

## Research Article

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# Investigating the effect of locally available volcanic ash on mechanical and microstructure properties of concrete

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**Abstract:** Environmental degradation is developing due to rising pollution from the depletion of raw materials and the growing mandate for concrete goods. Investigators and experts have focused on creating sustainable concrete utilizing renewable elements. Volcanic ash (VA) is a promising supplementary cementitious material among these minerals. Therefore, it is crucial to examine the attributes of voids in aggregate and how they impact the performance of concrete. VA from the Gini Chilas (Gigilat Baltistan) was used to prepare specimens. Mixing regimes of VA concrete with altering concentrations ranging from 0 to 40% replacement was cast. Water-to-cement ratio was reserved persistent for all the mixes. Chemical compositions of VA and properties of concrete in relation to workability, density, and compressive strength were carried out. In addition, thermo-gravimetric analysis, scanning electron microscope (SEM), and X-ray diffraction analysis were also examined. The analysis of results reveals that VA with 10% replacement gives an adamant response. This is due to the natural pozzolanic effect that details the creation of additional dense gel (C–S–H), and deviation of cracks is observed from SEM. VA<sub>10</sub> also exhibits thermally stable behavior at temperature with

less percentage mass loss. However, VA up to 10% replacement in cementitious concrete can exhibit better properties than normal specimens.

**Keywords:** compressive strength, scanning electron microscope, thermo-gravimetric, volcanic ash, workability, and X-ray diffraction

## 1 Introduction

Concrete is often regarded as the predominant construction material, exhibiting a substantial global use estimated to be in the billions of cubic meters worldwide [1–4]. Moreover, the outcome of cement is seeing significant increases in direct correlation with the consumption of concrete [5]. Nevertheless, it is worth noting that the cement production process, particularly during the grinding and clinker burning stages, is associated with significant levels of greenhouse gas emissions (GHGs) [6]. The manufacture of cement is accountable for around 6% of global carbon dioxide (CO<sub>2</sub>) emissions, thus contributing to the phenomenon of global warming [7]. Furthermore, the production of cement is responsible for 95% of the total CO<sub>2</sub> production associated with the manufacturing of one cubic meter of concrete [8]. The Portland cement (PC) industry is acknowledged as a significant contributor to global CO<sub>2</sub> emissions, accounting for an estimated 5–7% of the total emissions [9–11]. In addition, the annual global production of cement falls between 2.8 and 4.1 billion tons. It is reported that the production of PCs amounts to 4,000 million tons yearly and is projected to increase to around 6,000 million tons by the year 2060 [12]. These figures mentioned above can be further diminished through the augmentation of alternative materials in the process of concrete manufacture [13–18]. Therefore, the cement industry exhibits a rather high level of pollution in the atmosphere. Moreover, it is imperative to utilize financial strategies that can serve as viable substitutes for cement. Therefore, the implementation of these approaches results in a decrease in the utilization of cement within

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concrete mixtures [14]. Researchers conducted studies to explore the potential of utilizing industrial by-products and waste materials in concrete as a means of environmental conservation to develop green concrete [19–25]. Therefore, to reduce clinker production and CO<sub>2</sub> emissions, supplementary cementitious materials (SCMs) such as fly ash (FA) [26–28], silica fume (SF) [29–31], electronic arc furnace slag [32–34], rice husk ash (RHA) [35–37], wheat straw ash [38–40], and ordinary pozzolans are employed as substitutes for cement. These materials lack inherent binding qualities and are considered the most cost-effective alternatives to cement [41]. Hence, the incorporation of these materials can result in a decrease in the proportion of ordinary Portland cement (OPC) used in the manufacturing of concrete. The utilization of SCMs in the manufacturing of cement concrete offers additional environmental advantages. This practice mitigates the accumulation of excessive waste in open areas and landfills, which is known to contribute to environmental contamination and pose risks to human health. Furthermore, the incorporation of their presence inside cementitious composites (CCs) enhances various properties in the matrix, as depicted in Figure 1.

Durable building material SCMs can originate after manufacturing waste such as slag, FA, and ground granulated blast-furnace slag, as well as cultivated waste such as RHA and palm oil fuel ash [39]. The aforementioned SCMs demonstrate a notable degree of pozzolanic reactivity and fall under the classifications of natural pozzolans. Hence, these materials possess the potential to serve as limited substitutes for cement. This results in the attainment of cement mortar with acceptable performance or even concrete with enhanced performance [9]. Hence, the utilization

of pozzolans as a substitute for cement in concrete blends leads to a substantial reduction in GHGs that are sent into the environment [1,2,42,43]. The utilization of natural pozzolanas as a partial replacement for PC has been extensively practiced in many applications. This is primarily attributed to the presence of reactive SiO<sub>2</sub> in natural pozzolanas, which imparts several advantageous properties such as diminished heat generation, reduced permeability, and enhanced resistance to chemical deterioration in the CC. Therefore, it is possible to utilize abundant naturally occurring pozzolanic materials [3,44–46], such as volcanic ash (VA) [47–49], volcanic pumice [50–52], and calcined clay [53–55], as a viable substitute to fulfill the requirements of the concrete industry.

Volcanic concrete is an environmentally friendly material that incorporates components derived from naturally occurring igneous volcanic rock [56–58]. When volcanic rock is crushed into various particle sizes, it can be utilized as coarse aggregate, fine aggregate, or as a SCM in concrete production. VA offers significant environmental advantages compared to other SCMs, such as FA and slag. As a naturally available material, VA requires minimal processing, resulting in lower energy consumption and reduced carbon emissions. In contrast to FA, a byproduct of coal combustion, and slag, a byproduct of steel manufacturing, VA is not reliant on industrial processes, making it a more sustainable alternative. Its natural abundance in certain regions also reduces transportation needs, lowering the overall environmental impact. By partially replacing cement, VA helps decrease CO<sub>2</sub> emissions in concrete production, contributing to more sustainable construction practices.

