

DOI: <https://doi.org/10.24425/amm.2023.145477>W.M.W. IBRAHIM^{1,2*}, M. IBRAHIM^{2,3}, M.Z.A. AZIS¹, M.M. AL B. ABDULLAH^{2,3},
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INVESTIGATION OF MECHANICAL, PHYSICAL AND DURABILITY PROPERTIES OF METAKAOLIN-BASED GEOPOLYMER

Due to their potential to lower CO₂ emissions linked with the cement and concrete industries, geopolymer binders are a desirable alternative for Portland cement binders. However, if they are to become a viable alternative to conventional Portland cement materials, their resilience in harsh conditions has to be further investigated. This paper presented mechanical and short-term durability properties of metakaolin based geopolymer concrete at sulphuric acid (H₂SO₄) solutions exposed with the concentrations of 2%, 3%, 4% and 5% for 14 days. (0%) or unexposed sample also prepared as referral and comparison. The geopolymer concretes were synthesized using an alkali activation of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). The main objective of the study was to examine the durability and deterioration mechanism parameters like different acid percentages, changes in weight, compressive strength, density and water absorption. Morphology analysis also performed in this study. The results indicated that metakaolin geopolymer experienced some strength deterioration with increasing sulphuric acid concentration solutions which are from 32.58 MPa, 20.67 MPa and 4.25 MPa at unexposed (0%), 2% and 5% sulphuric acid immersion respectively. Furthermore, change in weight or mass loss and water absorption after the chemical attack resulted directly proportional to sulphuric acid concentration due to increment of crack on the sample. Among that, the metakaolin geopolymer submerged in 2% acid gives the optimum results in terms of durability, mechanical and physical qualities.

Keywords: Metakaolin-based geopolymer; durability; sulphuric acid; chemical attack; mechanical and physical properties

1. Introduction

As the usage of cement in concrete has expanded, environmental concerns have arisen over the exploitation of raw materials (limestone and clay) and the release of carbon dioxide during cement production. This has put pressure on the construction industry to limit its cement use. There are several of initiatives to minimise the amount of cement used in concrete in an attempt to reduce cement consumption. Among them include the use of metakaolin, fly ash, blast furnace slag, silica fume, natural pozzolans, and biomass ash [1,2]. In this regard, scientists are studying geopolymer or alkali-activated materials. Strong alkaline activating solutions like sodium silicate, sodium hydroxide, and a mixture of the two are used to make geopolymer materials as strong as OPC concrete [3-6,1-4].

Recent studies into geopolymer concrete have been prompted by the material's enhanced mechanical and chemical strength and

durability, as well as it is potential to build a strong bond between steel reinforcements and the components of concrete [7]. The advantages of geopolymer concrete over traditional Portland cement (OPC) concrete make it a viable alternative in certain cases [7-10]. In addition to being environmentally friendly, geopolymers have several great qualities, including high early strength, low creep, low shrinkage, and strong resistance to acid and sulphate attack [6,8-10]. The geopolymer concrete is most likely to be employed in fiber-reinforced composites, fire-resistant coatings, and chemical industry waste immobilisation solutions [11,12].

Recent research is primarily concerned with geopolymer concrete production techniques and the effect of manufacturing restrictions on the physical and mechanical characteristics of geopolymer concrete [7,13]. A limited study on the endurance of geopolymer concretes has been undertaken, and the findings show that geopolymer concretes outperform OPC concrete in a variety of acidic environments. The strength of the OPC

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is good under adverse circumstances. However, it has limited resistance to acid attack, sulphate attack, and high temperatures [14,15]. The acid attack severely damages OPC due to the deterioration of $\text{Ca}(\text{OH})_2$ and the decomposition of hydrated silicate and aluminium phases. This reaction may cause strength loss, expansion, cracking, and scaling of the surface layers in mortar. However, the geopolymer mortars demonstrated a unique mechanism. The primary geopolymerization product was unique from Portland cement hydration products and was more resistant to sulphate attack than those products [13,16,17].

