



Evaluation of the performance of concrete by adding silica nanoparticles and zeolite: A method deviation tolerance study

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ABSTRACT

Researchers have conducted extensive research to develop concretes with improved properties and suitable for harsh environments. The use of nanomaterials to improve the performance of concrete is one of the promising methods for developing optimized concrete. However, the non-uniform distribution of nanoparticles in the concrete matrix limits the reproducibility of the behavior of concretes studied, particularly in industrial or practical environments. The high surface energy of nanoparticles, limitations, and lack of standardization of the production process are responsible for this phenomenon. Addressing this challenge, this study aims to investigate practical implementation conditions of silica nanoparticle and zeolite reinforced concrete. The study examines deviations from the base method, involving synthesized silica nanoparticles via the sol-gel method as the main additive, alongside zeolite as a representative of pozzolanic materials. By examining four parameters influencing the behavior of concrete over a period of 7 days and 180 days, as well as conducting experiments in parallel groups to simulate the practical environment, this study attempted to improve the reproducibility of concrete with silica nanoparticles and zeolites improved behavior. As a result of the study, it was found that the presence of silica and zeolite nanoparticles in 7-day concrete weakens and improves the properties of the concrete by -10% and $+3\%$, respectively, for concrete after 7 days, and this effect increases by $+30\%$ and $+93\%$ for concrete after 180 days. This showed that there is a significant correlation between the healing effects of these substances and the amount of time they have been hydrated. Furthermore, concrete containing silica nanoparticles performs 35% better than the control sample. In contrast, concrete containing silica nanoparticles and zeolite provided a 165% improvement over the control sample. Using a correlation analysis of the effects of different parameters on the output performance of 7-day and 180-day concrete, this study presents a more detailed analysis of improved concrete response.

1. Introduction

Concrete, with an annual production of about 27.3 billion tons in 2015, is recognized as one of the most consumed materials in the world [1]. Its consumption is increasing to the extent that it is predicted by the year 2050, the demand for Portland cement, known as the main binder of concrete, will increase by 200% compared to 2010 and reach 6 billion tons per year [2]. This increasing demand is due to the strength, resistance, and durability of concrete against environmental destructive factors. However, since concrete is a relatively permeable material, it is still subject to environmental threats. Therefore, ensuring the high durability of a concrete sample, along with its high strength, is crucial

for increasing its useful life [3].

One of the methods used to improve the properties of concrete is the use of nanomaterials. This approach began in the late 1980s and has intensified since the 21st century [4]. One of the nanoscale additives is nano zirconia oxide (ZrO_2), which according to the report by Ruan et al. [5], can improve concrete fracture toughness by up to 400%, meaning an increase in concrete resistance against cracks and their propagation. Research results also show that adding nano titanium dioxide (TiO_2) as a filler to the cement matrix can not only create photocatalytic properties but also significantly increase the tensile and compressive strength of concrete [6]. Other nanomaterials used in cement-based composites include silica nanoparticles [7], carbon nanotubes, graphene

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nanoplatelets, polycarboxylates, nano kaolin, nano iron oxide particles, and so on [3]. Among these, silica nanoparticle is the most widely used nanomaterial in concrete due to its unique properties and availability.

Silica nanoparticles have high specific surface area and pozzolanic activity, which leads to the formation of calcium silicate hydrate (C-H-S) and increases particle density at the micrometer scale, resulting in increased strength at the macro scale [3–8]. According to the report by Zhang et al. [9], silica nanoparticles can increase the compressive and tensile strength of 28-day-old concrete by 48% and 16%, respectively. Also, due to their finer particle size compared to concrete aggregates, they have the ability to fill void spaces, reduce permeability, and increase the durability of concrete. In addition, using silica nanoparticles as an additive in cement paste can reduce the environmental costs of concrete production by reducing CO₂ emissions [10]. Various methods such as hydrothermal, microemulsion, sol-gel, etc. are used to produce silica nanoparticles. Among these, sol-gel with the ability to control particle size and morphology by monitoring the available parameters is considered as the preferred method for synthesizing silica nanoparticles [11–13]. Research also shows that the required amount of silica nanoparticles to achieve the same effect as silica fume is much lower, and this has made the use of silica nanoparticles relatively advantageous from various aspects. In the cement-water combination, silica nanoparticles have more reactivity compared to fumed silica nanoparticles [14]. Therefore, silica nanoparticles can accelerate the setting and hydration process. Thus, the presence of v can increase the compressive strength of hardened cement paste and the bond strength between aggregates and paste, and improve the transport properties more effectively than silica fume [15].

