Synthesis and characterization of fly ash based geopolymers in aggressive solutions

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The objective of this research is to improve the durability of geopolymer in harsh environmental conditions using a low concentration of alkaline activator by concentrating on fly ash and clay-based composites. Because of their low carbon footprints and effective waste management, geopolymer present a sustainable solution to the environmental issues associated with the manufacture of cement. The study investigates the durability of samples synthesized with fly ash, silica, aluminum hydroxide, clay, and a 5 molar potassium hydroxide solution in aggressive solutions (NaCl, K_2SO_4, H_2SO_4). Investigations using FT-IR, XRD, XRF, SEM, PL spectroscopy, and dielectric characteristics show major changes in the characteristics of geopolymer under adverse circumstances. The results provide useful information for enhancing the properties of geopolymer, offering an environmentally friendly alternative to building materials. The foundation for further investigation of geopolymer in building projects, particularly in different aggressive environmental conditions, is provided by this study.

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1. Introduction

The production of cement has long been connected with various environmental problems, like high carbon dioxide emissions. It is estimated that each 5 kg of Portland cement produces almost 5 kg of CO_2, which is very alarming to the serious environmental problem, global warming. It has been observed that the production of cement will be increase by 100% in near future as per demand of rapidly construction, and will increase environmental problems. Carbon dioxide, which is an important greenhouse gas, is acknowledged as the main cause of global warming as well greenhouse effect [1-4]. Moreover, beyond the emission of CO_2, some other environmental problems are concerned with production of cement like excessive use of energy, air pollution, water pollution and degradation of land. The burning of fossil fuel excessively and chemical breakdown of limestone as well production of waste product like kiln dust, slag and flyash, a result of coal fired power plants, contribute to these environmental problems [5-7].

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As a result of these environmental problems, it has been emphasizing to create and alternative cementitious material which can fulfill demand of building construction but reduce the emission of CO_2 and be environmentally friendly. Geopolymers, made up of inorganic, ceramic alumino-silicate structures that create long-range, covalently bound, amorphous networks, are gaining acceptance as an alternative of Portland cement worldwide. Geopolymers is highly acceptable due to many unique qualities like low emission of CO_2, low usage of energy, consuming of cheap raw materials, act as a binder and waste management. Beyond this some outstanding qualities include durability to high temperature as well aggressive chemical environment, high compressive strength and adhesive nature, making them a best alternative to Portland cement [6-12]. Geopolymer concrete, which is made up of industrial waste like silica (SiO_2) and alumina (AlO_3), uses flyash and grounded granulated slages instead of fossil fuels, which reduce the consumption of energy and emission of CO_2. This option, geopolymer, has the superior mechanical qualities such as durability, and a longer lifetime [13, 14].

The precursors obtained from industrial waste like rice husk ash or fly ash, are used in the process of polymerization while making geopolymers. The activators, which are often used are NaOH and KOH solutions, strengthen bonding in geopolymer concrete, high the concentration of alkaline activator, stronger will be the properties of geopolymer. But researchers are trying to use low concentration of KOH to observe whether its durability against aggressive environment increases. Despite all these advantages of geopolymer concrete, it still not has gained as much adoption as an alternative to Portland cement in construction [15-17]. In this study, a low concentration of alkaline activator is tried to produce a good quality geopolymer concrete and investigate its durability in several aggressive solutions such as sulfate, acid and chloride. The objectives are to provide useful insights into the performance of material under harsh conditions, giving critical information for further field research. Fly ash, which is high in amorphous alumina and silica, is essential in the synthesis of geopolymer, which increases the chemical reactivity and improving durability as well compressive strength. As the construction industry looks for more sustainable options, geopolymer concrete appears as a possible answer, addressing environmental problems and supporting a more eco-friendly approach to building materials. The main aim of this study is to examine and assess the increased resistance of geopolymers to harsh environments, especially in the presence of sulfate, chloride, and acid exposures. The main goals of this study are:

i. Combining clay and fly ash, cheap raw materials, to create geopolymers.