Due to a lack of standards in design rules and information, especially regarding durability, the use of geopolymer concrete in structural designs is presently restricted. It is the goal of this paper to look into the mechanical and durability properties of geopolymer concretes to figure out if geopolymer concretes can be used in structural design parameters and which concrete type is best when concretes are exposed to different amounts of acid.

2. Experimental

2.1. Materials

In this experimental study, metakaolin is employed as the raw material to generate geopolymer paste as the binder, to build concrete. Metakaolin used in this research was from Malaysia's Associated Kaolin Industries Sdn. Bhd. Metakaolin with chemical composition of SiO_2 , Al_2O_3 , Na_2O , K_2O and Fe_2O_3 at 56.84%, 35.60%, 2.4%, 1.04% and 1.31%, respectively were used in this experimental work. X-ray fluorescence (XRF) was used to examine their chemical compositions. The morphology of metakaolin was described as flaky and platy by SEM at magnifications of 1000 \times , as depicted in Fig. 1. Metakaolin particles were formed when larger flakes were generated and separated from one another. The diluting effect of alumina in metakaolin resulted in a smooth surface. Alumina binds metakaolin flakes and functions as a stacking agent before they are calcined. Tiny patches or small islands of flakes were detached and polished off the surface of metakaolin due to calcination [18].

The fabrication of geopolymer concrete is carried out using conventional concrete technology processes. Metakaolin has a silica alumina glassy chain that is exceptionally strong [19]. The strong chain must be broken to improve the reaction process. Hence, alkali activators are utilised to improve the reaction process. The sodium hydroxide used was Formosoda-P sodium hydroxide (NaOH) with a 99 percent purity from Taiwan's Formosa Plastic Corporation. Pallet NaOH was dissolved in distilled water to get the 10 M NaOH. The NaOH solution was stored at room temperature for 24 hour. The hydroxide anion (OH^-) produced by sodium hydroxide acts as a catalyst for dissolving aluminosilicates during the initial stage of geopolymerization, which leads to the creation of monomers. It was discovered that NaOH had a considerable impact on both the compressive strength and structure of geopolymers. The sodium silicate utilised in this study was obtained from South Pacific Chemi-

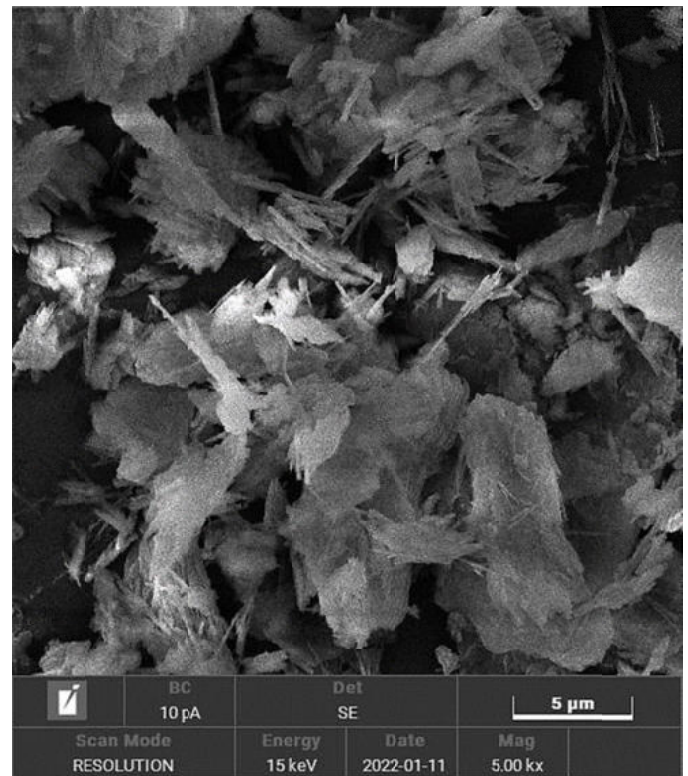


Fig. 1. SEM micrograph of metakaolin

cal Industries Sdn. Bhd. (SPCI), Malaysia, and had a chemical composition of 30.1% SiO_2 , 9.4% Na_2O , and 60.5% H_2O , according to the manufacturer.

