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Dielectric properties for characterization of fly ash-based geopolymer binders

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HIGHLIGHTS

- Variation of dielectric values is a good indicator of geopolymer binder's evolution.
- Effect of frequency, age, and constituents on dielectric properties is investigated.
- The relationship between dielectric properties and setting time is investigated.
- The relationship between dielectric properties and porosity is investigated.

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ABSTRACT

In this study, a method for determining the setting time and the porosity of fly ash-based geopolymer binders (FAGPBs) has been established. This method permits a real-time monitoring of the geopolymerisation and hardening progressions. A parallel plate capacitor cell technique was used to determine the dielectric constant ε_r' and dielectric loss factor ε_r'' of fresh and hardened mixes. Equivalent model-circuits of the measurement system were developed, and the accuracy of the system was verified. The dielectric properties were monitored at different ages until 56 days over a frequency range of 0.1–60 MHz. The alkaline solution to fly ash ratio, sodium silicate to alkaline solution ratio, and sodium hydroxide solution molarity were varied to study their effects on the dielectric properties. It was found that dielectric properties can provide valuable information when measured in the frequency range of 10–25 MHz. In this range, the dielectric values responded well to the variations with age and with mix properties. The relationships between the dielectric properties and the different physical states and constituent materials of FAGPBs could be used for quality control applications. The ε_r' and ε_r'' time plots were effectively utilized to determine the setting time and the rate of geopolymerisation of FAGPBs. Measurements showed a strong correlation between ε_r' and porosity; the ε_r' values decreased 2.3-fold over a porosity range of 3–15%. The proposed regression models can be further used in determining the porosity of FAGPBs at a high accuracy level.

1. Introduction

Geopolymer binders are inorganic polymers that can utilize several types of aluminosilicate wastes such as fly ash [1], bottom ash [2], ground granulated blast furnace slag [3], red mud [4], and mine wastes [5]. Recently, there has been a great interest in the development of these materials such that their application provides the opportunity to decrease CO_2 emissions and reduce the consumption of energy compared to the conventional cement and ceramic industries. Both low and high calcium fly ash have been used extensively in geopolymer binder production [6–9]. In Malaysia, most of the produced fly ashes are known for their high calcium content which is typically more than 13% by weight fractions [10]. High calcium FAGPBs have been successfully used in ordinary concrete production [11], soil stabilization [12], pervious concrete [13], coating [14], and high strength concrete [15].

Geopolymer reactions, also called geopolymerisation, involve fast chemical reactions in which aluminosilicate precursors (the

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source material) react with alkali-metal polysiliates under alkaline conditions to produce amorphous to semi-crystalline three-dimensional polymeric chain and ring frameworks. These frameworks are formed by sharing the oxygen atoms at the corners of the silica and alumina tetrahedrons. Alkali cations are required to balance the negative charge of the aluminate group. Geopolymerisation processes are mainly divided into three stages; dissolution, hydrolysis, and condensation. These stages occur simultaneously and can hardly be separated [16]. The presence of calcium oxides within the source material introduces a new reaction pathway. Calcium is activated in high alkalinity mediums and forms a new product (calcium aluminosilicate C-A-S) that does not combine (co-existed) with the geopolymer gel (sodium aluminosilicate N-A-S). Rather, it forms a separate region [12]. This reduces the porosity of the microstructure and strengthens the final geopolymeric product [17]. In the case of high alkalinity mediums (pH greater than 12) and sufficient calcium content, the precipitation of the C-A-S is dominant which due to its higher charge density. Whereas, at low alkalinity mediums, N-A-S is the main product of the reactions [18]. Calcium alters the reactions and accelerates the setting processes. Increasing the alkalinity of the medium (i.e. increasing the concentration of sodium hydroxide solution up to 18 M) is reported to increase the setting time, workability, and strength of the produced binder [6,11,19].

Even though geopolymer binders have superior properties compared to ordinary Portland cement (OPC), they experience some drawbacks that need to be addressed for their acceptance in the construction industry. For instance, some of these problems are: the need for an elevated curing temperature, cost, rate of productivity, safety issues, lake of long-term durability data, CO_2 footprint of the alkaline solution, and regulatory and institutional issues (absence of performance-based standards) [20,21]. In addition, they experience a major drawback in terms of reproducibility. Geopolymer binders' properties are largely influenced by the properties of the source material; size, quantity of reactive phases, silicon to aluminium ratio, and impurities content [21,22]. Additionally, these properties may significantly change for the same source from batch to batch [23]. Accordingly, until now there has been no standard way to predict geopolymer binders' properties based on their mixing proportions. On the other hand, using conventional testing methods is costly and time-consuming. Real-time monitoring systems and optimization of binder components are necessary to produce consistent and high-quality geopolymer binders.