Considering the effects of adding silica nanoparticles to concrete, namely improved microstructure, reduced permeability, reduced size of voids, and increased compressive strength, it can be argued that the use of silica nanoparticles in concrete reduces the rate of chloride ion penetration. The results of research conducted by Collepardi et al. confirm this [16]. Li's research has shown that concretes containing fly ash and silica nanoparticles will have higher strength than those containing only fly ash or conventional concrete [17]. Although, several studies have been done about the effect of adding silica nanoparticles to concrete, but there is no report that uses Method Deviation Tolerance studies in this aspect.

A "Method Deviation Tolerance Study" is a type of study that considers the factors of tolerance and deviation in a manufacturing or assembly process. It aims to understand the influence of part deformation and other factors on the performance of the final product. Method Deviation Tolerance is a concept that refers to the ability of a method or process to tolerate variations or deviations from its standard operating conditions without significantly affecting its performance or output. The goal of a Method Deviation Tolerance study is to determine the range of allowable variations in a process or method while maintaining a desired level of product quality or performance [18,19].

Method Deviation Tolerance studies are often used to evaluate the robustness of a particular experimental procedure or manufacturing process. By systematically varying different parameters or conditions within the process and measuring the resulting product quality or performance, researchers can identify the range of conditions that the process can tolerate without negatively impacting its output [20]. In the context of manufacturing, a Method Deviation Tolerance study might involve testing the effects of variations in temperature, pressure, or raw material composition on the quality of the final product. By analyzing the results, manufacturers can optimize their processes to be more resilient to variations while still producing high-quality products [21]. In scientific research, Method Deviation Tolerance studies are particularly important in fields such as chemistry, materials science, and engineering, where variations in experimental conditions can have significant impacts on the outcome of experiments or the properties of materials [22].

In this research, Method Deviation Tolerance study is applied to

investigate the effect of addition of silica nanoparticles to concrete performance by considering the effects of four variable parameters on 7-day and 180-day concrete behavior: (1) the required acid volume for silica nanoparticles synthesis, (2) the mixing method of acid and synthesis precursor, (3) the amount of silica nanoparticles added to concrete, and (4) the amount of added zeolite. Furthermore, the study evaluates the correlation between time passage and additives, and its impact on concrete performance.

2. Material and methods

2.1. Material

Waterglass, containing 30% wt silica (SiO₂) and sodium oxide (Na₂O) with a silica to sodium oxide ratio of 3.4:1 (Nafis Silicate Sepahan Company), 99.8% acetic acid (Parsian Company), Portland cement, Polycarboxylate-based superplasticizer (Alborz Chemie Asia Company), and Zeolite (SiO₂: 67.2%wt, Al₂O₃: 10.0%wt, Na₂O: 4.1%wt, K₂O: 2.2%wt, CaO: 1.4%wt, Fe₂O₃: 1.2%wt) which its XRF chemical analysis result is shown in Fig S1 (Afrand Touska Company).

2.2. Synthesis of silica nanoparticles

In the first step, 282 g of waterglass was dissolved in 400 milliliters of distilled water to prepare uniform colloid. Then, X milliliters (X = 60, 90, 120) of acetic acid was added to the mixture using method Y (Y=S, D, NC, refer to the Fig. 1 and SI for more details) for gel formation. The resulting sample was left without movement for 30 min. Subsequently, the sample was subjected to a two-stage washing process and finally placed in distilled water for 12 h. The nanoparticles are separated from water and dried using an oven and furnace, respectively.

2.3. Fabrication of silica-based concrete

Initially, 2 kg of cement and 6 kg of sand were mixed for 60 s. Subsequently, 500 milliliters of water with 25 g of superplasticizer were added to the mixture, and Z grams (refer to the Fig. 1 for more details) of silica nanoparticles and T grams (refer to the Fig. 1 for more details) of zeolite were gradually added to the mixture, which was uniformly mixed for 5 min using a mixer. Then, two molds of 5 cm x 5 cm x 5 cm and 15 cm x 15 cm x 15 cm were prepared. After smoothing and tapping for 25 times to release air, the molds were covered with a wet cloth and stored in the laboratory at room temperature for 24 h. Afterward, the molds were carefully opened, and the specimens were immersed in hot water for 7 days. In parallel, a control specimen without silica nanoparticles and zeolite was also prepared. ordinary Portland cement (Type II) was utilized for producing all concrete mixes. The chemical characteristics, oxide compositions, and main chemical compounds of cement are given in Table 1. The physical properties of cement are reported in Table 2.

2.4. Data analysis

For data analysis, Python 3 was utilized along with the SciPy library to calculate Pearson's correlation coefficient between two variables. Pearson's correlation coefficient is widely used as a statistical method to measure the strength and direction of the linear relationship between two continuous variables. Furthermore, a heatmap visualizing the correlation matrix between multiple variables was created using the Seaborn library.