ii. Utilizing a low concentration (5 molar) of potassium hydroxide as an alkaline activator.

iii. Describing the mechanical properties of geopolymers, such as their compressive and flexural strengths, in both a normal and aggressive environment.

iv. Performing various testing and analysis to determine the performance of geopolymers like durability when exposed to solutions of sulfate, chloride, and acid.

v. Comparing the performance of geopolymer with ordinary Portland cement when expose to harsh environment.

vi. Utilizing cutting-edge analytical methods to examine the microstructural alterations and chemical reactions that occur in geopolymers when exposed to hostile substances.

vii. Providing a useful advice and guidance for the efficient use of geopolymers in building projects with hostile surroundings.

2. Experimental method

The materials utilized in the present research work was fly ash (0.49 gram), silica (1.81 gram), aluminum hydroxide (1.1 gram), clay (1.5 gram), and potassium hydroxide solution (5mol). The aggressive solutions applied were sodium chloride (NaCl) solution (2%), potassium sulfate (K_2SO_4) solution (2%), and sulfuric acid (H_2SO_4) solution (0.2mol). The geopolymer samples are prepared by the combination of raw starting powders for 10 to 20 minutes to achieve a homogenous combination. The dry mixture formed was then mixed by adding potassium hydroxide solution to form a paste-like substance. To make a uniform dispersion of the alkaline activator throughout the paste, vigorous mixing was used. Four identical cubes were then cast from the slurry, providing four geopolymer samples. The geopolymer samples were cured in three stages. For the initial curing stage, the samples were heated in an oven to 70°C for 4-5 hours. After the initial curing phase, the cubes were removed from the oven and allowed to cool to room temperature. They were then put to the oven for another 24 hours at the same temperature (70°C). Following secondary curing, the samples were transferred to a high-energy furnace and annealed at 850°C for two hours. The geopolymer samples were then subjected to the aggressive solutions. The samples were immersed in their respective aggressive solutions for 18 days. After the exposure period, the samples were removed from the liquids and dried at 100°C in order to remove any remaining moisture. After this the dried samples were packed and sent for further analysis to the University of Peshawar (Pakistan). The XRD analysis was used to examine probable mineralogical transformations and changes in crystalline phases, while EDX analysis is used to identify the chemical compositions of the geopolymer samples and identify any differences in elemental composition. The SEM was used to study the microstructural changes of the geopolymers cubes after insertion to the aggressive solutions. The FTIR analysis was used to examine any chemical changes in the structure of geopolymer. Photoluminescence (PL) spectroscopy is used to studies the optical properties of the geopolymer cubes while Ultraviolet-visible (UV) spectroscopy was used to investigate the changes in the electronic structure of the geopolymer cubes after exposure to aggressive environment. The microwave dielectric properties are measured by using impedance analyzer.

3. Results and discussion

3.1. Fourier transform infrared (FT-IR) spectroscopy

The FT-IR analysis was used to examine any chemical changes in the structure of geopolymer. The FT-IR spectra of geopolymers, exposed to aggressive environment of NaCl, K_2SO_4 and H_2SO_4 and that of un-dipped or non-exposed to aggressive solution are shown in figure 1, in the geopolymer, before exposure, vibration near 700 cm^{-1} is assigned to bending vibrations of Si-O-Si or Al-O-Al bonds, shift in the samples exposed to harsh solutions are assigned to have changes in the bonding environment or the formation of new phase [18-20]. The next small peak at 780 cm^{-1} in un-dipped sample while that of at 770 cm^{-1} in other samples are assigned to the stretching vibrations of Si-O or Al-O bonds [21]. The strongest and intense band at 1020-1060 cm^{-1} in the un-dipped sample and at 1000 cm^{-1} in other samples are reported the asymmetric mode of vibrations of Si-O-Si bond while shifting peak could indicated the changes in the polymerization degree of geopolymer network [22, 23]. Vibration at 1350-1400 cm^{-1} assigned the bending vibration of O-Si-O or O-Al-O bonds, here difference between un-dipped and other samples assigned to have structural changes [24].