Non-destructive testing (NDT) methods are becoming more valuable in the construction industry. They can be used to evaluate the existing structures and as a quality control technique during the manufacturing processes. Among the various NDT methods available for the evaluation of concrete, the electromagnetic methods are more favourable due to their non-invasive nature, low cost, rapidity, applicability to large-scale structures, and real-time measurement. Electromagnetic methods have several successful applications in the characterization of concrete properties [24,25], durability evaluation [26,27], and determination of construction features and quality control applications [27–29]. The electromagnetic responses of materials subjected to an electrical field are ascertained from the estimation of their dielectric properties. Each material has its unique dielectric properties, and in composite materials, these properties become a function of the dielectric properties of the constituents. Complex relative permittivity (ε_r), is usually used to identify the dielectric properties of the concrete in a quantitative and a qualitative way [30,31]. The ε_r consists of two parts; the real part and the imaginary part. The real part is known as the dielectric constant (ε_r'), and it represents the amount of energy stored inside the medium. The imaginary part is known as the dielectric loss factor (ε_r''), and it indicates the dissipation (or loss) of energy within the medium.

Since geopolymer binders are aluminosilicate materials, they are expected to have high electrical insulating values. However, the presence of alkali cations and water inside porosities within the geopolymer matrix tends to increase their electrical conductivity [32]. Recently, a vast number of studies have been carried out on geopolymer binders [6,11,19–21]; however, most of these studies focused on source material characterization and mechanical and rheological properties. A limited number of studies have been conducted on the dielectric characterization of geopolymer binders as a construction material. To the best knowledge of the authors, the only studies carried out are the ones done by [19,23,33–36]. The results of these studies demonstrate that dielectric properties can be effectively used in monitoring the progress of geopolymerisation. However, these studies have investigated the dielectric properties at low-frequency levels (up to 1 MHz) and on fresh specimens. The other available studies have investigated the dielectric properties of geopolymer binders as electrical insulators and packaging materials [37–39]. The conversion of the dielectric values into geopolymer binder properties, such as compressive strength and porosity, has been rarely investigated.

The work presented here is a part of the ongoing research on the use of electromagnetic properties as a method for geopolymer concrete evaluation. In this paper, a parallel plate capacitor cell (PPCC) setup has been designed, calibrated, and used to measure the dielectric properties of different FAGPB specimens in the frequency range of 0.1–60 MHz. The dielectric properties were monitored continuously to assess the microstructure and strength development processes. The PPCC setup was used due to its ability to quantitatively measure the dielectric properties and its flexibility towards different specimen sizes. Technically, the PPCC setup may not be considered as an NDT method since it is usually used for laboratory measurements. However, the PPCC technique may be useful for quality control applications especially in the case of geopolymer concrete where its production is still limited to precast and ready-mix concreting. Nevertheless, PPCC can provide high accuracy of measurement of dielectric properties [40]. It would be highly advantageous to have such an accurate measurement at a wide range of frequencies, and this would help to determine the optimum frequency for different applications [41]. Understanding of dielectric properties of geopolymer binders and their response to the binders' properties is essential to enhance the quantitative interpretation capabilities. This would pave the way for further research including other possible electromagnetic methods for NDT of geopolymer concrete such as ground penetrating radar, eddy current, capacitive probe, and resonating waves. Furthermore, the results obtained using the PPCC setup can be coupled with other on-site electromagnetic techniques and then used as an NDT technique for the evaluation of concrete [31]. The results of this study show that dielectric properties can be used to monitor the geopolymerisation processes, and they can provide a substantial insight into the geopolymer binders' microstructure.

2. Materials and methods

2.1. Materials

The fly ash (specific gravity = 2.11) used in the geopolymer binder synthesis was obtained from Manjung power plant at Perak, Malaysia. The chemical composition of the fly ash is given in Table 1. This fly ash can be characterized as a class F fly ash according to ASTM C618 [42] as the sum of the silicate, ferrate, and aluminate oxides are greater than 70%, and the SO_3 and the loss of ignition (L.O.I.) percentages are less than 5%, and 6%, respectively. However, it has a high calcium content.