VA is a non-reactive siliceous pozzolanic substance that, when mixed with lime and water, produces a cementitious material with exceptional structural properties. The substance can efficiently occupy the empty voids among the bigger particles [57]. Thus, it diminishes the exothermic reaction of hydration and undergoes a chemical reaction with calcium hydroxide Ca(OH)<sub>2</sub>, resulting in the formation of calcium silicate hydrate [56]. Several academic studies have analyzed how ordinary VA affects the mechanical and microstructural properties of mortar and concrete. Sebayang [59] investigated the effect of the utilization of VA in concrete and reported a significant improvement of 9.8% in compressive strength and 5.99% in tensile strength, respectively. In addition, Karolina and Simanjuntak [60] revealed that the workability of concrete is reduced with an increase in the ratio of VA as a substitute for cement. Hossain and Lachemi [61] demonstrated that a compressive strength of 60 MPa was attained after 28 days when 20% volume of admixture (VA) was used as a substitute for cement. Furthermore, their findings indicated that the

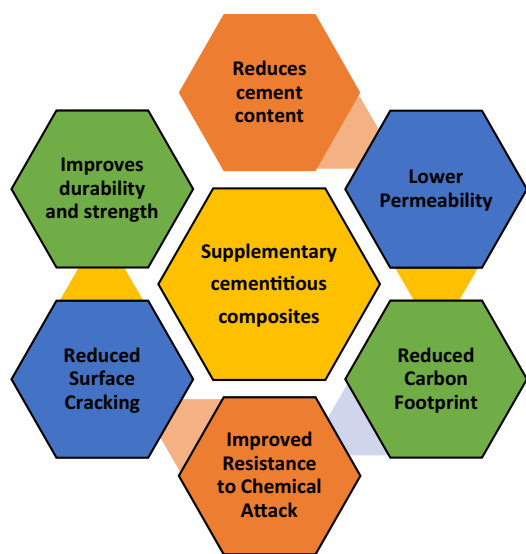


Figure 1: SCM benefits in CC.

optimal ratio for incorporating VA was determined to be 5%, regardless of the range of VA concentrations tested, which spanned from 0 to 20%. Khan *et al.* [62] demonstrated that the pozzolanic activity exhibited an upward trend in correlation with the degree of fineness of the material. Nevertheless, the use of heat treatment on VA yielded unfavorable results. Abdullah *et al.* [63] conducted a study that revealed the positive impact of volcanic pumice stone ash on the compressive strength of self-compacting concretes. This finding highlights the significance of the fineness level of VAs to the mechanical properties of such concretes. Al-Fadala *et al.* [64] investigated the effect of VA with varying concentrations ranging from 10 to 30% in cementitious matrix. The author reveals that the inclusion of VA at a 10% replacement level in cement led to a little decrease in quality. In contrast, the incorporation of VA in cement at replacement ratios of 20 and 30% resulted in notable decreases in strength as associated to the resistor samples. Additionally, the incorporation of VA powder as a partial substitute for OPC has been shown to result in reduced water absorption, sportively, and void content, as well as enhanced resistance to acid and sulfate assault, chloride permeability, and other factors, when compared to conventional concrete [65,66]. Al-Bahar *et al.* [67] discovered that the substitution of 10–30% of OPC with VA leads to a notable enhancement in both the mechanical and micro-structural characteristics of cement paste. Moreover, the

durability properties of volcanic ash concrete (VAC) were examined by Hossain and Lachemi [68,69]. Their investigation encompassed the evaluation of fresh, hardened, and durability characteristics. The findings of their study indicate that VAC exhibits superior durability capabilities in comparison to the control concrete sample containing 0% VA. Nevertheless, increasing the concentration of VA has a malignant consequence on the strength of VAC. Celik *et al.* [66] successfully formulated a high-volume natural pozzolanic concrete, which involved replacing 45% of OPC. The resulting matrix substantially exhibited a 28-day compressive strength of 34 MPa. Liu *et al.* [70] studied the impact of VA on the thermal stability of asphalt. The author indicated that the inclusion of VA led to enhancements in the mechanical characteristics of asphalt mixtures. Previous research studies have shown evidence that mechanical, chemical, and thermal treatments can enhance the properties of natural pozzolans. Additionally, the significance of VA in concrete is illustrated by a scientometric graphic, as presented in Figure 2. However, large-scale adoption of VA faces challenges, such as variations in its chemical composition, which can affect performance consistency in concrete. Additionally, in regions where VA is not readily available, transportation costs may hinder its widespread use.

The objective of this research was to examine the impact of incorporating VA as an auxiliary for cement in the production of a CC. The variable concentration of VA is

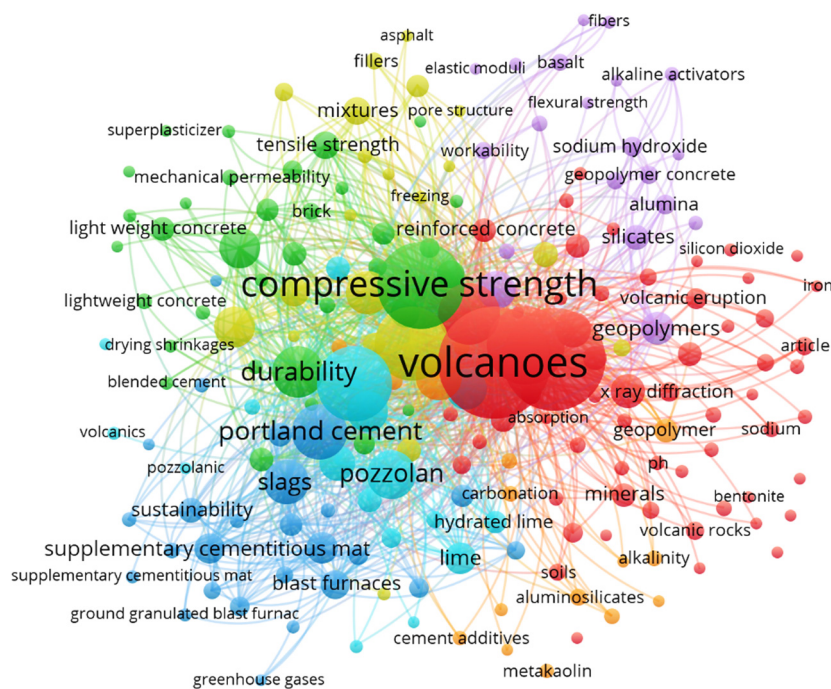


Figure 2: Scientometric diagram of VA.

substituted with a range spanning from 0 to 40%. The evaluation of fresh and hardened qualities is initially performed through experimental tests, specifically focusing on workability, density, and compressive strength. In addition, VAC specimens are subjected to a temperature range of 0–900°C to assess the effects on the pozzolanic material. The chemical analysis of VA concrete is performed using X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis techniques. Furthermore, a comprehensive analysis of the mixes was performed using XRD and thermogravimetric analysis (TGA) methods to assess the influence of VA on the microstructure of the CC.

