amount of alkali and aluminosilicates, so it creates the potential for the synthesis of polymeric materials. Some research in the synthesis of geopolymer with red mud has been carried out [1, 4–6]. More evidence point to red mud being a poor aluminosilicate geopolymer precursor, because the solubility of the aluminosilicate phases in alkaline conditions is low (i.e. low reactivity towards geopolymerization) as reported by Dimas et al [7]. Due to the low reactivity of red mud, the flexural strength of the geopolymer is reduced. For this purpose, the carbon nanotube (CNT) reinforcing phase is used to improve the mechanical properties of the geopolymer [8–14].
Chen et al. [9] claimed that CNT is a good choice as nano-reinforcements in OPC due to acceptable mechanical properties with a tensile strength of 63 GPa and Young’s modulus of 1 TPa. The authors pointed out in a review article the role of the CNT in the OPC matrix. They stated that CNTs could endure the load transferred from cement matrix, increasing conductivity and piezoresistivity of CNT–OPC composites and reduce the workability of the wet composite, if the CNTs uniformly dispersed [9]. Saffi et al. [10] examined the effect of multi-wall carbon nanotubes (MWCNT) on the mechanical and electrical properties of fly ash geopolymeric composite. Results revealed that the presence of MWCNT was useful and effective. The addition of MWCNT increased the flexural strength, Young’s modulus, flexural toughness, and electrical conductivity by 160%, 109%, 275%, and 194%, respectively. However, the uniform distribution of CNT must be considered in CNT-containing geopolymer. Heister et al. [11] showed that both CNT dimensions and surface functionalization have a significant influence on their dispersion. Khater et al. [12] examined the effect of MWCNT on the properties of slag geopolymer mortar. The results showed that the addition of MWCNT decreasing the water absorption values and then increases with further MWCNT increase and drying shrinkage in mortar composites with 0.1, 0.2, and 0.4% MWCNT reduced to about 92, 88, and 74% after 28 days, respectively [12]. Mesgari Abbasi et al. [13] investigated the effect of MWCNT on the mechanical behavior of metakaolin-based geopolymers. The researchers elaborated on this issue that the compressive and flexural strength were augmented up to 32% and 28%, respectively, as the carbon nanotubes were added about 0.5% [13]. Khater et al. [14] used MWCNT to the production of sulfate resistance geopolymer composite. The results displayed that the addition of MWCNT increased the sulfate resistance [14].

Carbon nanotubes are known for their structural nature as an important reinforcement in the construction of aluminosilicate-based ceramic bodies. The mechanism of increasing strength and resistance to failure in their presence is done by bridging and filling cavities and porosity. The aim of this study was to investigate the effect of percentage and distribution of carbon nanotubes on the mechanical and microstructural properties of geopolymer. Accordingly, the aim of this study is to evaluate mechanical properties and structural studies of geopolymer nanocomposite of metakaolin–red mud/carbon nanotubes, which were synthesized using 10 and 30 wt% red mud and 1–3 wt% MWCNT. These samples were characterized using XRD, FTIR, and SEM along with compressive and flexural strength measurements.

2. Materials and methods

2.1. Materials
For the production of geopolymer materials, Metakaolinite was obtained by calcining kaolinite (Code: ZK2, Iran China Clay Industries Co.) at 700 °C for 10 h. The main chemical composition before and after calcining as

| wt. Percent | SiO₂ | Al₂O₃ | TiO₂ | Fe₂O₃ | MgO | CaO | Na₂O | K₂O | L.O.I |
|-------------|------|-------|------|-------|-----|-----|------|-----|-----|
| ZK2 Kaolin  | 73   | 14.5  | 0.04 | 0.7   | 0.25| 5   | 0.2  | 0.2 | 7   |
| calcined ZK2 Kaolin | 75.1 | 14.9  | 0.04 | 0.7   | 0.26| 4.3 | 0.2  | 0.2 | 4.3 |
| red mud [15] | 17.18| 15.36 | 6.35 | 25.63 | 1.56| 19.01| 1.59 | 0.56| 12.56|

Figure 1. XRD pattern of calcined ZK2 Kaolin.
determined by x-ray fluorescence and the x-ray diffraction analysis of calcined kaolin are given in table 1 and figure 1.

Red mud was obtained as a byproduct of the Bayer process of obtaining alumina (production of alumina plant of Iran in Jajarm), which was also used in research by co-author [15]. Table 1 and figure 2 indicate the chemical analysis and XRD pattern of red mud. Figure 3 shows the SEM micrograph of the structure of red mud and metakaolin.

