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Case study

Mechanical properties and electrical resistivity of multiwall carbon nanotubes incorporated into high calcium fly ash geopolymer

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ABSTRACT

High calcium fly ash (HCF) is a pozzolan material and is available in large quantity in Thailand due to the existence of coal-based electrical power plants. It is used as a supplemental material to partially replace cement content in concrete as a movement toward concrete sustainability. In order to lift the sustainability level, a cementitious material without Portland cement called 'geopolymer' was introduced. Geopolymer can be produced from raw materials containing high alumina and silica, for example fly ash, blast furnace slag, and metakaolin. For high calcium fly ash geopolymer (HCFG), the unique properties include fast setting, and high early strength. In this study, in order to enhance the properties of HCF geopolymer, multiwall carbon nanotubes (MWCNTs) were introduced into the matrix. In addition to the investigation into basic properties, the effect of MWCNT on electrical resistivity was also investigated to determine its potential use in piezoelectric sensor applications. The results showed that the addition of MWCNTs improved the mechanical properties of HCFG. The maximum compressive and flexural strengths were obtained with a mix containing 0.2% MWCNTs. The EDS test also indicated the increase in geopolymerization and hydration products with the addition of MWCNTs. To investigate the piezoelectricity potential, the electrical resistivity under different levels of compression loads was investigated. The resistivity decreased with the increasing load level up to the first crack, and then decreased. The changes in electrical resistivity indicated the potential use of HCFG incorporated MWCNTs in self-sensing for structural health monitoring.

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1. Introduction

Geopolymer is a cementitious material synthesized from chemical reactions between alumino-silicates and alkali activator solution [1]. In general, materials with high aluminosilicate content, such as fly ash, ground granulated blast furnace slag, and metakaolin can be used as base materials to produce geopolymer. Although the properties of geopolymer depend mainly on base materials, but in general they exhibited relatively good strength, low permeability, and good durability compared to Portland cement system [2–5]. Geopolymer becomes not only an alternative cementitious material but also an environmentally friendly construction material because most raw materials used in geopolymer production are by-products or waste from other industrial processes. In Thailand, HCF is a byproduct from coal-based power plants, has been utilized as a main base material for geopolymer production. General properties of HCF geopolymer include fast setting, and high early strength compared to geopolymer with other base materials [6–8].

Generally, there are several ways to enhance the properties of concrete. For example, the use of short fibers to enhance concrete ductility and impact resistance [9–11], the use of phase change material to enhance concrete thermal properties [12–15], the use of viscoelastic material to enhance concrete damping properties [16], or the use of nanomaterials like carbon nanotube (CNTs) to enhance concrete fire resistance [5]. In this study, the focus is on the use of MWCNTs to enhance the properties of HCFG and to investigate its piezoelectric potential.

MWCNTs are a type of CNTs which is allotropes of carbon in tubular shape made of graphite. The tubes usually contain at least two layers with outer diameter ranging from 3 nm to 30 nm and a length-to-diameter ratio greater than 1000 [17]. CNTs have been used in various applications due to its excellent electrical, mechanical, and thermal properties, for example, enzymatic-based biosensors in biomedical field, microelectronics in electronic parts, energy storage, gas and water filtration [18–22]. In the case of civil engineering, the CNTs have been used in cementitious material to enhance mechanical properties and durability of Portland cement concrete. The addition of CNTs is capable of reducing porosity by filling up voids in hydrate gel and also improve the hydration reaction by increasing density of calcium silicate hydrate (C-S-H) products [23–25]. The distribution of CNTs in cement matrix acts also like reinforcing fibers which leads to the increase in strength and the reduction of microcracks [26–28]. In addition, the CNTs also act as a semiconductor to provide cement with higher electrical conductivity which can be used in self-sensor material application (i.e., cement-based sensor) [29–31].

For the geopolymer, the application of CNTs can enhance its properties. However, the properties of geopolymer mixed with CNTs depend strongly on the dispersion method of CNTs due to high specific surface area and the bond strength between carbon molecules [32,33]. Luz et al. [34] and Abbasi et al. [35] dissolved the MWCNTs in polycarboxylate-based superplasticizer using ultrasonication and mixed with metakaolin geopolymer at the rate of 0.1–0.5% by weight of binding materials. The increase in 32% compressive and 65% flexural strengths were observed in their study. In the case of low calcium fly ash geopolymer, the addition of MWCNTs less than 0.5% by weight also improved the flexural strength, toughness, and elastic modulus. The electrical resistivity of low calcium fly ash geopolymer also decreased with the increasing MWCNTs due to the improvement of conductivity network [36–38].