## 2 Materials and methods

### 2.1 Materials

For the manufacture of specimens, OPC Type 1 Grade 42.5-N and natural sand conforming to ASTM C150 [71] and ASTM C128 [72] standards were utilized. The VA used in this study was sourced from Gini Chilas, located in Gilgit Baltistan, Pakistan, as shown in Figure 3. This particular source was chosen due to its accessibility, abundance, and established history of local use in construction. Additionally, it was selected based on the initial chemical analysis, which indicated suitable pozzolanic activity for use in concrete mixtures. The chemical composition of the cement and VA was analyzed using XRF, with results presented in Table 1. Tables 2 and 3 show the physical properties of the materials used in the VAC mix.

It is important to note that the properties of VA can vary depending on the source due to differences in mineral composition, geographic location, and volcanic activity. If VA from other regions was used, the results of the concrete's mechanical



Figure 3: VA specimen.

Table 1: Chemical composition of cement and VA

Oxide	VA (% age by mass)	PC (% age by mass)
SiO <sub>2</sub>	53.69	21.5
Al <sub>2</sub> O <sub>3</sub>	17.43	6.00
Fe <sub>2</sub> O <sub>3</sub>	9.52	3.75
CaO	7.00	62.00
MgO	3.87	2.8
Na <sub>2</sub> O	3.57	0.2
K <sub>2</sub> O	0.86	1.00
SO <sub>3</sub>	0.16	2.75
Lime saturation factor	3.89	96.40
Silica modulus	1.99	2.5
Aluminum modulus	1.83	1.25
LOI	1.3	6.64

and durability properties could differ. Variability in silica, alumina, and other oxide contents, as well as particle fineness, could affect the pozzolanic reaction, leading to variations in the concrete's strength development, workability, and overall performance.

### 2.2 Mix proportions and methods

A comprehensive set of 81 samples was prepared with distinct mix proportions. These formulations include a control sample that exclusively utilized cement as a binder. A water-to-cement ratio of 0.5 was employed for all the mixtures. Table 4 provides comprehensive information regarding the mix proportion and laboratory testing conducted on all the formulations at various ages. The ingredients of the formulation were mixed in a pan mixer. Initially, the mixer was filled with both fine and coarse materials, which were then followed by the addition of cement. A period of 1 min was allocated for the dry mixing of the ingredients, with a rotational speed of 180 revolutions per minute (rpm) at a slow rate. Subsequently, a quantity equivalent to half of the total volume of water was introduced into the mixture, with the

Table 2: Physical properties of ingredients

Characteristics	Cement	VA
Insoluble residue (% mass)	0.55	—
Specific gravity (g·cm <sup>-3</sup> )	3.15	2.67%
Specific surface area (m <sup>2</sup> ·g <sup>-1</sup> )	0.83	—
Particle size ( <i>d</i> <sub>50</sub> ) (μm)	16.58	—
loss on ignition (% mass)	2.21	—
Soundness	No expansion	No expansion



**Table 3:** Physical and chemical properties of aggregates

	Fine aggregate		
	Physical properties	Chemical composition	
Size (mm)	—	CaO	9.97
Specific gravity ( $\text{g}\cdot\text{cm}^{-3}$ )	2.64	$\text{SiO}_2$	49.12
Water absorption (%)	1.62	$\text{Al}_2\text{O}_3$	2.48
Bulk density ( $\text{kg}\cdot\text{m}^{-3}$ )	1,546	—	—
Crushing value	—	$\text{Fe}_2\text{O}_3$	38.97
Fineness modulus	2.25	$\text{K}_2\text{O}$	1.24

**Table 4:** Concrete mix proportions

Concrete mix composition					
Mixes	Cement ( $\text{kg}\cdot\text{m}^{-3}$ )	VA ( $\text{kg}\cdot\text{m}^{-3}$ )	Water ( $\text{kg}\cdot\text{m}^{-3}$ )	Fine aggregate ( $\text{kg}\cdot\text{m}^{-3}$ )	Coarse aggregate ( $\text{kg}\cdot\text{m}^{-3}$ )
Control sample	320	0	160	640	1,280
V <sub>5</sub>	304	16	160	640	1,280
V <sub>10</sub>	288	32	160	640	1,280
V <sub>15</sub>	272	48	160	640	1,280
V <sub>20</sub>	256	64	160	640	1,280
V <sub>25</sub>	240	80	160	640	1,280
V <sub>30</sub>	224	96	160	640	1,280
V <sub>35</sub>	208	112	160	640	1,280
V <sub>40</sub>	192	128	160	640	1,280

ongoing process of stirring being maintained at its original velocity for an additional duration of 2 min. Subsequently, the addition of the VA was carried out, followed by the introduction of the remaining water. The mixing process was then extended for 3 min, maintaining a rotational speed of 360 revolutions per minute (rpm). Initially, the slump test was conducted according to ASTM C143 [73], immediately after mixing, to assess the workability of fresh concrete. For compressive strength testing, 81 cylindrical specimens (100 mm × 200 mm) were prepared, with nine specimens for each mix. After demolding at 24 h, specimens were cured at 23°C and 95% humidity. Compressive strength tests were carried out at 7, 14, and 28 days, following ASTM C39 [74].