8 M industrial-grade NaOH pellets (99% purity), and sodium silicate (9.75% of Na₂O, 29.25% of SiO₂, 61% of H₂O) solutions were mixed at a mass fraction of 1:2 sequentially and applied as the activator solutions in order to make red mud and metakaolin-based geopolymers prepared. It should be noted that no changes or dilutions
Table 2. The Physical properties of MWCNTs.

| %Amorphous carbon | Surface area (m²/g) | % Purity | % Ash | Ash (µm) | Outer diameter (nm) |
|-------------------|----------------------|----------|-------|----------|---------------------|
| 3                 | 250–280              | > 95     | 0.2   | 10       | 10–20               |

Table 3. Synthesis conditions used in red mud-metakaolin (GRM) based geopolymer.

| Sample codes | CNT (%) | Calcined kaolin (gr) | Red mud (gr) | Alkaline solution (gr) |
|--------------|---------|----------------------|--------------|------------------------|
| GRM10        | 0       | 202                  | 33 (10 wt%)  | 150                    |
| GRM30        | 0       | 156                  | 69 (30 wt%)  | 150                    |
| GRM10-CNT 1  | 1       | 202                  | 33           | 150                    |
| GRM10-CNT 2  | 2       | 202                  | 33           | 150                    |
| GRM10-CNT 3  | 3       | 202                  | 33           | 150                    |
| GRM30-CNT 1  | 1       | 156                  | 69           | 150                    |
| GRM30-CNT 2  | 2       | 156                  | 69           | 150                    |
| GRM30-CNT 3  | 3       | 156                  | 69           | 150                    |

Figure 4. XRD patterns of all geopolymer samples; MK: calcined kaolin, K: Kaolinite, Q: Quartz, C: Calcite, A: Albite, O: Orthoclase, F: Hematite, G: Goethite (FeO(OH)), S: Sodalite (Na₈(Al₆Si₆O₂₄)Cl₂).
were made in the preparation of the activator (combination of sodium silicate and sodium hydroxide with the mentioned specifications) during mixing.

MWCNTs were synthesized (in Research Institute of Petroleum Industry (RIPI) of Iran) by using Co-MgO-based catalysts. The physical properties are given in Table 2.

2.2. Preparation and dispersing of the MWCNTs

Usually, CNT is difficult to disperse and is adhered together due to Van der Waals forces [16]. By applying polycarboxylate-based superplasticizer, Shah et al realized an effective distribution of MWCNTs with different lengths and concentrations in cementitious materials [12, 16]. In this study, Ether polycarboxylate-based superplasticizer (code: Carboxal HF5000, Alborz Chemical Co.) was mixed with the MWCNTs for dispersing. The solution was ultra-sonicated in a water bath and added to geopolymers paste at ambient temperature for 20 min. Furthermore, The superplasticizer was added to all samples at a ratio of 0.3% MWCNTs.

2.3. Sample preparation

Geopolymers were designed by using metakaolin and red mud as aluminosilicate sources. Red mud replaced between 10 to 30% of the metakaolin. The mass ratio of the solid material and activator liquid for the reaction optimized as 1.5:1. The dry solid materials and activator solution were mixed for 5 min, followed by addition of solutions containing 1, 2, and 3 wt%MWCNT. In order to accomplish the compressive and flexural strengths tests, the geopolymer pastes were rapidly poured into a cubic brass mold with 50 mm × 50 mm × 50 mm and

![Figure 5. Geopolymer compressive strength change during curing time for (a) 10 GRM, and (b) 30 GRM samples with and without CNT.](image)
plaster of Paris mold with 19 mm × 28 mm × 200 mm. Then, within a saturated humid environment, the samples were cured at atmospheric pressure at ambient temperature before the tests. The samples were tested at 3, 7, 14, and 28 days. The details of the mixed design are shown in table 3.

2.4. Characterization methods
Based on the ASTM C 109 and ASTM C 109 standards, the compressive and the flexural strength were measured using the Hydraulic universal Testing Machine (UTM) the Compression Test machine (ELE) Model No. 36-4150/01 and the Hydraulic universal Testing Machine Model No. WAW-300 E, respectively. By x-ray diffraction (XRD) analysis using a GNR-MPD 3000 Diffractometer with Cu-Kα radiation phase identification of the samples was accomplished. The samples were verified with FESEM (Hitachi, S-4160) after polishing, etching and coating with a thin film of gold. Having used the KBr pellet technique, the FT-IR spectra of the samples were obtained in the 400–4000 cm⁻¹ range with a spectrometer at room temperature (Bruker, Vertex70).