In the case of high calcium fly ash geopolymer mixed with MWCNTs, a number of studies have been carried out. Heister et al. [39] reported that the use of sodium hydroxide solution (NaOH) can eliminate the electrons on the carbon nanotube surface to achieve better dispersion. Jittabut and Horpibulsuk [40] mixed the MWCNTs and NaOH with different concentrations and found that the 15 M concentration gave the highest improvement in the compressive strength. The optimum for the MWCNTs dosage was observed at 1%. Unfortunately, both studies did not investigate the effect of MWCNTs on the electrical properties of HCFG. Therefore, there is a lack of information in this regard.

In this study, the effect of MWCNTs on mechanical and electrical properties of HCFG was investigated. The MWCNTs were incorporated into geopolymer mortar up to 0.6% by weight of fly ash. Additional studies also included the EDS analysis and piezoelectric property of geopolymer mixed with MWCNTs by measuring the variation of electrical resistivity when subjected to load up to about 20% of ultimate load.

2. Experimental procedure

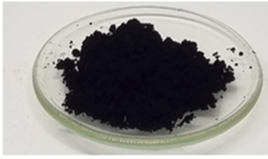
2.1. Materials

High calcium fly ash (FA) with specific gravity of 2.61 and its chemical composition are shown in Table 1. As well-known, geopolymer is a friendly construction material and also the waste river sand with grain size between 1.19 and 4.75 mm was used. was used as fine aggregate. It had specific gravity of 2.58, fineness modulus of 2.85, and water absorption of 0.91%. Alkali activator solution consisted of sodium hydroxide (NaOH) and sodium metasilicate (Na_2SiO_3) solutions. The 12 M NaOH solution was prepared by dissolving NaOH flakes with deionized water and left to saturate for 24 h. For Na_2SiO_3 solution, the chemical compositions consisted of 34.2% SiO_2, 16.3% Na_2O and 49.5% water. MWCNTs are obtained by using a chemical vapor deposition (CVD) process which is a domestic industrial product and the properties are shown in Table 2. Superplasticizer which consists of formaldehyde and melamine

Table 1
Chemical composition of fly ash (by weight).

Element (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	LOI
Fly ash	31.85	15.89	14.07	26.76	3.66	1.95	1.95	2.45	0.17

Table 2
Properties of MWCNT.

Properties	Description	Appearance
Type	Multi-wall carbon nanotube	
Appearance color	Black	
Diameter	12 nm.	
Length	3–12 μm.	
Thickness	3 nm.	

based on type A&F (ASTM C494-81) was used as a surfactant. The electrodes were made of copper sheet C1100 grade (99.96% pure copper) with the dimension of 10 × 70 × 0.29 mm.

2.2. Mix proportion and specimen preparation

The mix proportion of HCFG with MWCNTs is given in Table 3. The amount of MWCNT of 0%, 0.1%, 0.2%, 0.3%, 0.4%, 0.5% and 0.6% by weight of fly ash was used. The liquid to binder (L/B) ratio was set at 0.45 and the sodium hydroxide to sodium silicate ratio (NaOH: Na_2SiO_3) was 1. The superplasticizer (SP) was added into mixed proportion with 5% of fly ash weight.

From several research [34,35,39], the MWCNTs can well dissolve with superplasticizers and NaOH. Prior to the start of mixing process, MWCNT and superplasticizer were premixed in NaOH solution. To disperse them, they were blended in NaOH solution for 20–30 s. The mixing process began with dry mixing of sand and fly ash for 1 min. The premixed NaOH solution were then poured into the dry base (sand and fly ash) and mixed for 1 min. The Na_2SiO_3 solution was subsequently added and the mixing was continued for another 1 min. The fresh geopolymer was cast into molds by filling one half at a time. Each filling was consolidated with a vibrating table to remove air voids. Two specimen types were prepared: cubic specimens with dimension of 50 × 50 × 50 mm for compression test and prism specimens with dimension of 40 × 40 × 160 mm for flexure test. For electrical resistivity measurement, the cubic specimen was mounted with four strips of 10 × 70 × 0.29 mm copper plates as electrical terminals.

After the casting, all specimens were stored in a room with controlled temperature of 25 °C and relative humidity of 50%, demolded at 24 h after casting and wrapped with a plastic sheet for 28 days. The specimen weight and dimension were measured before the test.

2.3. Experimental series

The experiment series consist of 4 tests: flow test, compression test, flexural test, and electrical resistivity test. For each test, at least three specimens were tested.