## 2.3 Test methods

### 2.3.1 Slump test for VAC

Slump tests were passed according to ASTM C143 [73] to determine the consistency of the mixes. The workability of fresh concrete was assessed by the slump test, which involved utilizing a steel cone with certain dimensions: a top diameter of 100 mm, a bottom diameter of 200 mm, and

a height of 300 mm. Consistency in the slump testing was ensured by using the same water-to-cement ratio and mixing procedures for all batches, regardless of VA concentration, with experienced personnel conducting the tests to maintain accuracy.

### 2.3.2 Tests for hardened concrete

Hardened VAC was evaluated and compared with OPC concrete by conducting tests for dry density and compressive strength following ASTM criteria [74,75].

### 2.3.3 XRD

XRD spectra of specimens were obtained by using a JPX 3522 JEOL with a sensitive detector. The finely ground samples of the specimens were placed in metal sample holders and inserted into the diffractometer. XRD measurements were conducted using Cu K $\alpha$  radiation with a wavelength of 1.54 Å, at 40 mA and 40 kV. XRD data were collected using a continuous scan mode with  $2\theta$  angles ranging from 0 to 160°. To ensure accuracy, calibration was performed using a standard silicon sample, which is widely

accepted for calibrating XRD instruments. The calibration ensured that peak positions and intensities were accurate and reliable. While XRD effectively identifies crystalline phases, it has limitations in detecting amorphous materials, which may be significant in VA. Additionally, small quantities of crystalline phases might not be accurately identified due to XRD's detection limit.

### 2.3.4 TGA

TGA was utilized to measure the quantity of calcium hydroxide (CH), identify other phases in the cement pastes, and assess the extent of pozzolanic reactions. TGA was conducted using a STA 8000 Perkin Elmer instrument from the USA, which can measure mass changes as small as 1  $\mu\text{g}$ . The apparatus was controlled using a dynamic heating ramp of  $10^\circ\text{C}\cdot\text{min}^{-1}$  between 30 and  $1,000^\circ\text{C}$ . The samples were crushed to a size of 0.4  $\mu\text{m}$  without any additional preprocessing to prevent destabilization of hydrates. The test was conducted in a nitrogen atmosphere provided at a flow rate of  $60\text{ mL}\cdot\text{min}^{-1}$ . TGA was used to measure calcium hydroxide (CH) content, identify other phases in cement pastes, and assess pozzolanic reactions. The analysis was performed with a STA 8000 Perkin Elmer instrument, capable of detecting mass changes as small as 1  $\mu\text{g}$ . The apparatus operated with a dynamic heating ramp of  $10^\circ\text{C}\cdot\text{min}^{-1}$  between 30 and  $1,000^\circ\text{C}$ . Samples were crushed to a size of 0.4  $\mu\text{m}$ , and the test was conducted in a nitrogen atmosphere at a flow rate of  $60\text{ mL}\cdot\text{min}^{-1}$ . TGA provides insight into the thermal stability of phases but may struggle to distinguish between phases with similar decomposition temperatures. Additionally, sample preparation (such as crushing) could introduce artifacts, potentially affecting the accuracy of the analysis.

### 2.3.5 Microstructural analysis of VAC

The concrete sample's microstructure was analyzed with a scanning electron microscope (SEM). SEM examination was conducted at Peshawar University, Pakistan, using a JEOL-JSM 5910LV microscope with a magnification range of  $0\times$  to  $300\times$  and a maximum resolving power of 2.3 nm. The samples for SEM analysis of concrete specimens were extracted from the fractured particles of specimens subjected to compressive strength testing. The samples were sliced to a diameter of 20 mm and a height of 10 mm. The samples were kept in the laboratory under ambient conditions for 7 days to ensure that they were well dry before being coated with gold for SEM imaging.

## 3 Results and discussion

The chemical composition of unprocessed VA, as determined by XRF investigation, is presented in Table 1. The principal elements found in VA are silica ( $\text{SiO}_2$ ), alumina ( $\text{Al}_2\text{O}_3$ ), iron oxide ( $\text{Fe}_2\text{O}_3$ ), calcium oxide ( $\text{CaO}$ ), and magnesia ( $\text{MgO}$ ). Silica has the highest weight content, with a value of 47.02 wt%. The combined percentage of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Fe}_2\text{O}_3$  is approximately 70%, indicating that VA meets the minimal criteria established by ASTM C618-15 [76] for natural pozzolans.

### 3.1 Workability

Figure 4 illustrates the feasibility of incorporating VA into CCs. The data reveal that the slump of volcanic concrete varied from 39 to 75 mm as the replacement level of VA increased. Furthermore, Table 1 shows a decline in workability as VA concentrations increased. This reduction in workability can be attributed to the increased water absorption capacity of VA due to its smaller particle size. Additionally, VA exhibits rough-edged polygonal particles, unlike the generally rounded particles found in cement. This shape difference may result in reduced wetting and increased inter-aggregate frictional resistance. As a result, the reduction in free water content while maintaining a consistent water-to-cement (W/C) ratio leads to decreased workability, as VA absorbs more water, reducing fluidity.

To address this, several strategies can improve workability in mixes with high VA concentrations. Adding

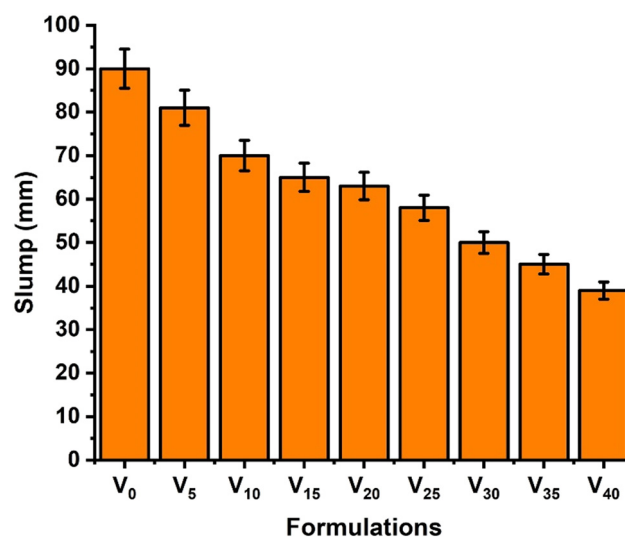


Figure 4: Slump test of intruded VA in concrete.

superplasticizers or other water-reducing admixtures can significantly enhance fluidity without altering the W/C ratio. These admixtures counterbalance the increased water absorption and frictional resistance by dispersing the cement particles more effectively, thus improving flowability [77]. Additionally, using finer VA particles or pre-wetting the ash before mixing may mitigate workability issues by reducing water absorption during mixing [78].