3. Results and discussion

3.1. Explanation of the study procedure

In this study, an attempt has been made to investigate a portion of

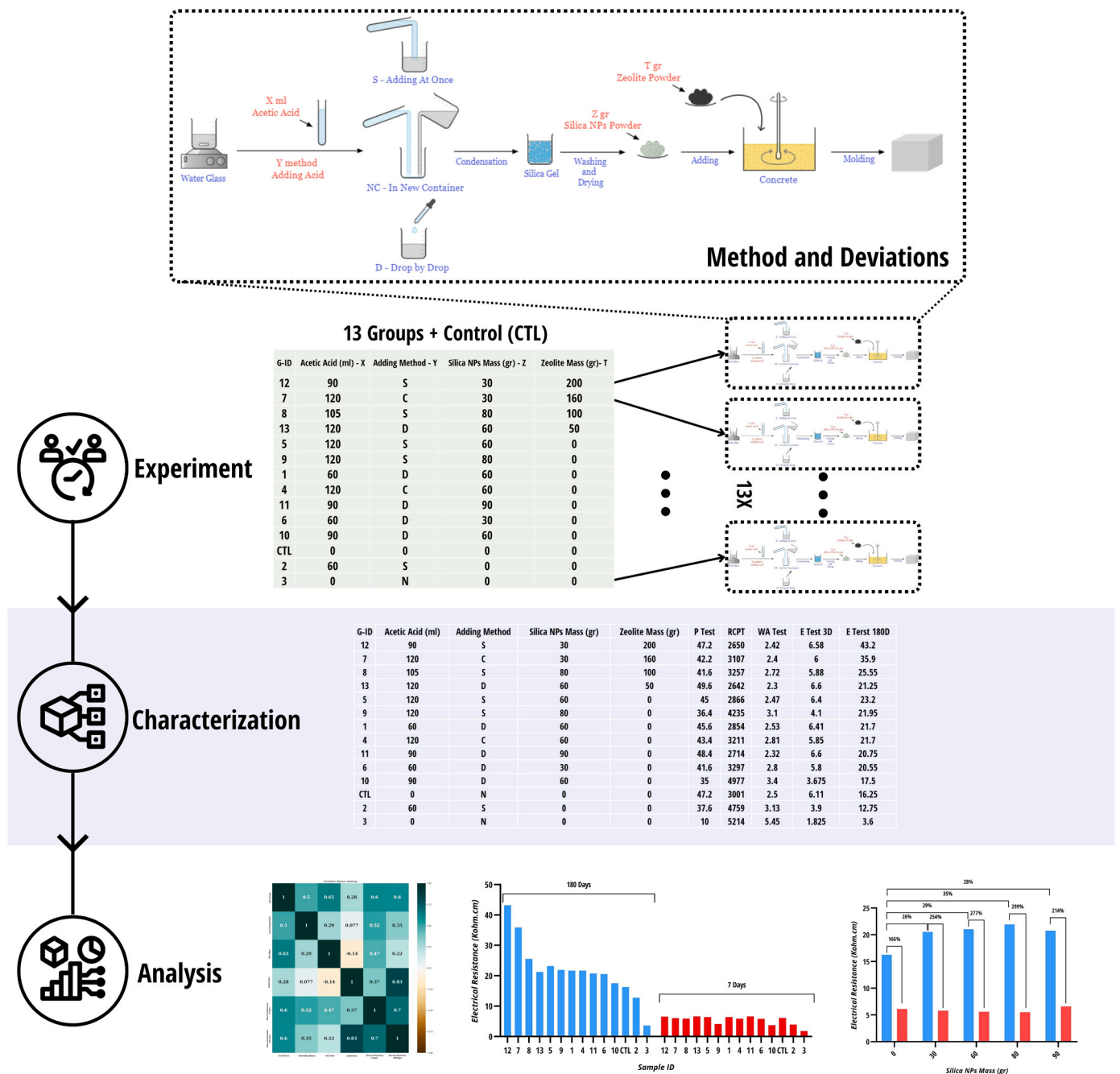


Fig. 1. The designed protocol for investigating the operational conditions of concrete reinforced with silica nanoparticles, which includes a sol-gel method for synthesizing silica nanoparticles and the graphical abstract of the study.

the operational conditions of concrete reinforced with silica nanoparticles in the form of deviations from the base method. To study this objective, a simple and an efficient protocol based on the sol-gel method for synthesizing silica nanoparticles is presented, with two variable parameters: the volume of acid required for synthesis and the method of mixing of the acid and precursors. SEM images of prepared silica particles are depicted in Fig. S2. Furthermore, in the preparation step of concrete reinforced with silica nanoparticles, two variable parameters are defined: the amount of silica nanoparticles added to the concrete and the amount of zeolite added to the base concrete as a representative of pozzolanic materials. The designed protocol for this study can be seen in Fig. 1. This study was conducted by 13 different experimental groups under the supervision of a research team based on the designed protocol, performing the experiments in parallel and collecting the required

results. The implementation of the study by different experimental groups in this study can ensure the repeatability of the results in various practical and industrial conditions, which was the main goal of this study. Moreover, the parallel use of different experimental groups can serve as a model for accelerating fault tolerance studies and improving the repeatability of research.