1. The mini-slump flow test was carried out in accordance with the ASTM C1437-20. The fresh geopolymer mortar was poured into a mold and compacted by tamping for 20 times. After compaction, the mold was then lifted up slowly in vertical direction which allowed the mortar to flow freely. The diameter of the mortar after free flowing was then measured in four perpendicular directions. The average of free-flow diameter was then compared with the original diameter to determine flow percentage.
2. The compression test was carried out in accordance with the ASTM C109/C109M - 20b using a universal testing machine. The load was applied at the rate of 1000 N/s until failure.
3. The flexural test was conducted following the ASTM C348-20 using three-point loading. The clear span was set at 120 mm and loading rate was set at 2640 N/s.
4. The electrical resistivity test was carried out by measuring the change in electrical resistivity under different levels of applied loads. During testing, the electrical resistances were measured by an LCR meter with four-probe technique. The direct current (DC) of 1000 mV was supplied to a specimen via two outer terminals while the resistance was measured from two inner terminals. During the test, the specimen was placed and held by a steel support, and then a compressive load was applied to the specimen using an instrumented torque wrench. The wrench was calibrated with a load cell. The load applied to specimens was increased at a step of 5% of specimen's ultimate load to the highest level of 20% (Fig. 1).

Table 3
Mix proportion (kg/m^3).

Label	MWCNTs	Fly ash	Fine aggregate	NaOH solution	Na_2SiO_3 solution	SP
0 M (control)	0	880	1100	200	200	44
0.1 M	0.88					
0.2 M	1.76					
0.3 M	2.64					
0.4 M	3.52					
0.5 M	4.40					
0.6 M	5.28					

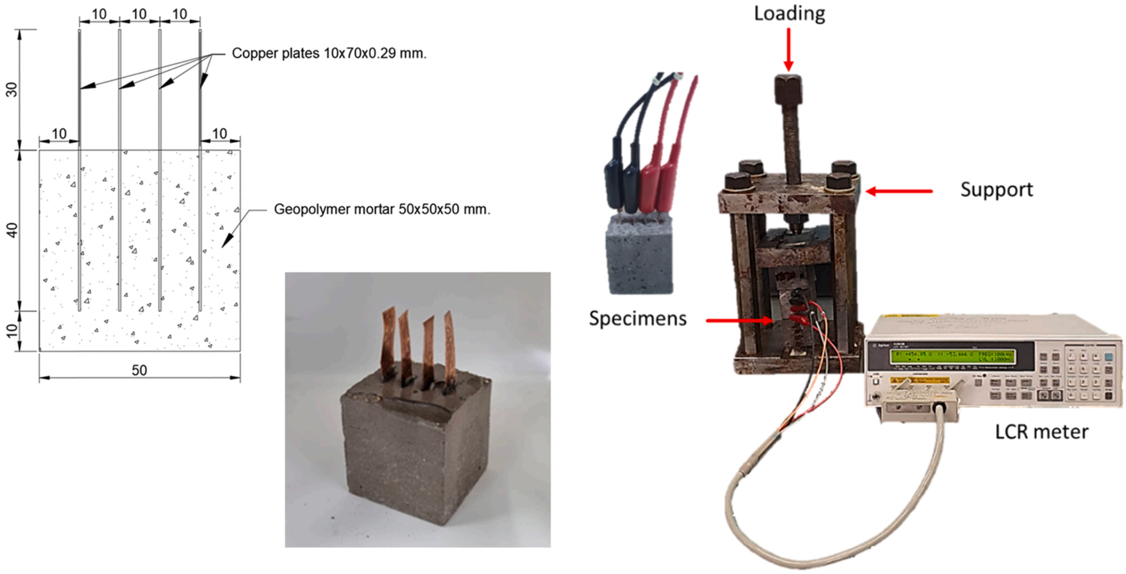


Fig. 1. Electrical resistivity measurement method.

To compare without a geometrical factor, the electrical resistance was converted to the electrical resistivity (ρ) using Eq. 1.

$$\rho = \frac{RA}{l} \tag{1}$$

where ρ is the electrical resistivity (Ω-m), R is the electrical resistance (Ω), A is the area of copper sheet which embedded in specimen (m^2) and l is the spacing between copper sheets (m).

The electrical resistivity changes (ERC) can be calculated by Eq. 2.

$$ERC \quad (\%) = \frac{\Delta\rho}{\rho} \times 100\% \tag{2}$$

where $\Delta\rho$ is the change of electrical resistivity at any applied load (Ω-m) and ρ is the initial electrical resistivity (electrical resistivity without applied load) (Ω-m).

3. Results and discussion

3.1. Physical properties

The change in physical appearance of HCFG due to the addition of MWCNTs was observed as shown in Fig. 2. The color of HCFG depended on the type of substrate material. In this study, the lignite fly ash resulted in geopolymers with gray/brown color. With the

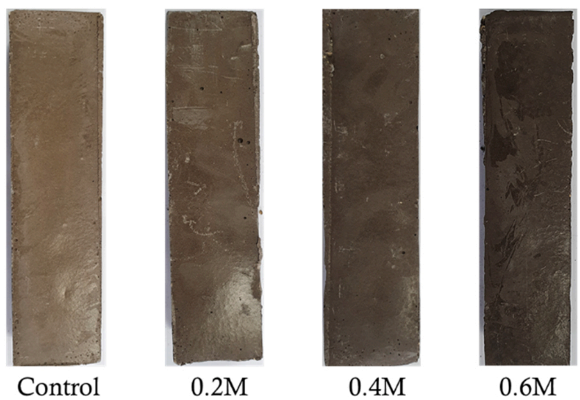


Fig. 2. Color of specimens.

addition of MWCNTs, the color of HCFG became darker and shifting toward blackish shade.