### 3.2 Density

The density of concrete plays a crucial role in influencing its mechanical properties and durability. Higher-density concrete generally exhibits enhanced strength, reduced porosity, and lower permeability to water and chemicals, contributing to increased durability and longer service life. As shown in Figure 5, the series of concrete mixtures containing VA demonstrated slightly higher densities compared to the reference concrete after a 28-day curing period. This increase in density can be attributed to the filler effect of VA, which fills the pores within the concrete matrix, improving compactness by freeing trapped water. Silva *et al.* [79] also highlighted the limited filler capacity of materials used as cement substitutes, indicating that beyond a certain threshold, further increases in density may not occur. However, in this study, the VA-enhanced particle arrangement contributed to the observed increase in density, which in turn resulted in enhanced strength and durability.

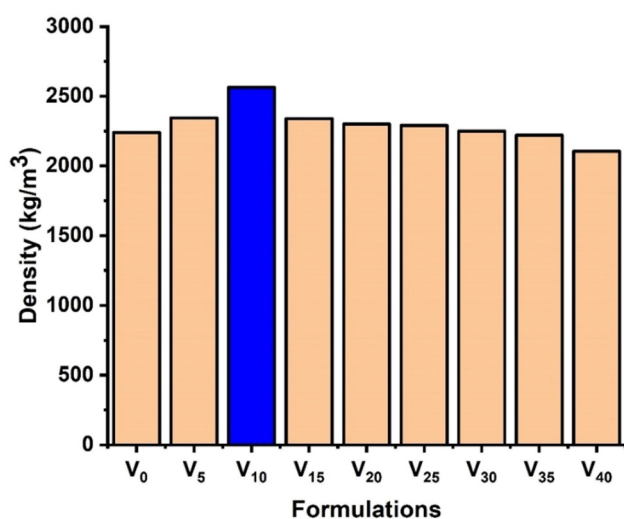


Figure 5: Formulation density.

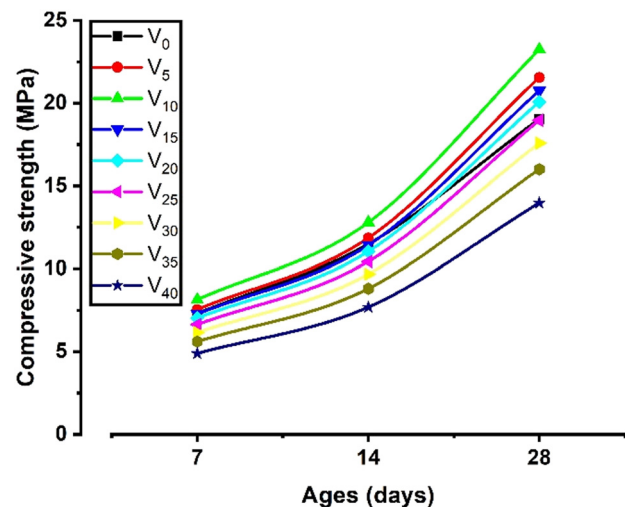


Figure 6: Compressive strength of VAC.

### 3.3 Compressive strength of VAC

The compressive strength data for concrete reinforced with nano media at various time intervals are presented in Figure 6. The compressive strength of the VA concrete exhibited superior performance compared to the reference sample, up to a concentration of 10%. The compressive strength exhibits an initial increase followed by a subsequent drop in comparison to the control sample. The observed increase in strength can be due to the presence of reactive silica and alumina in VA, which have the potential to undergo pozzolanic reactions with calcium hydroxide (lime) generated during the hydration process of PC. These reactions have a role in the generation of SCMs. Consequently, this leads to an improvement in the overall