After the completion of the experiments, the prepared samples were evaluated by the research team for characterization and required tests in the second step. For the prepared concrete at the age of seven days, compressive strength, water absorption, Rapid Chloride Permeability Test (RCPT), and electrical resistance tests were performed (Table S1), and SEM images of the samples were taken (Fig. S3). The same samples were tested for electrical resistance at the age of 180 days, and SEM images were taken (Fig. S4). The summarized data of the tests performed

Table 1
Chemical properties and Oxide compositions of Portland cement.

Chemical properties and Oxide compositions	Test results	IQS No.5/2019 limits for OPC
Calcium Oxide (CaO)	63.3%	-
Silicon Dioxide (SiO ₂)	20.48%	-
Aluminum Oxide (Al ₂ O ₃)	4.34%	-
Ferric Oxide (Fe ₂ O ₃)	4.1%	-
Magnesium Oxide (MgO)	1.8%	< 5%
Sulfur trioxide (SO ₃)	2.3%	< 2.8%
Sodium Oxide	0.15%	-
Potassium Oxide	0.65%	-
Chlorine	0.21%	-
Loss on ignition (L.O.I)	1.32%	< 4%
In Soluble residue (I.R.)	0.52%	< 1.5%
Main Compounds (Bogue's equation)		
Tri calcium silicate (C ₃ S)	60.47%	-
Di calcium silicate (C ₂ S)	13.10%	-
Tri calcium aluminate (C ₃ A)	4.56%	-
Tetra calcium alumino ferrite (C ₄ AF)	12.48%	-

Table 2
Chemical properties and Oxide compositions of Portland cement.

Physical properties	Test results	IQS No.5/2019 limits for OPC
Initial setting time (min.)	179	> 45
Final Setting time (min.)	220	< 600
Blaine fineness (m ² /kg)	345.6	> 230
Expansion (mm)	0.2	< 10
Compressive Strength (MPa) (3 Days)	27.4	> 20
Compressive Strength (MPa) (28 Days)	43	> 42.5
Flexural Strength (MPa) (3 Days)	4.7	-
Flexural Strength (MPa) (28 Days)	6.2	-

at this stage can be seen in Fig. 1 in the characterization section.

After summarizing the reports of the experimental teams and the data obtained from the characterization step, the obtained data were examined for the performance of the concrete in the third step. To investigate the correlation of the parameters involved in the experiments, the SciPy and Seaborn libraries in the Python programming language were used to calculate the Pearson correlation coefficient and draw the correlation heat map (Fig. 1, Analysis section).

3.2. Performance assessment of 7-day concrete

For the 7-day concrete, compressive strength tests, water absorption test, RCPT, and electrical resistance test were performed. Due to the high correlation coefficient between 7-Days concrete performance and the amount of acid used in synthesis, The results of the experiments based on the variable volume of acid used in the synthesis are drawn in Fig. 2.

As seen in Fig. 2 A-D, the improvement in concrete performance behavior had a direct relationship with an increase in the volume of acid used. However, the weakness of the performance of most nanocomposite specimens compared to the control (CTL) specimen is an important point in these graphs.

Based on the observations of the experimental groups and the results obtained, several possible reasons for this behavior have been suggested, including the agglomeration of synthesized nanoparticles in the washing and drying processes and inadequate distribution in the process of making reinforced concrete, along with the incomplete hydration process of the concrete [23] as the main reasons for this observed behavior, which are confirmed by the results observed below.

As seen in Fig. 2 A, the worst performance is related to the mechanical compressive strength test, which almost all 7-day concrete specimens demonstrated weaker performance than the reference specimen, which can confirm the claim of nanoparticle agglomeration effect

in the concrete fabrication process. Other tests (Fig. 2 B-D) showed a similar behavioral trend, which will be further investigated in more detail.

3.3. Investigating correlations between characteristic tests

After completing the tests of the 7-day concrete and considering the similarity of the behavioral trends of the tests performed, Pearson correlation analysis was performed between the tests, and the results are shown in Fig. 3 A. The very high correlation (0.93, -0.97, -0.92) between the electrical resistance test and the compressive strength test (Fig. 3B), RCPT (Fig. 3 C), and water absorption test (Fig. 3D) confirms the initial observations. The negative correlation between the electrical resistance test and the RCPT and water absorption tests also matches with the concepts completely and confirms the accuracy of the results obtained. In other words, as the electrical resistance decreases, it indicates more passage and penetration of water and ions in the concrete, indicating the degree of porosity and connectivity of the voids inside the concrete as seen in Fig. S3.