Fig. 3 shows the results on flow test. The flow of fresh HCFG decreased with the increasing of MWCNTs content. The flow of the control mix (0 M) was 111% and decreased to the lowest value of 32% at the 0.6% MWCNTs (0.6 M). This is because of the tubular structure of carbon nanotube similar to short fibers which created particle interlocking when applied in large volume [37]. In addition, the high specific surface area of MWCNTs also reduced free water content in alkali activator solution and caused the flow to decrease.

3.2. Mechanical properties

Results on compressive strength, flexural strengths, and differential strength percentage between control mix (0 M) and HCFG with MWCNTs mixes are shown in Table 4. For the control mix (0 M), the compressive strength of 25.4 MPa was observed. For HCFG mixed with MWCNTs, the strength increased noticeably with the increasing MWCNTs content up to 0.2%, and then decreased. The optimum MWCNTs content with the highest compressive strength of 34.3 MPa was observed at 0.2% (0.2 M mix). Further addition of MWCNTs beyond 0.2% caused the compressive strength to decrease gradually. With the MWCNTs ranging from 0.2% to 0.6%, the compressive strength dropped from 34.3 MPa to 32.2 MPa. Although, the strength of 0.6 M mix is lower than that of 0.2 M mix, it was still higher than the 0 M mix.

Similar to the compressive strength, the Flexural strength increased from 3.5 MPa in 0 M mix to the highest value of 4.4 MPa in 0.2 M mix. With the increase in MWCNTs beyond 0.2%, the flexural strength decreased to the lowest value of 3.8 MPa in 0.6 M mix. The increase in both strengths with the incorporation of MWCNTs up to 0.2% was caused by the effect of MWCNTs as reinforcement in the hardened geopolymer matrix and filling up of pores (filler effect) similar to those reported by Chaipanich et al., [41] and Sedaghatdoost and Berfaniah [42].

The comparison of the compressive and flexural strength of geopolymer mixed with MWCNTs between this research and previous research is shown in Figs. 4 and 5. Luz [34] and Abbasi [35] agreed with the addition of 0.1% MWCNTs in metakaolin-based geopolymer given their maximum strengths. They reported that the short fiber appearance of carbon nanotubes also acts as reinforcements in the geopolymer matrix, which increases the flexural strength and reduces crack propagation similar to those reported by Naeem [43] and Hawreen [44]. In addition, the strengths were found to be higher than in this research, which used the HCF-based geopolymer and 0.2% MWCNTs, perhaps the difference being pozzolan-based. For low calcium fly ash-based geopolymer, Rovnanik [45] and Saafi [36] found the maximum strength in 0.1% and 0.5% of MWCNTs respectively but also lower than HCF-based geopolymer in this research.

However, at high MWCNT content, a decrease in strength was observed due to the high specific surface area of MWCNTs, which increased the water requirement and caused mixing and compacting difficulties [34,36].

3.3. Elemental analysis

In Portland cement system, the strength relies largely on calcium-silicate-hydrate (C-S-H) phase. Several studies indicated that the addition of nanoparticles enhanced the hydration activity which led to an increase in C-S-H content and strength [46–48]. In the case of HCFG, the strength relies on both geopolymerization and hydration reaction. The geopolymerization provides HCFG with sodium aluminosilicate hydrate (N-A-S-H) gel while the hydration provides C-S-H gel which are the main core to the strength of geopolymer [36,39,49,50].

To observation on the chemical composition of HCFG with MWCNTs addition, the energy-dispersive X-ray spectroscopy (EDS) with scanning electron microscopy (SEM) analysis was performed to analyze the element of samples 0 M and 0.2 M. The point of EDS analysis and intensity of element were shown in Fig. 6.