2.2. Preparation of metakaolin geopolymer

As a precursor, high reactivity metakaolin was used in every geopolymer concrete. The sodium silicate to sodium hydroxide ratio and the solid to liquid (metakaolin and alkaline activator) ratio were set at 1.00 and 0.8, respectively. The mixture was stirred for 10 minutes after the alkaline activator was introduced, or until it was completely integrated. The mould (50 mm \times 50 mm \times 50 mm) was greased before the geopolymer slurry was put in to prevent the samples from sticking to the mould during removal. The metakaolin geopolymer was then cured for 24 hours at 60 degrees Celsius. At this curing temperature, the hardening process is sped up and the physical characteristics of the geopolymer samples are improved as stated by Mo et al. [20]. The specimens were taken out and dried at room temperature before being analysed in the laboratory for 7 days.

2.3. Testing and analysis

In this research, measures relating to permeability and chemical assault were used to assess the durability of geopolymer concrete. Water absorption measures were used to determine permeability, and acid attack was used to investigate the chemical assault. This test was carried out according to ASTM

C 642-21 at 7 days to determine the relative porosity or permeability properties of the concretes [21]. For this test, 50 mm × 50 mm × 50 mm cubes were employed as the specimens. Eq. (1) was used to compute the percentage absorption of a substance.

$$\begin{aligned} \text{Water absorption (\%)} &= \\ &= \frac{\text{Saturated weight, } W_s - \text{Dry weight, } W_d}{\text{Dry weight, } W_d} \times 100\% \quad (1) \end{aligned}$$

To conduct acid attack investigations, immersion methods were used in this study based on the general standards in ASTM C267 [22]. Three samples of 50 mm × 50 mm × 50 mm cube specimens from each mix were submerged in sulphuric acid of 2%, 3%, 4% and 5% after 7 days of curing. In the current investigation, the acid electrolyte was prepared using H₂SO₄ of the Analytical Reagent grade following Eq. (2). For comparison, unexposed H₂SO₄ also were studied on their physical, mechanical and durability properties.

$$C_1V_1 = C_2V_2 \quad (2)$$

Where

- C_1 – Initial concentration of sulphuric acid (98%),
- V_1 – Volume to add in volumetric flask, x ml,
- C_2 – Final concentration, 5% (want to prepare),
- V_2 – Volume to prepare, 1000 ml.

Using formula:

$$\begin{aligned} C_1V_1 &= C_2V_2 \\ (98\%) (x \text{ ml}) &= (5\%) (1000 \text{ ml}) \\ x \text{ ml} &= 51.02 \text{ ml} \end{aligned}$$

As a result, 51.02 ml of concentrated H₂SO₄ were added to 948.98 ml distilled water.

To ensure consistency, the solution was maintained at room temperature and stirred constantly, at least twice a day. The pH of the sulfuric acid solution was 1.0 and the acid solution to sample volume ratio was regulated at 3.0. To maintain the concentration of the solution during the test time, the solution was replenished at regular intervals. The assessments were carried out 14 days following the immersion date. Following the removal of the specimens from the solution, the surfaces were cleaned with a soft nylon wire brush under running tap water to eliminate any weak products or loose debris. The specimens were then allowed to dry on the surface before all measurements were obtained. The weight loss/gain was evaluated using the starting measurement and measurements taken at certain intervals.