Electrical resistance tests were selected as a suitable method for investigating the 180-day concrete performance based on the results of the correlation analysis and non-destructive nature of the tests. A correlation analysis was performed between the 180-day concrete test and the 7-day concrete test after performing the electrical resistance test on 180 days of concrete (Fig. 3E), and the correlation coefficient was 0.7. There seems to be a significant portion of the difference in the behavioral trend of these two samples to be attributed to the fact that the concrete has had 180 days to fully hydrate, which is a sufficient amount of time for the concrete to fully hydrate. It is also important to note that this claim is supported by the fact that zeolite is a representative of the pozzolanic materials and this material requires time before it can have an effect on the concrete's performance and needs time to complete the hydration process. It must be noted that this statement is well-supported by the fact that there is a marked difference in the behavior of the groups that used zeolite as an additive[24], [25]. In the next section, this idea will be discussed in more detail.

3.4. Comparing the performance of 7-day and 180-day concrete with the control sample

To compare the performance of 7-day and 180-day concrete, see Fig. 4A-C, different approaches were used to compare the performance of these two types of concrete with a control sample. In Fig. 4 A, all specimens were classified into three main categories: specimens containing silica and zeolite nanoparticles, specimens with only silica nanoparticles, and specimens containing silica gel (the synthesis process was stopped at the gel stage, and the drying or synthesis of the nanoparticles was not completed), and were examined with the control sample at 7 days and 180 days of age. It is evident that in this approach, other parameters involved in the experiment are variable (Fig. 1 Experiment section), but this comparison can demonstrate that zeolite and silica nanoparticles have a significant effect despite the variations in the range of variables that are used to conduct the experiment. According to Fig. 4 A, the effect of the enhancing factors has not been fully exploited at the 7-day age of the specimen. Specimens with only silica nanoparticles have shown a 10% reduction in performance compared to control samples. In 180-day concrete, on the other hand, specimens containing only silica nanoparticles were found to show a 30% improvement on average, whereas specimens containing both silica and zeolite nanoparticles were found to show a 93% improvement. The performance of all specimens containing silica gel in both 7-day concrete and 180-day concrete was significantly lower than that of the control sample in both cases. The reason for this performance reduction is that the specimens that contained gel contained voids and inclusions that the particles were unable to fill with hydration as a result of their presence [23–25]. Furthermore, because of the alkalinity of cement, when it