The addition of MWCNTs caused the silicon (Si) atom to a slightly increase from 10.09% to 10.65%, sodium (Na) atom from 9.20% to 10.21%, and calcium (Ca) atom from 5.57% to 6.48% (Table 5). However, the few additions of MWCNTs (0.2% by fly ash weight)

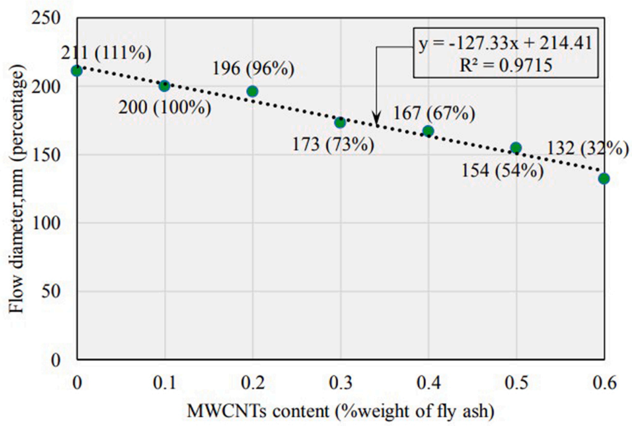


Fig. 3. Flow diameter (percentage flow) vs MWCNTs content.

Table 4
Compressive and flexural strength of HCFG with MWCNTs mixes.

Label	Compressive strength (MPa)		Flexural Strength (MPa)	
	28 days	%Control	28 days	%Control
0 M (control)	25.4 ± 1.2	100	3.5 ± 0.3	100
0.1 M	30.6 ± 1.3	120	3.8 ± 0.7	109
0.2 M	34.3 ± 0.9	135	4.4 ± 0.2	126
0.3 M	33.6 ± 1.0	132	4.2 ± 0.2	120
0.4 M	33.0 ± 0.6	130	4.0 ± 0.4	114
0.5 M	32.8 ± 0.2	129	3.9 ± 0.4	111
0.6 M	32.2 ± 0.3	127	3.8 ± 0.2	109

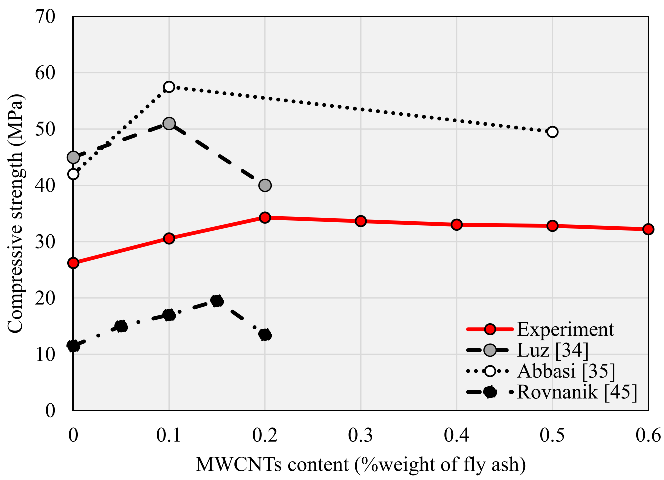


Fig. 4. Compressive strength compared with others research.

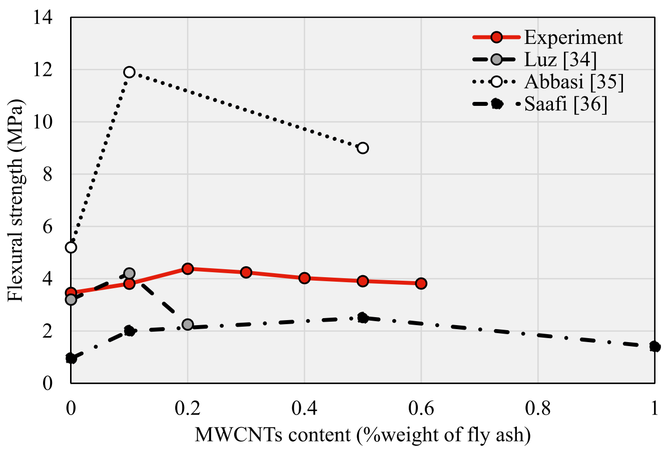
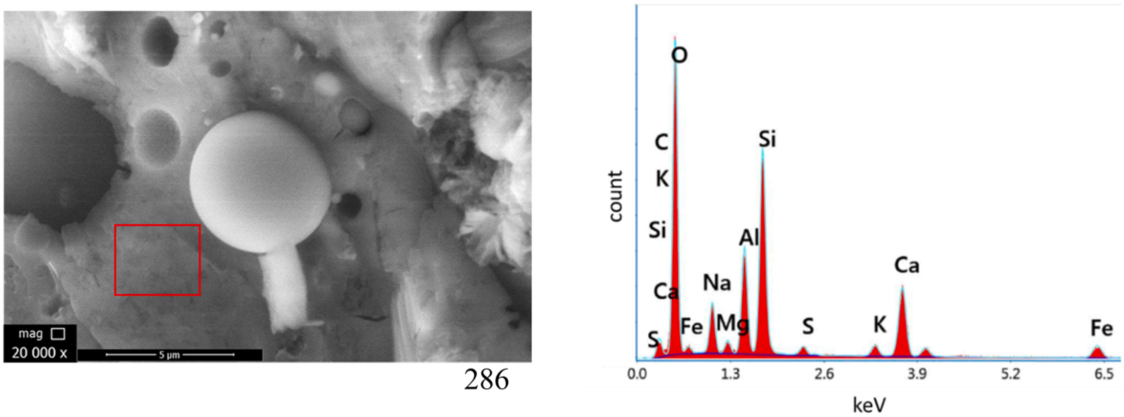


Fig. 5. Flexural strength compared with others research.