The compressive strength also tested for geopolymer was carried out according to ASTM C109 by using Hydraulic Compression Testing Machine at the rated load speed of 0.6 N/mm²/s [23]. Then, the metakaolin geopolymer was tested for density after being exposed at different acid attacks by dividing the weight of the specimen by the volume of the geopolymer cube following ASTM C642 [21]. Scanning Electron Microscopy (SEM) is a technique used to observe the morphology of the samples including the raw materials at magnifications of 1000×

3. Results and discussion

3.1. Durability and mechanical properties analysis

3.1.1. Water absorption

Fig. 2 illustrates the results of water absorption of a sample exposed and unexposed in various concentrations of sulphuric acid. Overall, sulfuric acid at 2% had the lowest water absorption, while sulfuric acid at 5% had the greatest water absorption, resulting in water absorption values of 7.67% and 27.31%, respectively. The amount of water absorbed increased linearly in proportion to the rise in the acid percentage of sulphuric acid in the solution. The results of this test are consistent with the results of the compressive strength test. In terms of water absorption by immersion, the 2 percent of an acid percentage that has the least water absorption is the one with the maximum compressive strength. Geopolymer samples with a 5 percent acid concentration exhibit water absorption rates of around 27.31 percent. This means that compressive strength is directly influenced by open porosity as stated by Vieira et al. [24]. This evidence can be supported by the geopolymeric mortar with the lowest open porosity having the highest compressive strength [25-27].

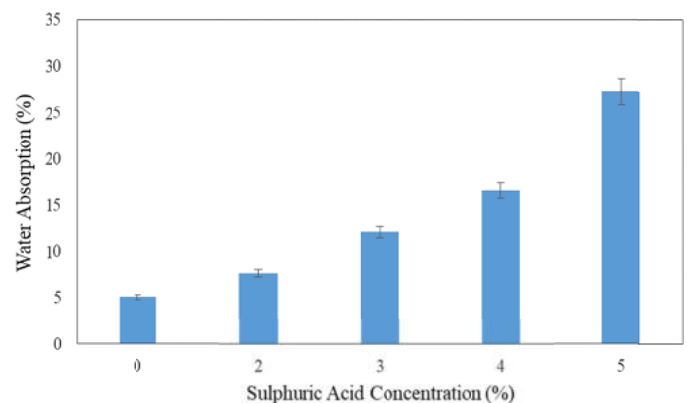


Fig. 2. Water Absorption of metakaolin geopolymer specimens after 14 days of exposure to different concentration of sulphuric acid

3.1.2. Compressive strength

For the compressive strength test, five different samples were employed, as shown in Fig. 3. It was discovered that the control sample had the maximum compressive strength (32.5810 MPa), whereas the sample submerged in 5% acid had the lowest compressive strength (4.2523 MPa). Low compressive strength is caused by a sample with a high acid value. As was to be expected, the exposure of each sample to acid resulted in a decrease in its strength, and this decrease in strength increased in proportion to the concentration of acid content. This is because acid reacts with basic or alkaline to generate this reaction. Alkaline compounds, such as geopolymers, are produced when a strong alkali activator is used, and

these materials react with acids. The acid-basic interaction that took place between the geopolymer and the acid solution was primarily responsible for the reduction in strength. According to Bougueremouh et al. [28], hydrochloric acid immersion reduced the compressive strength of the sample. After the sample was immersed in sulphuric acid, a fracture formed. The stress value of compressive strength decreases as a result of the presence of voids between the sample and the structure. The cracks were most likely caused by the shrinkage of the corroded gel layer and serve as channels for sulphuric acid penetration into the internal microstructural [29-32].