3. Results and discussion
3.1. X-ray diffraction patterns
Figure 1 displays the XRD pattern of calcined kaolin. Kaolinite peaks were still observed in the diffractogram, suggesting that the thermal conversion of kaolin into metakaolin was not complete. On the other hand, the calcined kaolin also shows a broad hump in the 2θ = 10°–26° range, indicating the presence of noncrystalline phases such as metakaolin and disordered silica units (figures 1 and 4). The geopolymer specimens are cured at ambient temperature, so clearly they are not calcined.

Table 4. The Compressive and flexural strength of the geopolymer samples with and without CNT.

| Sample code          | Compressive strength (MPa) - curing time (day) | Flexural strength (MPa) - curing time (day) |
|----------------------|-----------------------------------------------|-------------------------------------------|
|                      | 3 days | 7 days | 14 days | 28 days | 3 days | 7 days | 28 days |
| GRM 10               | 9.23   | 16.93  | 17.34   | 20.16   | 2.95   | 4.7    | 5.38    |
| GRM 30               | 7.98   | 8.76   | 9.9     | 10.77   | 2.65   | 2.91   | 3.18    |
| GRM10-CNT 1          | 10.92  | 20.74  | 22.2    | 23.33   | —      | —      | 6.34    |
| GRM10-CNT 2          | 14.85  | 22.56  | 25.64   | 27.63   | —      | —      | 7.32    |
| GRM10-CNT 3          | 12.32  | 21.43  | 24.01   | 25.66   | —      | —      | 6.94    |
| GRM30-CNT 1          | 8.37   | 16.99  | 18.78   | 19.58   | —      | —      | 5.4     |
| GRM30-CNT 2          | 9.81   | 18.68  | 20.69   | 21.53   | —      | —      | 6.04    |
| GRM30-CNT 3          | 8.91   | 18.28  | 19.31   | 20.55   | —      | —      | 5.69    |
The sharp peaks also indicate the presence of crystalline phases, including quartz, Albite, Orthoclase, Calcite, and Kaolinite. Figure 2 presents the XRD spectra of Red mud. Red mud contained Hematite, Calcite, Sodium calcium silicon oxide, Kaolinite, Katoite, Anatase, Rutile, and Cancrinite phases but no observable broad humps, suggesting that the amorphous phases are not present in any large quantity.

The XRD spectra of geopolymer samples are shown in Figure 4. For all geopolymers, a weak broad hump to the $2\theta = 10^\circ$–$20^\circ$ is observed. The patterns of the geopolymers show the presence of peaks of crystalline phases from parent materials, both red mud, and calcined kaolin, with variable peak intensities, as reflected by the peak intensities from quartz and minerals in red mud. This confirms that the crystalline phases are not reactive or involved in geopolymerization, but simply present as inactive fillers in the geopolymer network.

### 3.2. Compressive and flexural strengths

Table 4, and figures 5 and 6 depict the compressive and flexural strengths of the geopolymer samples. The results showed that the compressive and flexural strengths of samples have been increased with curing time. Longer curing time improves the geopolymerization state resulting in higher compressive and flexural strengths. It was also observed that increasing the red mud from 10 to 30% reduced the compressive and flexural strengths of samples. Xu et al [17, 18] pointed out that high-reactivity source materials are usually needed to synthesize a high-strength geopolymer. Generally, the calcined substances are more reactive, since most crystalline sources at high temperatures collapse and are amorphous after calcination [18]. In this research, kaolin and red mud were
used as calcined and uncalcined, respectively. Regarding the above-mentioned cases, the reduction of strengths is observed with the increase of red mud by replacing calcined kaolin with uncalcined red mud.

In addition to the above, it seems that another parameter also contributes to strength reduction. Phase analyses by XRD suggests that red mud contains higher quantities of non-reactive crystalline phases (e.g., calcite and hematite) compared to calcined kaolin. Therefore, geopolymerization in metakaolin-based geopolymers are
more complete than red mud—containing geopolymers, resulting in greater strength. According to Duxson [19], these impurities in source materials may increase reaction pathways during geopolymerization, which causes some changes in properties during synthesis and in the final product. One of the most important impurities is iron as hematite in red mud, which according to table 1 is 28.78%. Kaya et al [2] stated that reduced strength due to increased red mud could be related to iron ions. Based on what Kaya et al [2] asserted the iron species obtained from red mud in this system is considered as a significant agent in observed the structural and mechanical specifications. Iron species in the geopolymer system inclines to re-deposit quickly in the form of hydroxide or oxyhydroxide phases eliminating OH− ions from the solution phase and decrease the dissolution of the residual raw materials, in this case, red mud and calcined kaolin [2].