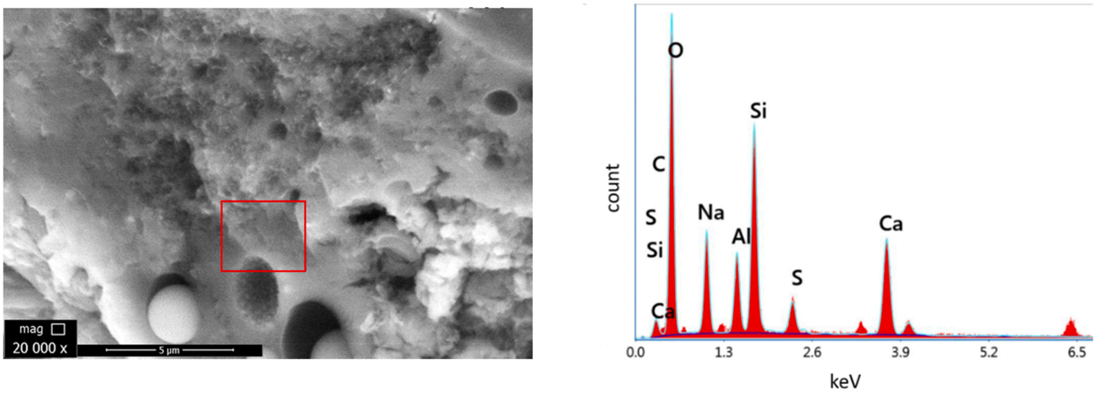
are insignificant to the geopolymerization reaction that led to strength development in HCFG. In the other hand, Jittabut [40] and He [51] reported that the high content of MWCNTs (beyond 1%) in low calcium fly ash-based geopolymer increases in both Si and Na related to the increase in geopolymerization reactions due to formed Si-O-Al products.

3.4. *Electrical resistivity*

To inspection and health monitoring of concrete structures, the measurement of stress, strain, or cracks that lead to deterioration or strength loss of the structure is necessary. The change of electrical properties under compression load can be applied to self-monitoring sensor. In this research, the electrical properties of HCFG that improve by MWCNTs were investigated.



(a) 0%MWCNTs (0M)



(b) 0.2%MWCNTs (0.2M)

Fig. 6. Elemental analysis with EDS.

Table 5
Chemical composition.

Element	Weight (%)		Atom (%)	
	0 M	0.2 M	0 M	0.2 M
C	0.95	2.78	1.60	4.58
O	52.72	51.79	66.29	64.60
Na	10.54	11.58	9.20	10.21
Al	6.69	5.20	5.00	3.91
Si	14.07	15.03	10.09	10.65
S	1.60	1.85	1.00	1.17
Ca	11.10	12.76	5.57	6.48

The results on electrical resistivity of HCFG with MWCNTs subjected to compression load are shown in Fig. 7(a)-(e). Regardless of load level, the resistivity decreased with the increases in either MWCNT content or testing frequency (Figs. 8 and 9). Carbon nanotube distributed in HCFG matrices provides path for electron flow and contributes to the improved electrical conductivity [36,38]. To evaluate piezoresistivity performance, the HCFG with MWCNTs mixes were loaded in compression up to 20% of ultimate load and the electrical resistivity were monitored. The results showed that at the load level lower than 10% of the ultimate load, the resistivity decreased with the increasing load level. But, as the load increased beyond 10%, the resistivity increased as shown Fig. 10. This could be explained as follows: under the load level less than 10%, the material response was in elastic state and the HCFG paste was compressed and remained fully intact. With this compression load, the carbon nanotubes were pressed closer to each other and became more connected. This resulted in better network for electron flow and hence, better electrical conductivity [39,41,45,52]. When the load exceeded 10% of ultimate load, although the system was in compression state, microcracks were initiated inside the specimen. These cracks discontinued the conductivity network, interrupted the electron transfer, and caused the electrical resistivity to increase.