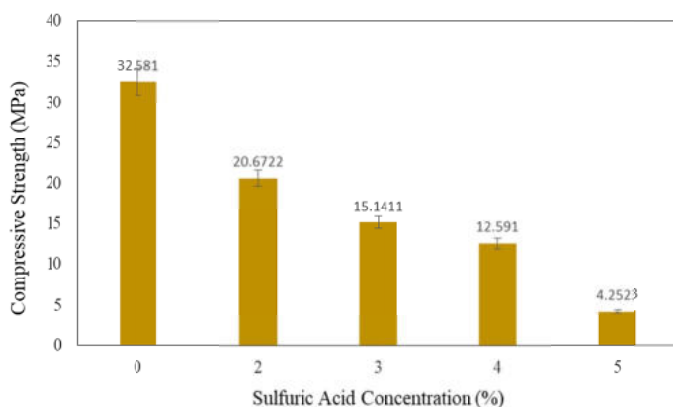


Fig. 3. The compressive strength of metakaolin geopolymer specimens after 14 days of exposure to different concentration of sulphuric acid

3.1.3. Density

The density of the sample before and after immersion in various concentrations of sulphuric acid is shown in TABLE 1. When the sample was submerged in a higher concentration of sulphuric acid, the density value of the sample decreased. The difference in density of the sample before and after immersion in 2 percent sulphuric acid was 17.07 g/cm^3 , whereas the change in density after immersion in 5 percent sulphuric acid was 62.13 g/cm^3 . This may be explained by the fact that the more the proportion of acid present, the decrease the acid resistance and the greater the differential between the density sample and the density following immersion in sulphuric acid, as illustrated in Fig. 4. As claimed by Sata et al., the reaction between the calcium hydroxide ($\text{Ca}(\text{OH})_2$) found in the sample and the acid,

TABLE 1

The density result of metakaolin geopolymer before and after immersed in different sulphuric acid concentration.

Sulphuric Acid Percentage (%)	Density before (g/cm^3)	Density after immersed in Sulphuric acid (g/cm^3)	Density loss (g/cm^3)
0	227.428	227.428	0.00
2	222.482	205.410	17.07
3	230.534	202.714	27.82
4	234.733	195.749	38.98
5	227.564	165.427	62.13

which may cause tensile stress and lead metakaolin geopolymer to crack and fractured was the main cause of weight loss in metakaolin based geopolymer [33]. Additionally, a high acid value will cause corrosion on the surface of the metakaolin geopolymer sample [14,16]. As a consequence, the value of density loss will increase, which will ultimately lead to an increase in the water absorption value, which will result in a decrease in compressive strength. As stated by Risdanareni et al., a drop in density results in a decrease in compressive strength, which is consistent with the findings above [34].

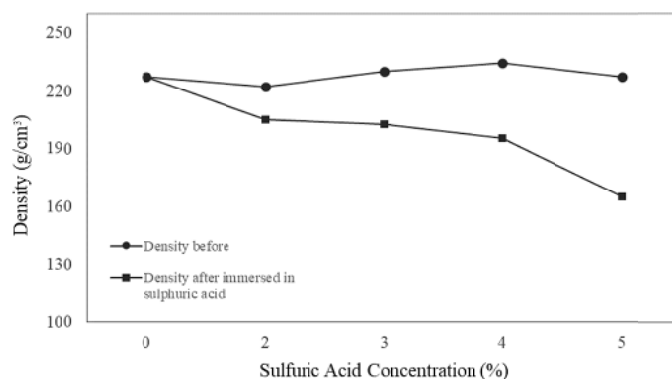


Fig. 4. The density of metakaolin geopolymer specimens after 14 days of exposure to different concentration of sulphuric acid

3.1.4. Microstructure analysis

The microstructure of metakaolin geopolymer paste at different acid concentrations is seen in Fig. 5. Testing and investigation were conducted on the variations in acid percentages of metakaolin-based geopolymer samples ranging between 2 percent and 5 percent. To assess the crack distribution on metakaolin geopolymer paste, this research was carried out to determine the crack distribution. Cracks and porosity are less common when the acid concentration is 2 percent as opposed to 5 percent. Hence, the structure after exposed at 5% of H_2SO_4 resulted in less dense. This clarifies the compressive strength value decreases in direct proportion to the number of fractures visible on the surface.