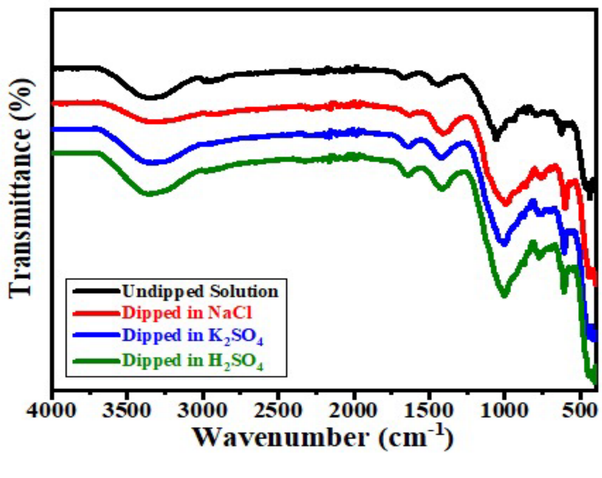


Fig. 1. FTIR spectra of fly ash geo-polymer samples.

The small peak appears at 1570 cm^{-1} assigned the bending vibrations of hydroxyl groups (-OH) associated with geo-polymer network [25]. The thickening of the spectra at 1900-2000 cm^{-1} assigned the presence of water or other volatile compound in samples [26]. Largest vibration appears at 3500.0 to 3600.0 cm^{-1} assigned the stretching mode of –OH group associated with geopolymer network and the small peak in only un-dipped sample assigned the stretching mode of C-H bonds which indicate the existence of organic compound [27, 28]. The result of FT-IR spectra shows that the exposure of geopolymer samples to solution leads changes in their structure and composition, which could affect their properties as well performance [29, 30].

3.2. XRD analysis

Figure 2 shows the XRD pattern of the fly ash samples dipped in different aggressive solution. The analysis was used to examine probable mineralogical transformations and changes in crystalline phases [31]. The longest peak which appears at 27° indicates the presence of quartz, which suggests the major crystalline phase present in all samples [32]. As, this peak is common in all samples, which shows that the geo-polymerization process resulted the formation of a consistent crystalline phase across all samples [33]. Another intense peak at 43° indicates the presence of silica or alumina, which is often, gives indication of another crystalline phase, both these intense peaks suggest the primary crystalline phases of the samples [34].