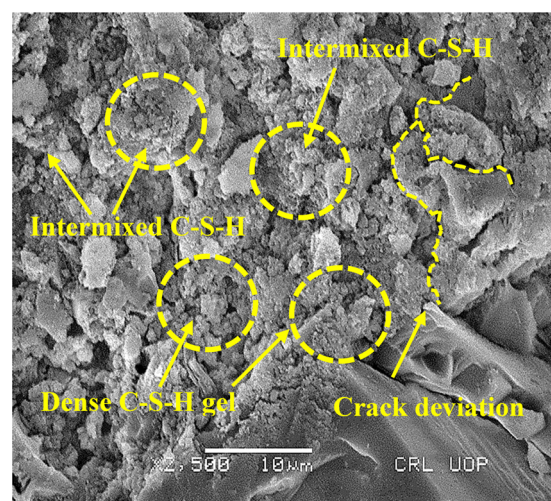
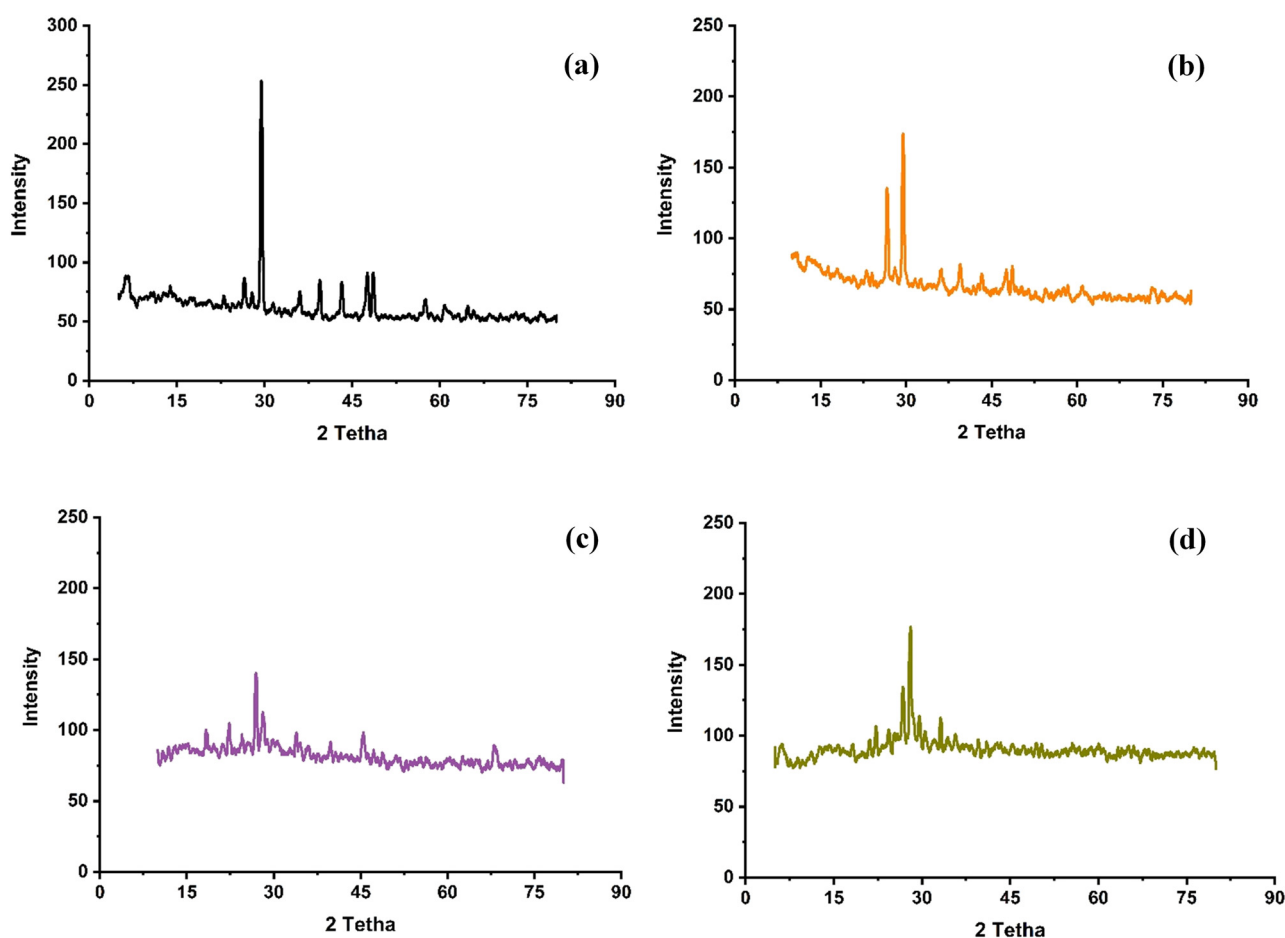


Figure 7: SEM of VA with 10% concentration.

structural integrity of the concrete material. Furthermore, the particle size and distribution of VA have the potential to influence the arrangement of particles inside the concrete mixture. An ideal configuration for packing can result in enhanced density and heightened strength. Nevertheless, the presence of large quantities of tiny particles of volcanic ash (VA) and unreactive silica might give rise to complications, including heightened water requirements and diminished workability. As a result, this leads to a detrimental effect on the overall strength. Furthermore, the SEM analysis of the V10 sample is illustrated in Figure 7. The infiltration of VA occurs within an extremely compact and dense C–S–H gel. The increased density of the gel structure has a significant role in enhancing the overall strength of the concrete. Additionally, deviations from the conventional linear crack patterns have been noticed.

### 3.4 Analysis of XRD of VA-based concrete

The assessment of the impact of VA on concrete is conducted by using XRD analysis, a technique that aids in the identification and characterization of its crystalline or pozzolanic properties, as illustrated in Figure 8. The control sample depicts the presence of high  $\text{Ca(OH)}_2$  as compared to the remaining formulations, as shown in Figure 8(a). In addition, the presence of amorphous and glassy silica in VA undergoes a transformation into calcium–silicate–hydrate (C–S–H) upon interaction with portlandite, a constituent of cement. During the process of pozzolanic reactions, the ash particles undergo a combination with the  $\text{Ca(OH)}_2$  phase present in the cement matrix. This combination results in the formation of supplementary C–S–H gels, as illustrated in Figure 8(b) and (c). The



**Figure 8:** XRD: (a) control specimen; (b) VA with 10% replacement; (c) VA with 20% replacement; and (d) VA with 40% replacement.