At $2000\times$ magnification, samples of metakaolin geopolymer paste with 2 percent acid percentage labelled (a) and 5 percent acid percentage labelled (b) are displayed. Due to the increasing porosity of the samples, each picture indicated the degraded layer of the geopolymers, which was characterized by a bigger proportion of cracks. Each sample's degraded layer has cracks of varying diameters in addition to the greater porosity. It can be stated that there a little crack occurs at 2% acid percentage compared to a 5% acid percentage which shows clearer and wider cracks occur. According to previous researchers, the metakaolin geopolymer paste developed more fractures as the acid proportion of sulphuric acid concentration increased [14,35].

Cracks were seen in both the deteriorated layer and the unaffected core of the samples, perhaps generated by high temperatures before treatment with metakaolin and alkaline activator

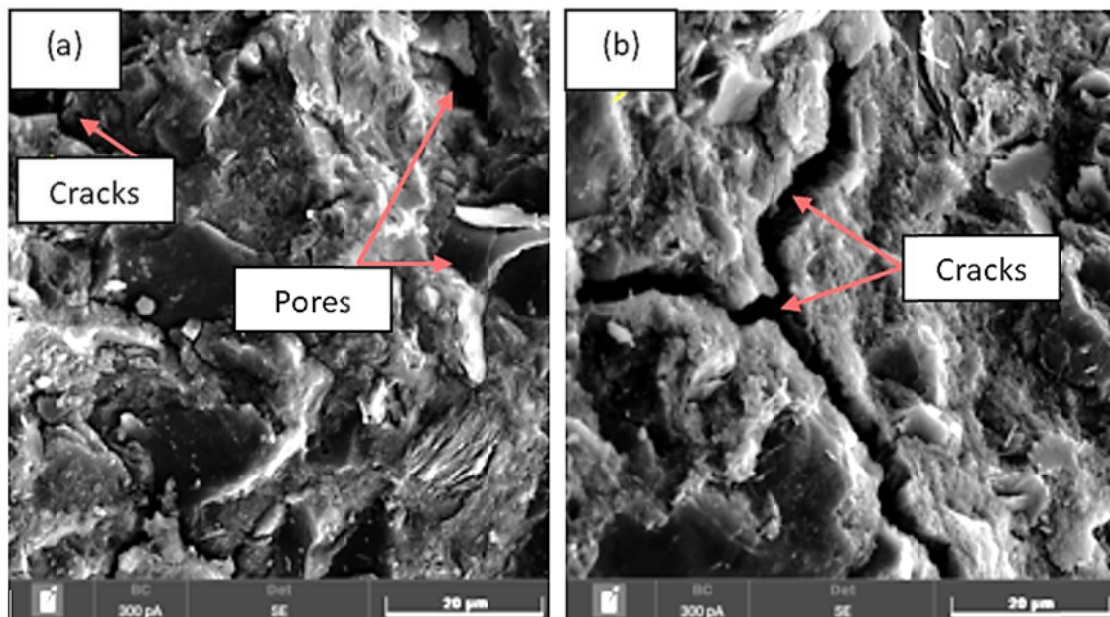


Fig. 5. SEM images of geopolymer concrete exposed to different concentration of sulphuric acid; (a) 2% H_2SO_4 and (b) 5% H_2SO_4

during sample preparation. On the degraded layer, there were more cracks, notably parallel to the transition zone between the degradation layer and the sample's presumably undamaged core [36]. Due to the high viscosity of metakaolin geopolymer pastes, it was unable to eliminate air trapping using a vibration table, as shown by the rare dark patches in the region of the seemingly undamaged core that reveal air holes.