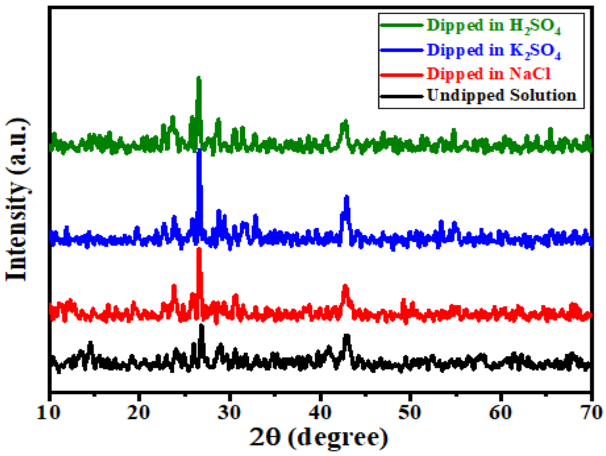
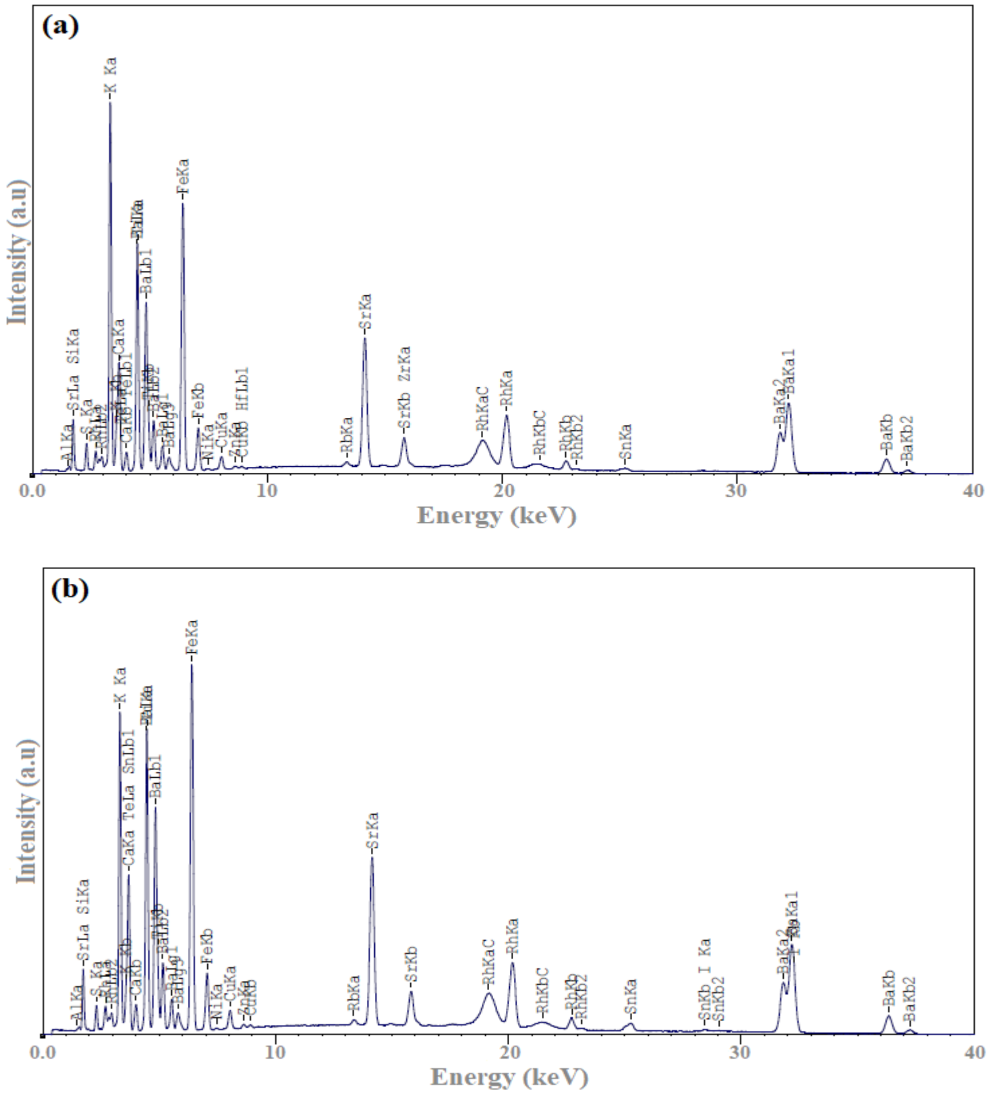


Fig. 2. XRD pattern of the fly ash geo-polymer samples.