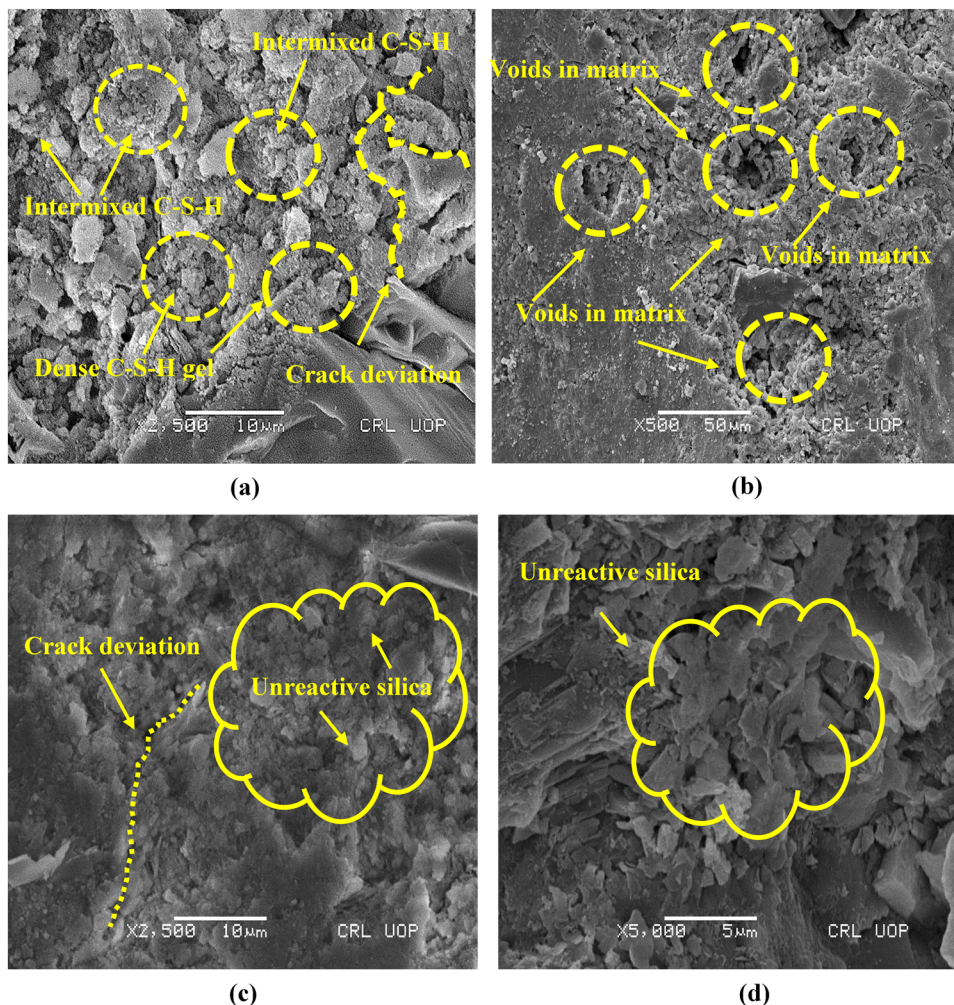


augmentation of additional C–S–H gels in this production process contributes to the enhancement of the concrete's mechanical strength. Therefore, the decrease in the presence of the portlandite phase within the cement paste can be utilized as a reliable signal to assess the pozzolanic capacity of different cement materials. The control concrete sample demonstrates a significantly elevated  $\text{Ca}(\text{OH})_2$  intensity in comparison to the remaining samples. The reason for this is that the control samples consist of a notable proportion of cement, which plays a crucial role in the process of hydration and the creation of  $\text{Ca}(\text{OH})_2$ . On the contrary, an inverse relationship was observed between the fraction of VA and the level of  $\text{Ca}(\text{OH})_2$  in the remaining samples. The observed reduction in  $\text{Ca}(\text{OH})_2$  concentration can be attributed to either the inert properties of VA or the limited presence of cement in these particular mixes as demonstrated in Figure 8(d). As a result, this phenomenon results in a decline in compressive strength beyond the appropriate dosage of VA.

### 3.5 Microstructure analysis of VA-based concrete

VA has been identified as a pozzolanic material capable of enhancing the strength and durability of concrete, as shown in Figure 9. The control sample, illustrated in Figure 10(a), exhibits fractures and voids, which lead to reduced strength and density. These inherent flaws in the microstructure compromise the concrete's workability, strength, and durability by creating pathways for the ingress of water and corrosive chemicals, ultimately reducing its lifespan.

In contrast, the microstructural improvement observed when 10% of the cement is replaced with VA, as depicted in Figure 9(b), highlights the formation of a compact and dense C–S–H gel. The pozzolanic reaction between the reactive silica in VA and calcium hydroxide generated during cement hydration results in the development of supplementary C–S–H, contributing to increased strength and durability.



**Figure 9:** VAC specimens: (a) control specimen; (b) 10% VA; (c) VA with 20% replacement; and (d) VA with 40% replacement.

The enhanced gel structure reduces porosity, while the irregular morphology and coarse surface of VA particles hinder crack propagation. As cracks encounter the non-uniform VA particles, they deviate from linear paths, leading to improved resistance to cracking and enhancing the overall structural integrity [80].

However, when the VA content exceeds the optimal replacement level, as shown in Figure 9(c) and (d), the compressive strength of the concrete decreases. This reduction can be attributed to the presence of unreactive silica, a weak interfacial transition zone, and the formation of voids in the microstructure. Excessive VA particles may absorb more water, leading to increased porosity and compromised mechanical properties.

When compared to other SCMs, such as FA or SF, VA exhibits a similar pozzolanic reaction but with distinct characteristics. FA typically offers better workability due to its spherical shape, while SF can enhance compressive strength but may reduce workability. Although VA improves durability and strength to a certain extent, higher concentrations can compromise workability, akin to the effects observed with SF. However, VA's rough texture and larger particle size compared to SF can influence its effectiveness as a filler material [81].

Additionally, the particle size of VA plays a significant role in determining the microstructural properties of the concrete. Smaller VA particles tend to exhibit higher pozzolanic reactivity, which leads to more C–S–H gel formation and increased strength. Conversely, larger VA particles may act as inert fillers, reducing the effectiveness of the cementitious matrix and increasing porosity. Achieving an optimal balance between reactivity and packing density is crucial for maximizing performance benefits.

### 3.6 TGA of VA specimens

The TGA quantified the  $\text{Ca}(\text{OH})_2$  content in the specimens by analyzing the weight loss during thermal decomposition between 100 and 800°C, as shown in Figure 10. The TGA data indicate that the controller mix exhibited the highest concentration of portlandite (C–H) compared to the further mixtures. Furthermore, specimens through a 10% volume of VA exhibit a notable decrease in the portlandite phase due to the high responsiveness of the well-amorphous silica present in the specimens. This results in increased ingesting of the C–H phase to produce more C–S–H phases in the matrix. The VA sample shows a more significant decrease in the portlandite (C–H) phase due to the presence of amorphous silica in the sample. This is due to the reactive components such as amorphous silica. When incorporated into concrete, these reactive components can participate in pozzolanic reactions with calcium hydroxide formed during cement hydration. This can lead to the formation of additional cementitious compounds, which may reduce the mass loss during TGA by contributing to the densification of the concrete matrix. Furthermore, at lower temperatures, the presence of reactive components in the VA may contribute to a reduction in mass loss due to pozzolanic reactions. However, at higher temperatures, the decomposition of VA components or interactions with other constituents in the concrete mix may lead to increased mass loss, as shown in Figure 10.