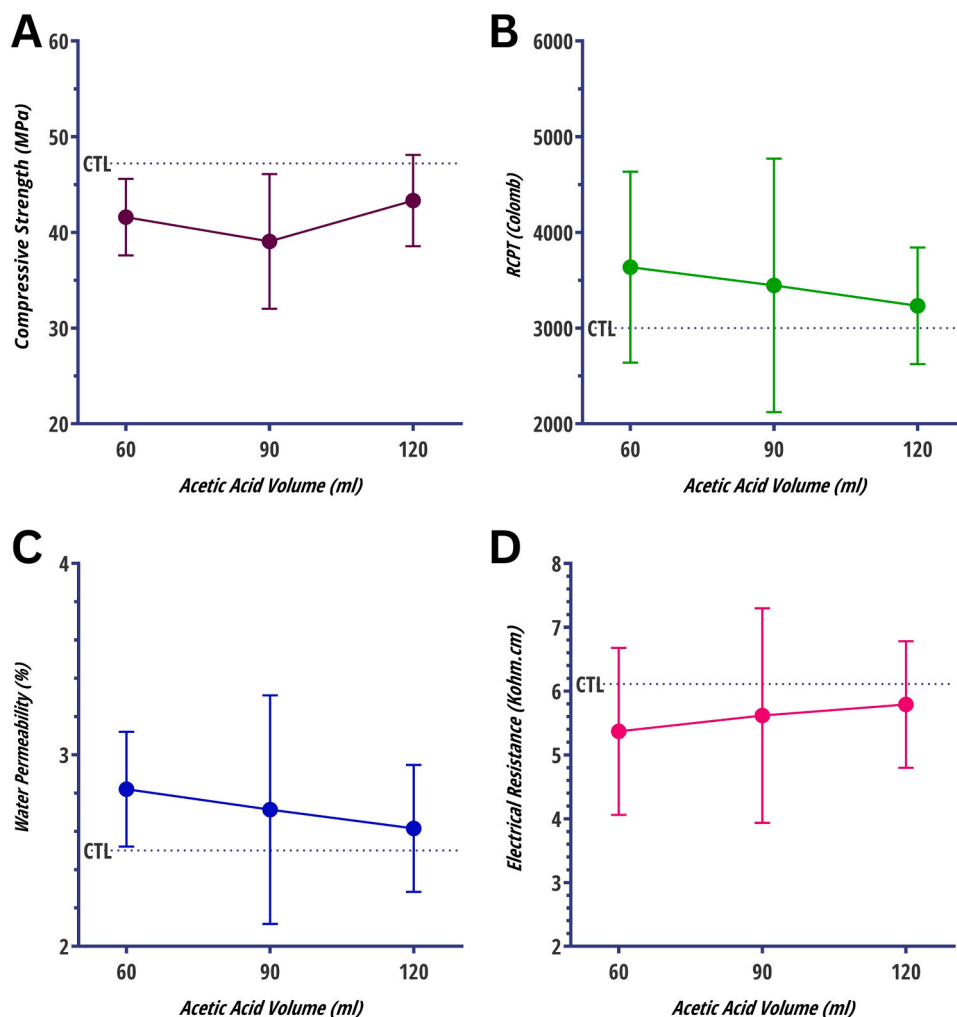


Fig. 2. 7-day concrete performance tests in comparison with control sample (CTL), A) compressive strength test, B) water absorption test, C) Rapid Chloride Permeability Test (RCPT), D) and electrical resistance test.

reacts with acidic gel, the cement particles are exposed to chemical attacks, resulting in a reduction in durability, shrinkage, and flaking of the cement as a consequence [26].

It can be seen in Fig. 4 B and C that the 7-day concrete is compared with the 180-day concrete using the approach of varying the amount of silica nanoparticles (B) and zeolite (C) in the concrete samples. There is a zero sample in both graphs which indicates that it is a control sample. Each of the 180-day concrete samples was compared to the 7-day concrete sample and the control sample, and the performance improvement was represented as an average percentage improvement in each case. Based on Fig. 4 B, it is evident that with an increase in the amount of added silica nanoparticles, the concrete's performance also increases, reaching its peak at 80 g of silica nanoparticles. Furthermore, the comparison in Fig. 4 C was based on the amount of zeolite added to the sample of the experiment. As can be seen in the graph, there is also a direct correlation between performance improvements and the amount of zeolite added to the sample, but it is evident that the effect of the zeolite parameter is much greater than that of the added nanoparticle parameter. In the following section, we will discuss this observation in greater detail in order to provide a deeper understanding. The second observation to be made in both of these graphs is the fact that time has an effect on both of these parameters as well. When 180-day concrete was compared with 7-day concrete, in Fig. 4B and C, a higher improvement in performance was observed in the samples containing zeolite, a representative of pozzolanic materials. For a complete

understanding of the effectiveness of these materials, sufficient time must be provided for hydration.

3.5. Performance of 7-day and 180-day concrete and investigation of their correlation

This section examines the relationship between four influential variables that were defined in this study and the performance indicator of concrete (electrical resistance) in 7-day and 180-day concrete (Fig. 5A-E). In the first step, the Pearson correlation coefficient was calculated in order to determine the relationship between the variables and the performance indicator (electrical resistance) from 7 to 180 days of age, and displayed as a heat map in Fig. 5A. A comparison of the results of 7-day concrete and 180-day concrete can be seen in Fig. 5B to E, based on variables such as the acidic volume used during the synthesis of silica nanoparticles, the method of adding the acid to the precursor solution, the mass of silica nanoparticles added, and the mass of zeolite added, respectively. There are dashed lines connecting these figures to the calculation of the correlation coefficient and the performance indicators (electrical resistance) at 7-day and 180-day, respectively.

According to the results obtained for the 7-day concrete, the most significant correlation was found between the volume of acid used to synthesize the silica nanoparticles and the performance indicator, which had a correlation coefficient of 0.6 according to the results obtained. For 180-day concrete, this coefficient was kept constant at the same value. It