The short peaks at 28-32° in NaCl dipped and un-dipped samples, and at 28-35° in the K2SO4 dipped and H2SO4 dipped samples suggests the variations in the crystallographic orientations [35]. The downward peak in NaCl dipped samples at 67° shows the decrease in crystalline phase of the sample, possibly due to dissolution of some components in this sample [36]. Differences of the peaks (28-32°, 23°, 50°, 54°, 67°) show crystallographic configurations that may have been altered by exposure to various aggressive solutions [37]. The EDX technique is used to finds the investigated composition of the products. Figure 3 presents the XRF findings. The primary components of fly ash include Fe, K, Na, Mg, Ti, P, silica, calcium, aluminum, and iron. The ultrafine reactive flyash (R) have greater ca concentration, while conventional flyash (FA) have higher Al concentration. The flyash obtained from municipal waste products are incinerators (B) were analyzed to determine its chemical composition. The results indicated that the principal ingredients in the ash were Ca, cl, O, S, K, Na, and Zn elements. As per ASTM - C618 – 2 database, fly ash produced by the burning process is categorized as class F, while fly ash from municipal trash incinerators is classed as class C based on oxygen composition. Remember that the base material's oxygen content affects how well geopolymer linkages form. Too much calcium could speed up the materials' reaction and prevent them from forming the three-dimensional structure that characterizes geopolymers [38]. The oxide composition of the materials examined is displayed in Figure 3. The largest concentration of silicon and aluminum oxides, which are essential for the polymerization process, was found in fly ash (F). Furthermore, the composition or contents are hypothetically very dangerous elements like Y, Ce, and Sr in all of these flyashes are rather low and not exceeded the limits for hazards wastes products. This makes it permissible from a legal standpoint to use these flyashes are uses as a raw starting material for construction related items [38, 39]. High calcium fly ash has been demonstrated in earlier studies to be promising material sources for high-quality geopolymers products [40, 41]. The high strength Ca concentrations are uses to form bonds inside the geopolymer network, where they function as cations that balance electricity [42].