Figures 7 and 8 show carbon nanotubes changes on compressive and flexural strengths. It is observed that in the GRM10 sample, by adding 1% carbon nanotubes, the compressive strength increased from 20.16 MPa to 23.33 MPa and the flexural strength increased from 5.38 MPa to 6.34 MPa in 28 days. That is, the compressive and flexural strengths increased by 15.72% and 17.84%, respectively. By adding 2% carbon nanotubes, the compressive and the flexural strengths increased to 37.05% and 36.06%, respectively due to the bridging mechanism. In fact, due to the reaction between carbon nanotubes and structure components, nanotubes act like bridges against cracks and prevent the growth of cracks in the field [12, 13, and 20]. On the other hand, the proper distribution of CNT can contribute to the increase of compressive and flexural strengths, which can be due to hydrophilic hydrocarbon groups (–COOH) of polycarboxylate plasticizers forming covalence-modified CNTs to improve interfacial interactions in composites. The addition of CNT may also fill the cavities and porosity or reduce their size, which results in structural compactness and increased compressive strength and flexural strengths [12].

According to figures 7 and 8, the compressive and the flexural strengths of the samples increase to 2% of CNT and then decrease. In the GRM10 sample, the compressive strength of 28 days (compared to without CNT) increased by 15.72% by adding 1% of CNT. However, with the addition of 3% of CNT (in the GRM10 sample), the compressive strength of 28 days from 20.16 MPa (in the no CNT sample) reached 25.66 MPa. Although the compressive strength of this sample increased by 27.28%, in contrast to the 2% CNT sample, it is about 7.13% less strength. The flexural strength is like compressive strength. Although, with the addition of 3% CNT, flexural strength has increased compared to the control sample (without CNT), but decreased compared to 2% CNT.

The distribution of CNT in geopolymer is a very important factor for observing the effect of CNT in the matrix so that its poor distribution causes flaws in the matrix and reduces the CNT reinforcement effect. In
Figure 9. Microstructure images of geopolymers made with carbon nanotubes are shown. The phenomenon of bridging by nanotubes is seen in the images (as figure 9(a)). Nanotubes, by bridging the particles, can enhance the mechanical properties of the samples. In the sample, GRM10-CNT2 (figure 9(b)), the number of nanotubes is higher, and the bridging is far better. But in GRM10-CNT3 (figure 9(c)), the amount of nanotubes has increased, causing their agglomeration. This has considerably weakened the pull-out and ‘crack-bridging mechanism’ and weakened mechanical properties. This is the same as increasing the number of carbon nanotubes in GRT30-CNT2 and GRM30-CNT3 specimens (figures 9(e) and (f)). Khater et al [12] stated that excessive CNT was agglomerated in the aluminosilicate gel due to the carboxylate-based superplasticizer was ineffective in dispersing in larger quantities CNT and prevent the distribution of the three-dimensional networks. Therefore, the reduction of compressive and flexural strengths by more than 2% could be due to agglomeration and the non-uniform distribution of carbon nanotubes in the geopolymer matrix. On the other hand, according to the results of table 4, it seems that only by adding 2% CNT to the specimen containing 30% red mud (GRM30-CNT2), it can be strengthened to near the strength of 10% red mud. In fact, the decrease in strength due to the addition of red mud can be compensated by adding a small amount of CNT.

3.3. Infrared spectroscopy
Fourier transform infrared (FTIR) spectroscopy was used to investigate the formation of a geopolymer network. In the IR spectrum of calcined kaolin (MK), bands are detected at 472.96, 798.79, 1094.40, 1632.91, and 3419.12 cm$^{-1}$ (figure 10). According to others [21, 22], peaks of 1632.91, and 3419.12 cm$^{-1}$ show stretching vibrations of OH groups. The most intense, asymmetric, broad feature at 1094.40 cm$^{-1}$ is attributed to stretching Si–O bands. The broadband located in the mid-frequency region at about 798.79 cm$^{-1}$ was attributed...
to the Al–O stretching vibrations in AlO$_4$ tetrahedra in metakaolin. The low-frequency band at 472.96 cm$^{-1}$ was speculated to be related to the Al–O–Si bridge of aluminosilicates [21, 22]. Figure 10 demonstrates FTIR spectrum of untreated raw red mud. Mucsi et al and Kaya et al have allocated the band at the range of 990–1090 cm$^{-1}$ to the possible mode of Si–O–Si and Al–O–Si bonds. As a result, the band observed at 994.84 cm$^{-1}$ in untreated raw red mud might be due to asymmetrical stretching vibrations of the Si–O–Si and Al–O–Si bonds mostly in sodalite/cancrinite phases that were found in XRD patterns [2, 23]. The band vibrations of 4-membered and 6-membered rings from the cancrinite ε-cage framework indicated to be active in the IR spectrum of red mud and samples at 622.20 cm$^{-1}$ [24]. The bands at about 470 cm$^{-1}$ are due to the
stretching vibration of Fe–O related to the iron compounds such as hematite in red mud [25]. This band is also attributed to the bending vibrations of Si–O–Si, and O–Si–O bonds [26].