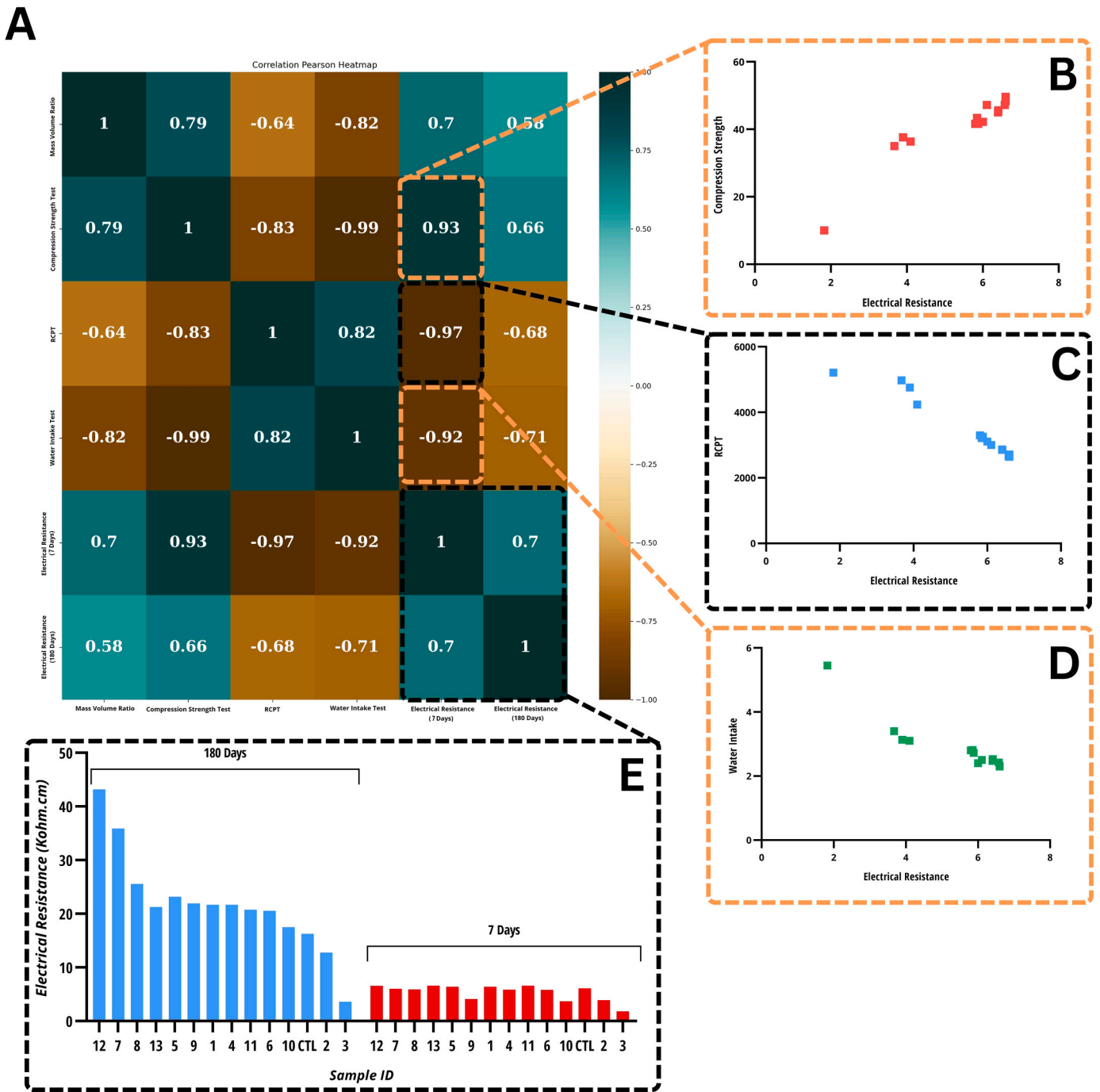


Fig. 3. Results of the correlation analysis performed between the tests conducted on 7-day concrete, A) Pearson’s correlation coefficient heat map of characteristic tests, B) the compressive strength test related to electrical test C) RCPT test related to electrical test, D) water absorption test related to electrical test, E) Comparison of the behavior of 7-day and 180-day samples.

was found that the zeolite mass used in the sample had the lowest correlation with the performance indicator in 7-day concrete, because there was not enough opportunity and time for this parameter to affect the sample, while it had the highest correlation in 180-day concrete. As a result of this increase in correlation, it can be concluded that this parameter has a significant impact on the overall behavior of the performance indicator in 180-day concrete, which confirms previous findings.

A further conclusion that can be drawn from Fig. 5C is that the most effective way to mix the acid and precursor in the synthesis process is to use the third container in the synthesis process. The difference between

the electrical resistance of the samples at 7-day and 180-day ages, based on the average electrical resistance of the samples, indicates that this method provides the highest level of efficiency due to the high level of rapid contact between the precursor and acid.

4. Conclusion

This study successfully elucidated the influential role of zeolite and silica nanoparticles on the reproducibility and performance of concrete specimens under various environmental conditions. Extensive experimentation involving multiple parameter variations and parallel testing