While TGA effectively quantifies  $\text{Ca}(\text{OH})_2$  and offers insights into pozzolanic reactions, it may not fully detect other phases in VA, such as unreactive silica or crystalline quartz, which can affect concrete properties. Additionally, minerals like feldspar and zeolites may not show clear

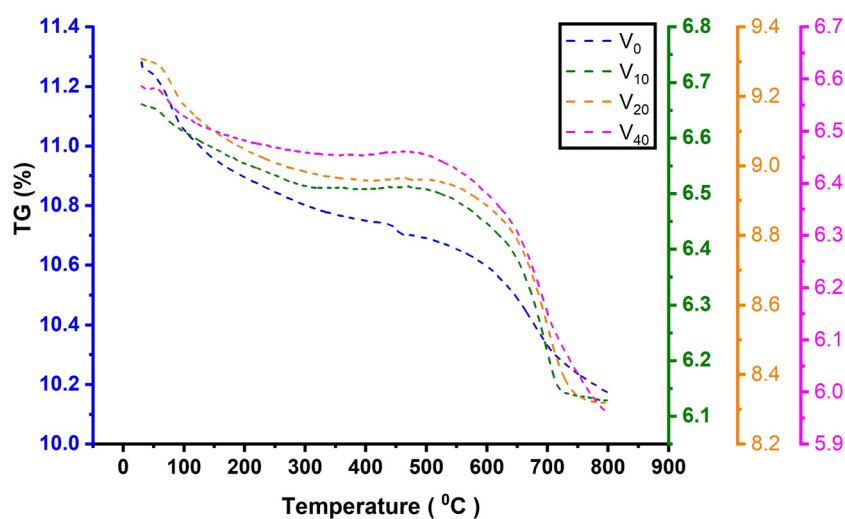


Figure 10: TGA of VA specimens in concrete.

thermal transitions in the TGA range, potentially underestimating their impact on the concrete matrix [82].

XRD complements TGA by detailing the crystalline phases in VA concrete, including portlandite, quartz, and C–S–H. However, it may not quantify amorphous phases, which are common in VA. Thus, using both techniques together is essential for a comprehensive assessment of pozzolanic activity.

From the above discussion, it can be deduced that VA concrete exhibits enhanced thermal stability compared to conventional concrete due to its lower porosity and denser microstructure, resulting from pozzolanic reactions between reactive silica in VA and calcium hydroxide during cement hydration. These reactions create additional C–S–H phases, improving the concrete's structural integrity and thermal performance.

In contrast, conventional concrete has higher porosity, making it more susceptible to thermal degradation and cracking under high temperatures. TGA indicates that VA concrete experiences less mass loss at elevated temperatures, highlighting its improved resistance to heat. Thus, VA concrete is a more reliable choice for applications exposed to high temperatures.

## 4 Conclusion

The current study aimed to assess the consequence of using ordinary VA as an additional for cement in producing environmentally friendly concrete. The impact of VA on the mechanical characteristics was investigated. XRD and TGA investigations were performed to study how VA impacts the microstructure of the specimens. Below is the conclusion reached from the experimental findings.

- The slump of the VAC decreases with increased concentration ranging from 0 to 40%. This is due to the particle size and shape, water demand, and pozzolanic activity. As the concentration of VA increases, the amount of pozzolanic reaction taking place within the concrete mix also increases. Pozzolanic reactions consume calcium hydroxide and water, forming additional cementitious compounds. This can result in a reduction in the amount of free water available for lubricating the concrete mix, leading to a reduction in slump.
- Concrete density significantly increased by substituting cement with VA, thanks to its pozzolanic activity and micro fillers. The combined impact of the pozzolanic reaction and micro filling of VA enhanced the density characteristics of concrete. A higher dose of VA negatively impacts density because of the lack of flowability. Various researches

suggest different optimal dosage adjustments based on the source of vitamin A. The normal optimal dose of VA varies from 10 to 20%.

- The use of VA as a partial cement increases the compressive strengths of the composite. This increase can be attributed to the high pozzolanic reactions in the VA with matrix. The addition of VA up to a concentration of 10% results in an improvement in compressive strength compared to the control specimen. This improvement could be attributed to factors such as pozzolanic activity, particle packing, and enhanced densification of the concrete matrix due to the presence of VA. In addition, a decrease in the matrix is observed after optimal dosage due to unreactive silica, and excessive water demand.
- XRD examination indicated that the presence of 10% VA led to a notable decrease in the concentration of calcium hydroxide, attributed to its superior pozzolanic properties associated with the other mixtures.
- Microstructure SEM analysis reveals that VA particles react with the CH to form a densified C–S–H gel. In addition, deviation of cracks is observed, which is a good sign for strength and durability.
- TGA indicates that specimens with 10% VA exhibit a notable decrease in the portlandite phase due to the high responsiveness of the very tiny formless silica present in the specimens. This leads to increased ingesting of the C–H phase to produce more C–S–H stages in the matrix.

## 5 Limitations, recommendations, and applications

This study offers significant insights into the potential of VA as an SCM, though it has several limitations. First, while the reduced portlandite phase improves durability by lowering permeability, further research is needed to understand its impact on long-term concrete performance, especially under real-world environmental conditions. Additionally, the experimental setup primarily focused on short-term performance, which may not fully reflect VA concrete's behavior over extended periods. Modifying experimental procedures to incorporate accelerated aging and environmental exposure could provide a more comprehensive view of VA's durability. A key limitation relates to the VA's mineral composition, which may vary by region and influence performance outcomes; future studies should consider this variability for broader applicability. The potential environmental implications of sourcing and processing VA should also be assessed to align with sustainability goals.



Practical applications of VA concrete are promising, particularly in sustainable construction. VA's pozzolanic activity contributes to reducing cement content, lowering CO<sub>2</sub> emissions, and enhancing material durability. This aligns with the industry's broader goals to develop low-carbon, durable building materials. Expanding this research to evaluate VA in other CCs could further contribute to sustainable construction practices. Future work could explore the environmental life-cycle impact of VA-based concrete, especially for infrastructure exposed to harsh conditions.

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