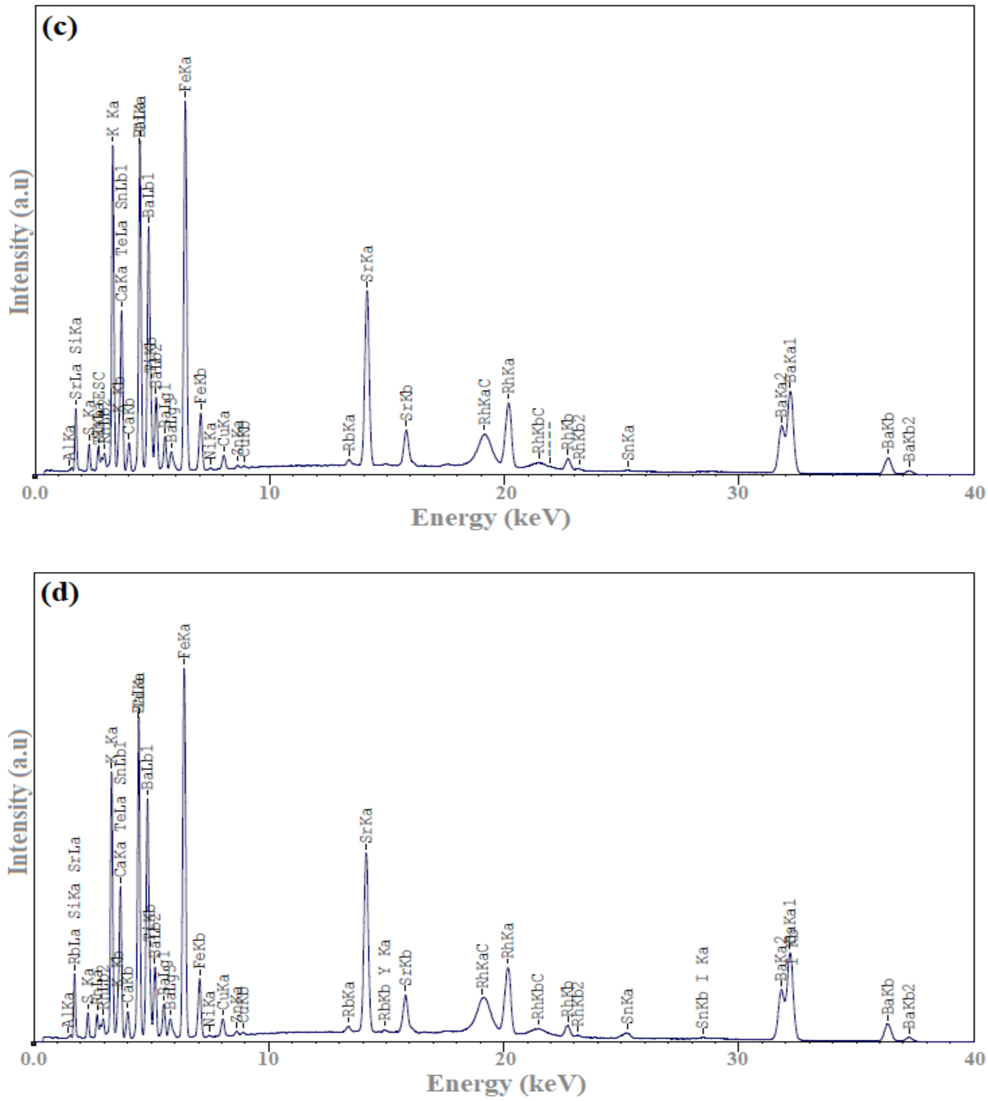


Fig. 3. XRF pattern of fly ash geo-polymer samples, (a) Undipped solution, (b) Dipped in NaCl, (c) Dipped in K2SO4, and (d) Dipped in H2SO4.

3.3. SEM analysis

SEM micrographs are used to study the microstructural changes of the geopolymer samples after exposure to the aggressive solutions along with the particle size distribution curve as shown in the Figure 4. The fly ash was a pebbles like shape particles with a smooth surface. The molecule estimate was around 2–20 µm, with the normal molecule estimate of 2.23 µm. The workability of the geopolymer composite as a work of concentration is displayed within the test. The essential item appeared as low porosity and small size crystallites which is due to surface micro strain and inhomogeneity. All things considered, the aggressive solution agents increment driven to the products of unused grain and increasing in porosity within the sample. By using ImageJ software, the estimated fly ash waste products materials are encourage examining, and evaluated discoveries are gotten for all test. These components are expected to influence the physical properties of the base sample i.e. structural, optical, dielectric, and basic characteristics, emphasizing the prerequisite for improvement in these zones [43].