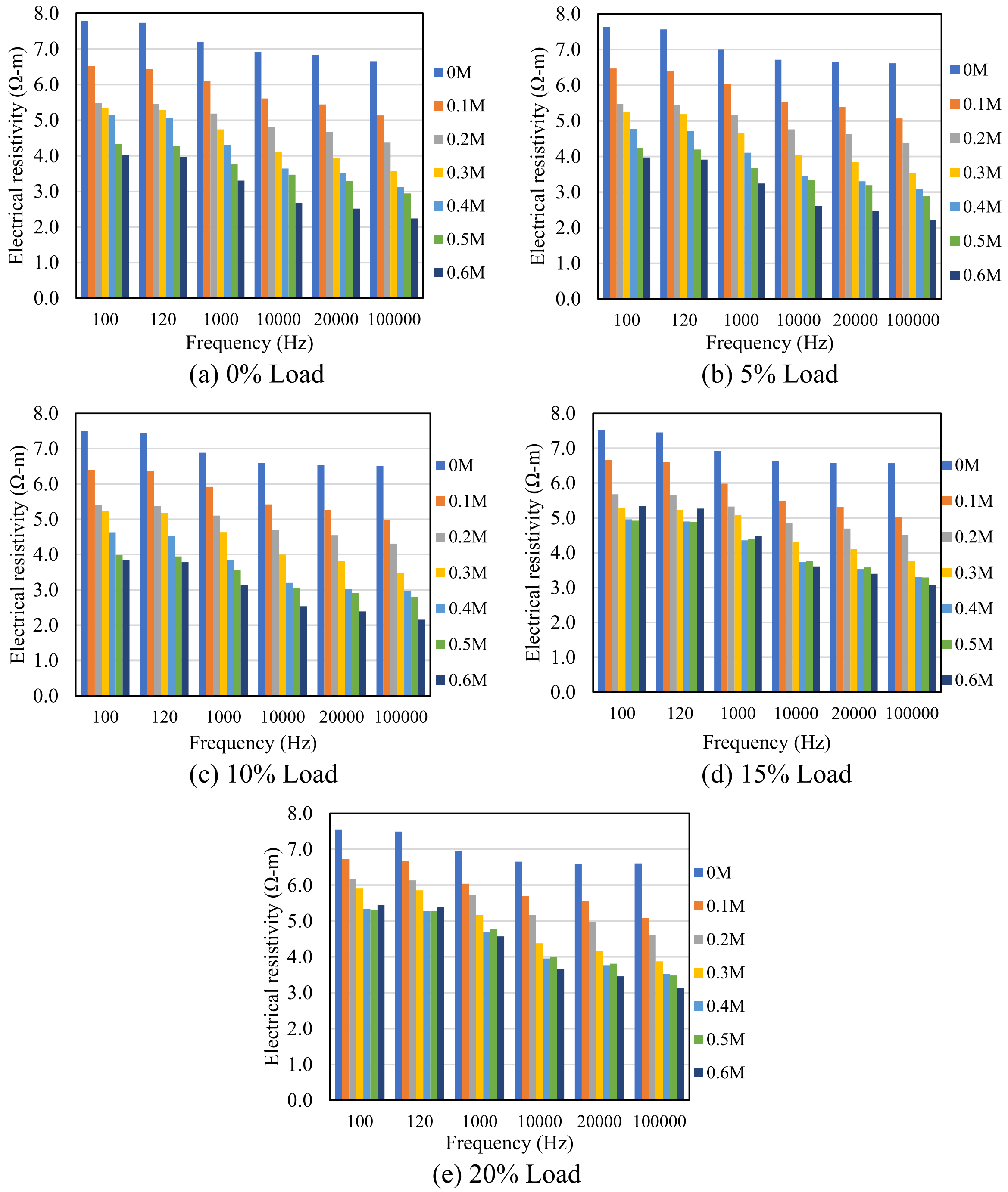


Fig. 7. Electrical resistivity.

3.5. *Piezoelectric effect*

The variation of resistivity results was also presented with the percentage of ERC as shown in Fig. 11. The loading at 5% and 10% of ultimate load lowered the ERC value. The ERC reduction was also inversely proportional to the MWCNT content. The load level around 15–20% was defined as a cracking zone in which the ERC was found to increase significantly. The variation in electrical resistivity with load is an important parameter in piezoelectric property (piezoelectricity). The results indicated that HCFG mixed with MWCNTs showed good potential for use in the concrete structural health monitoring applications of structures under loading.

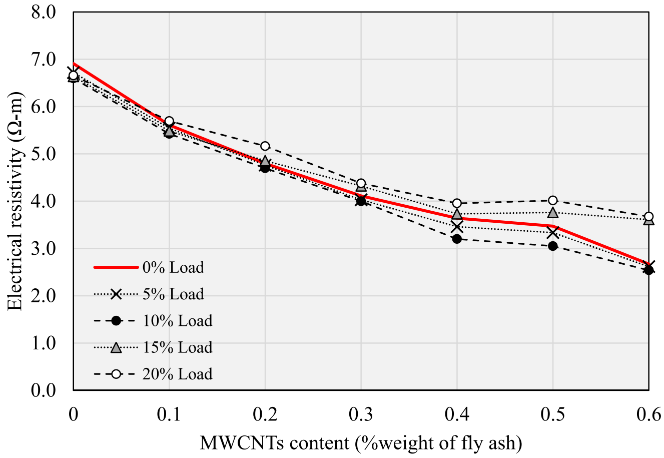


Fig. 8. Effect of MWCNTs addition on electrical resistivity at 10,000 Hz.