Figure 10 shows FT-IR spectrum of geopolymer samples in the range of 400 and 4000 cm$^{-1}$. In the IR spectrum of all geopolymer samples, the peak formed in the range of 995.85–1083.55 cm$^{-1}$ is attributed to asymmetric stretching vibrations of T–O–Si (T: Si or Al) bonds that are formed as a result of TO4 reorganization during geopolymer formation, which confirms the realization of the geopolymerization process in the structure [2]. The peaks in the range of 1652.98–1636.73 cm$^{-1}$ and 3852.00–3412.28 cm$^{-1}$ show stretching vibrations of OH groups in the water molecule of geopolymer structure [2, 21].

The location of the main Si–O–T stretching band indicates the length and angle of the Si–O bonds in the silicate network and stretching vibration of inner SiO2 groups, and this peak occurs at about 1100 cm$^{-1}$ for amorphous silica [27–29], in this research, main Si–O–T stretching band occurs at 1094.40 cm$^{-1}$ indicating the
stretching vibrations of surfaces SiO$_2$ groups [29]. According to figure 10, the peak of 1094.40 cm$^{-1}$ has shifted to 1017.85–1083.55 cm$^{-1}$ in GRM 10 CNT geopolymer samples and 995.85–1036.92 cm$^{-1}$ in GRM 30 CNT geopolymer samples attributed to the asymmetrical stretching vibrations of Al–O and Si–O bonds [30–32]. This shift shows that the solid silica is dissolving and changing in molecular structure [27]. Shifts to lower wave numbers in geopolymer samples indicate changes in the silicate network and the dissolution of the initial solid materials and the formation of a new aluminosilicate phase during geopolymerization. An increase of non-bridging oxygen in silicate sites, charge balancing by sodium cations in the system, or increasing the substitution of tetrahedral Al in the silicate network can be some of these changes [8, 27]. Therefore, this displacement can be due to the incorporation of Al$^{3+}$ to SiO$_4$ tetrahedral forming the Si–O–Al stretching vibrations bond of the geopolymer network which indicates the continuously geopolymerization process [27, 30]. A band of OH$^-$ stretching vibration positioned at around 3400 cm$^{-1}$ in the geopolymeric powder. Due to the presence of weak H$_2$O bonds adsorbed at the surface or caught in cavities of the geopolymer structure H–O–H bending vibrations of water molecules was also found at around 1645 cm$^{-1}$ [2, 30]. At about 2360 cm$^{-1}$, an H–O–H vibration under a strong hydrogen bond was located [30, 33]. Stretching vibrations of O–C–O bonds might have been conductive to the appearance of peaks in the range of 1434–1525 cm$^{-1}$, mainly because of the existence of carbonate in the samples [8]. The bands in the range of 793–799 cm$^{-1}$ are assigned to the symmetric stretching vibrations of Si–O–Si and Al–O–Si bonds [2, 12, and 14]. The bands in the range of 463–484 cm$^{-1}$ possibly were due to the bending vibrations of Fe–O [2]. As Khater states, this band could also be relevant to (Si–O–Si and O–Si–O) bending vibration due to quartz [14].

4. Conclusions

The result of this study focused on the evolution of the mechanical properties and structural studies of geopolymer nanocomposite of metakaolin–red mud/carbon nanotubes, that were synthesized using 10 and 30 wt% red mud and 1–3 wt% MWCNT and investigated by XRD, FTIR, SEM, compressive, and flexural strength measurements. The results showed that the compressive and flexural strengths of samples increased with curing time. The addition of red mud was reduced the compressive and flexural strength of geopolymer samples due to less reactivity and the presence of non-reactive impurity in red mud. By adding 2% carbon nanotubes, the compressive and the flexural strengths increased to 37.05% and 36.06%, respectively due to the bridging mechanism and filling the cavities and porosity. FTIR spectra showed the growth of the asymmetric stretching vibrations of T–O–Si (T: Si or Al) at about 995.85–1083.35 cm$^{-1}$, which confirms the realization of the geopolymerization process in the structure.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Availability of data and materials

The data presented in this study are available on request from the corresponding author.

Competing interests

The authors declare no conflict of interest.

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Authors’ Contributions

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