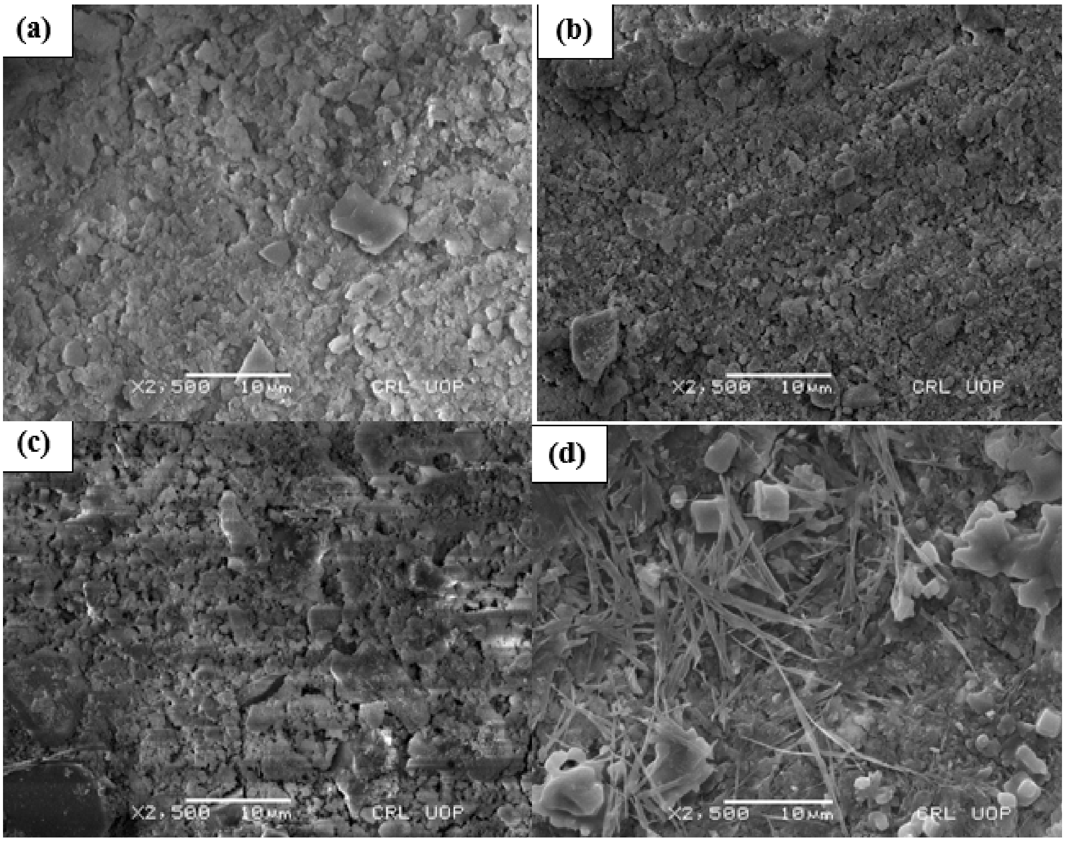


Fig. 4. SEM images of fly ash geo-polymer samples, (a) Undipped solution, (b) Dipped in NaCl, (c) Dipped in K2SO4, and (d) Dipped in H2SO4.

3.4. PL analysis

Photoluminescence (PL) spectroscopy is used to studies the optical properties of the geopolymer sample and uniformity of the geopolymer samples as shown in the Figure 5. In the given figure, all the four samples have exact same λmax 684 nm, which suggests a consistent photo-luminescent behavior across all the samples, and could be attributed to the presence of to certain elements or compounds in all samples that emits light at this wavelength when get excited [44].

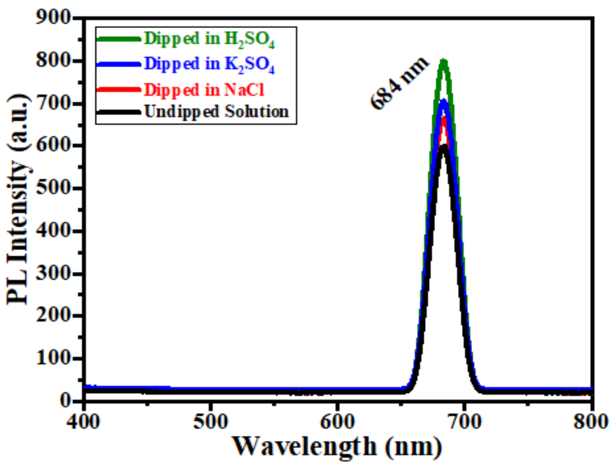


Fig. 5. The PL spectra of all samples, (a) Undipped solution, (b) Dipped in NaCl, (c) Dipped in K2SO4, and (d) Dipped in H2SO4.

This also shows that the geopolymer materials are homogenous having same optical properties. The absence of other peaks suggests the absence of other significant light-emitting species. The homogeneity of all samples shows that there is no effect of photo-luminescent properties by exposure to different aggressive environment like NaCl, H_2SO_4 and K_2SO_2 [45].