Likewise, sulphuric acid (H_2SO_4) is one of the most harmful acids to act on concrete because of the combination of acid and sulphate attacks, claim Sata et al. [33]. After being exposed to a high concentration of sulfuric acid, the metakaolin geopolymer suffered damage after the growth of the cracks, as shown in Fig. 6, due to the penetration of sulfuric acid into its constituent parts.

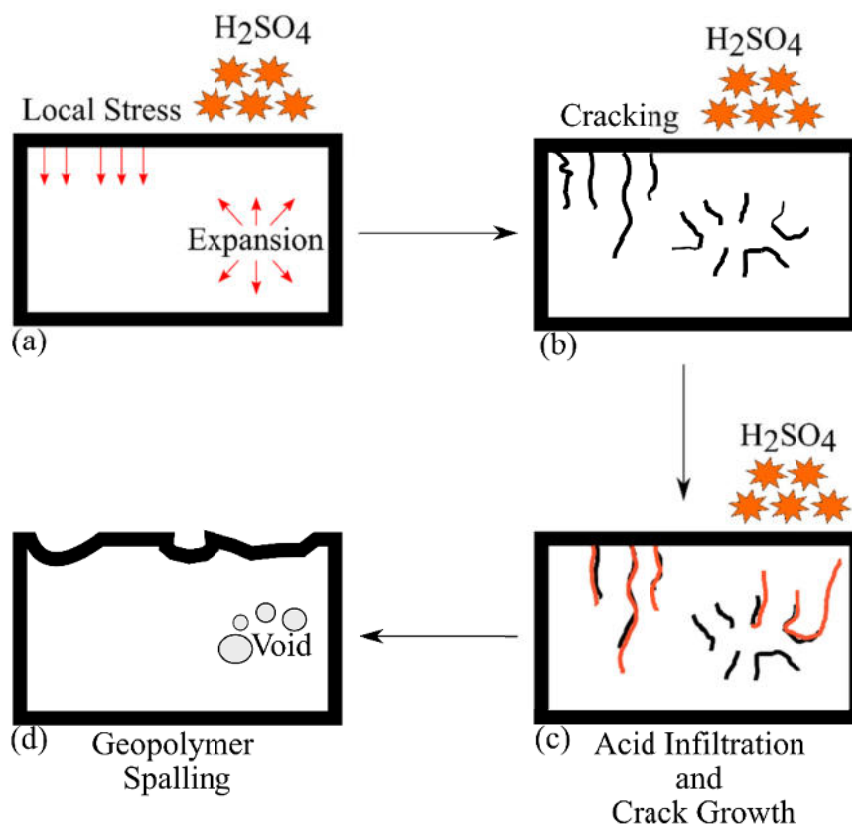


Fig. 6. Damage to the metakaolin geopolymer concrete as a result of (a) development of tension, (b) cracking of the concrete, (c) enlargement of cracks and acid penetration and (d) damage to the concrete itself

4. Conclusion

In this work, the durability qualities of geopolymer were investigated when exposed to different acid percentage attacks. The microstructure investigation on the geopolymer paste that had been immersed in various sulphuric acid percentages resulted in the formation of a wider crack, resulting in a drop in the compressive strength value. Degradation parameters such as acid percentage, weight change, and compressive strength of geopolymer specimens were also studied. The rising of sulphuric acid concentration causes a loss in compressive strength, an increase in water absorption, and a drop in density. All testing, including durability, mechanical, and physical tests, showed that the metakaolin geopolymer paste submerged in a 2% acid solution generated the best results. This outcome will increase the workability of the final product while also increasing its strength. To be specific, metakaolin based geopolymer were more resistant and durable to sulphate attacks than the conventional Portland cement mortars since they contained pozzolans like metakaolin with low calcium content owing to the more stable cross-linked aluminosilicate polymer structure.

Acknowledgement

The author would like to acknowledge the support from the Fundamental Research Grant Scheme (FRGS) under a grant number of FRGS/1/2019/TK10/UNIMAP/02/21 from the ministry of Education Malaysia.

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