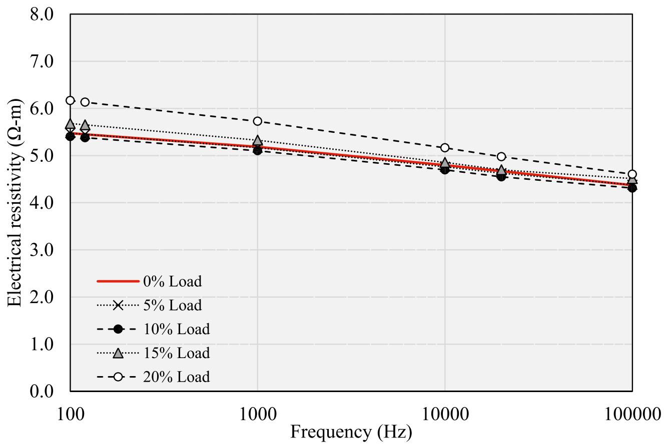


Fig. 9. Effect of frequency on electrical resistivity at 0.2 M.

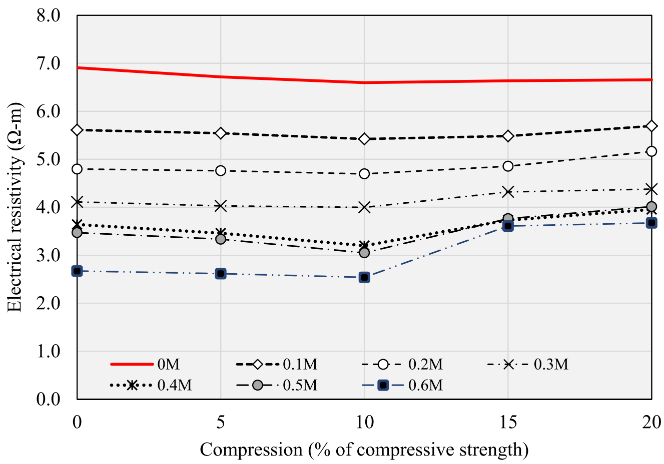


Fig. 10. Effect of compression load on electrical resistivity at 10,000 Hz.

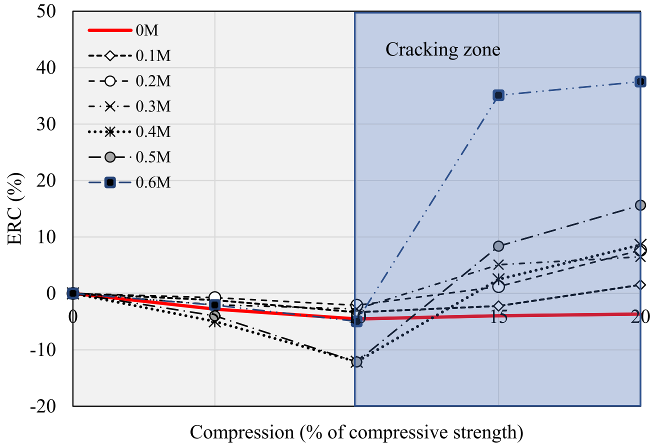


Fig. 11. ERC at 10,000 Hz.

4. Conclusions

The investigation on mechanical and electrical properties of HCFG incorporated MWCNTs can be concluded as follows:

1. MWCNTs was able to improve the mechanical properties of HCFG. The optimum dosage was 0.2% MWCNT by weight of binder for both compressive and flexural strengths.
2. The elemental analysis showed that the few additions of MWCNTs in HCFG are insignificant to element change for strength development. Therefore, in this study, the main effect of the increases in strength was result by the physical properties of MWCNTs.
3. The MWCNT improved the electrical conductivity of HCFG through the increase in electron conductivity network inside the geopolymer paste. The addition of MWCNT content allowed the electron to flow better which led to the decrease in electrical resistivity. Under the applied load less than 10% of the ultimate load, the electrical resistivity decreased even further due to the closer contact between MWCNTs. Beyond 10% load level, the resistivity increased due to the formation of microcracks inside the HCFG paste. The change in resistivity showed that the HCFG incorporated MWCNTs exhibited piezoelectricity properties and showed a good potential to be developed as a sensor in structural health monitoring application.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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