3.4. Dielectric properties

Figure 6 shows the variation of the relative permitivity with varying frequency of fly ash dipped in different aggressive solution samples. Big dielectric constant dispersions may influence the existence of many polarization types, such as dipolar, interfacial, ionic electronic and spce charge polarizations [46]. The image feature are shows the effects of blocking as well as the dispersion of the material. The main sources of this dispersion are polariz composition of the samples under investigation with charge carrier [47]. The dielectric constant decreases with frequency because of the permanent dipole moments, which may affect the overall polarization mechanism of the materials (see Fig. 6) while the Electrical polarization is caused by the introduction of a local field because the material's electrons slightly move in the dipole moment the direction opposite to the field. Strong dipole moments that orient it in the direction of the field cause the field to fluctuate at high frequencies. The dipole moment, which is never constant inside the material for an extended length of time, is the main cause of the–frequency variation of the dielectric constant. Figure 7 shows the variation of tangent loss with varying frequency of the geopolymer samples are due to the different kinds of polarization mechanism. Tangent loss slightly decreases with increasing frequency; this low tangent loss geopolymer fly ash is suitable for the applications wireless communication system. Our research thus indicates that the value of the dielectric constant and tangent loss decreases with frequency.

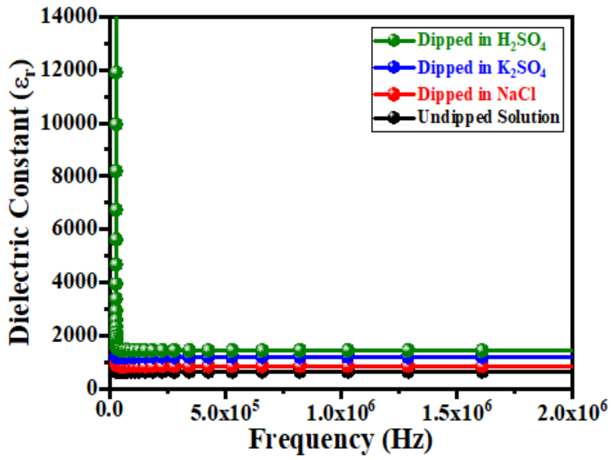


Fig. 6. The variation of dielectric constant with frequency of the fly ash geopolymer.

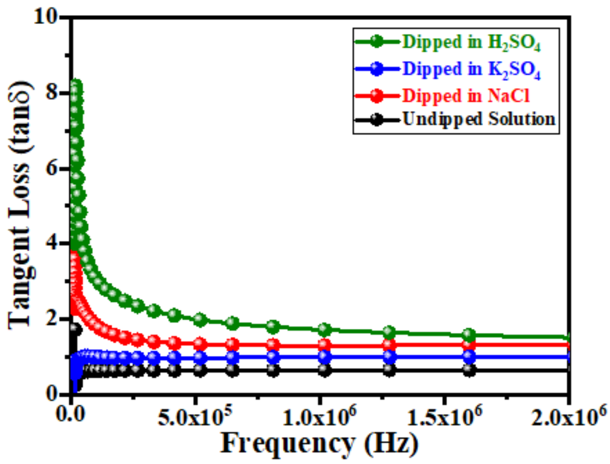


Fig. 7. The variation of tangent loss with frequency of the fly ash geopolymer.

4. Conclusion

The results of this study make significant contributions to our knowledge of how to optimize the properties of geopolymer to increase their durability in harsh environments. It has been investigated whether fly ash and clay can be combined with potassium hydroxide at a low concentration to function as an alkaline activator and produce geopolymer with better mechanical and durability qualities. A thorough examination of geopolymer samples subjected to aggressive solutions demonstrates significant changes in their microstructure, mineralogical makeup, and chemical structure. The study addresses environmental issues and promotes eco-friendly building materials by confirming the potential of geopolymer as a sustainable substitute for conventional route; SEM photoluminescence (PL) spectroscopy, X-ray fluorescence, diffraction, dielectric property studies, and FTIR, all add to enhancing our comprehension of how geopolymer behave under harsh conditions. These results provide useful information for the effective application of geopolymer in building projects, especially those that challenge challenging environmental conditions. As a result, the production of durable and sustainable building materials becomes easier by this research, which provides the groundwork for future investigation and application of geopolymer in the building sector.

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Conflict of interest

The authors declare no conflicting of interest.

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