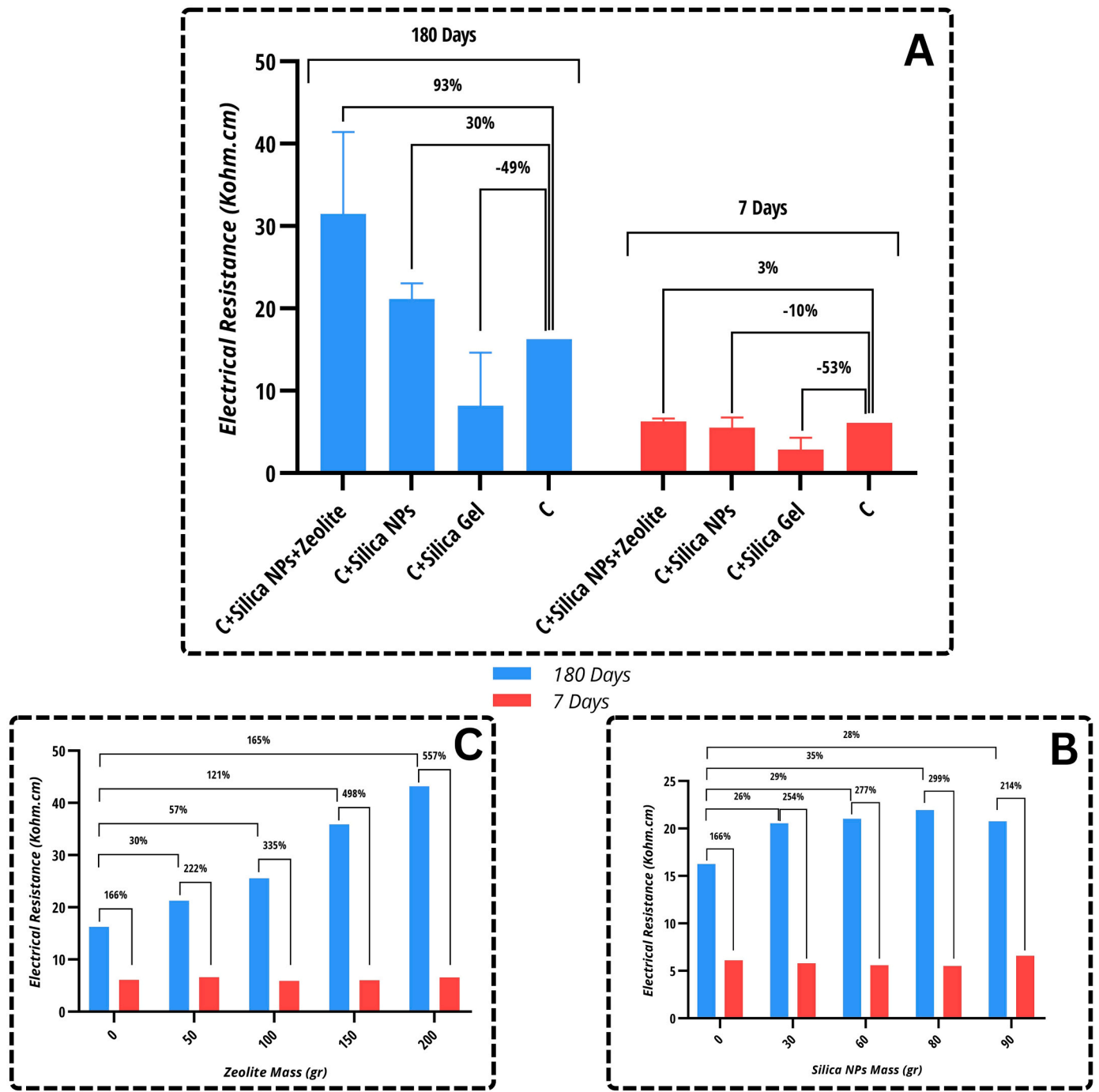


Fig. 4. (A) Comparison of 7-day and 180-day concrete samples with control samples in three groups containing silica nanoparticles and zeolite, only silica nanoparticles and samples containing silica gel B) Analyzing the performance of 7-day and 180-day concrete samples in relation to the amount of silica nanoparticles used in the samples C) Comparison of the performance of 7-day and 180-day concretes based on the amount of zeolite used in each sample.

groups helped simulate real-world implementation scenarios and ensured repeatable results.

The key findings of this work demonstrate that the inclusion of silica and zeolite nanoparticles can enhance concrete properties over time. While 7-day specimens saw performance reductions and improvements of -10% and 3% respectively, the effects were amplified in 180-day specimens, with improvements of +30% and +93%. This validates the significant impact of the hydration period on nanoparticle efficacy. Specimens containing only silica nanoparticles outperformed plain concrete by 35%. However, the combination of silica and zeolite provided the greatest enhancement of 165%. Correlation analysis revealed

the changing influence of individual parameters, like zeolite content and silica synthesis method, on 7-day versus 180-day behavior.

Overall, this research highlights the potential of silica and zeolite nanoparticles to develop more durable and resilient concrete in industrial scenarios. The robust and repeatable methodology can guide future work toward standardized production of nanomodified concrete. With continued refinement, such optimized formulations may find widespread application in infrastructure projects demanding high performance under arduous field conditions. The findings contribute valuable insights for researchers advancing the fields of construction materials and nanotechnology.

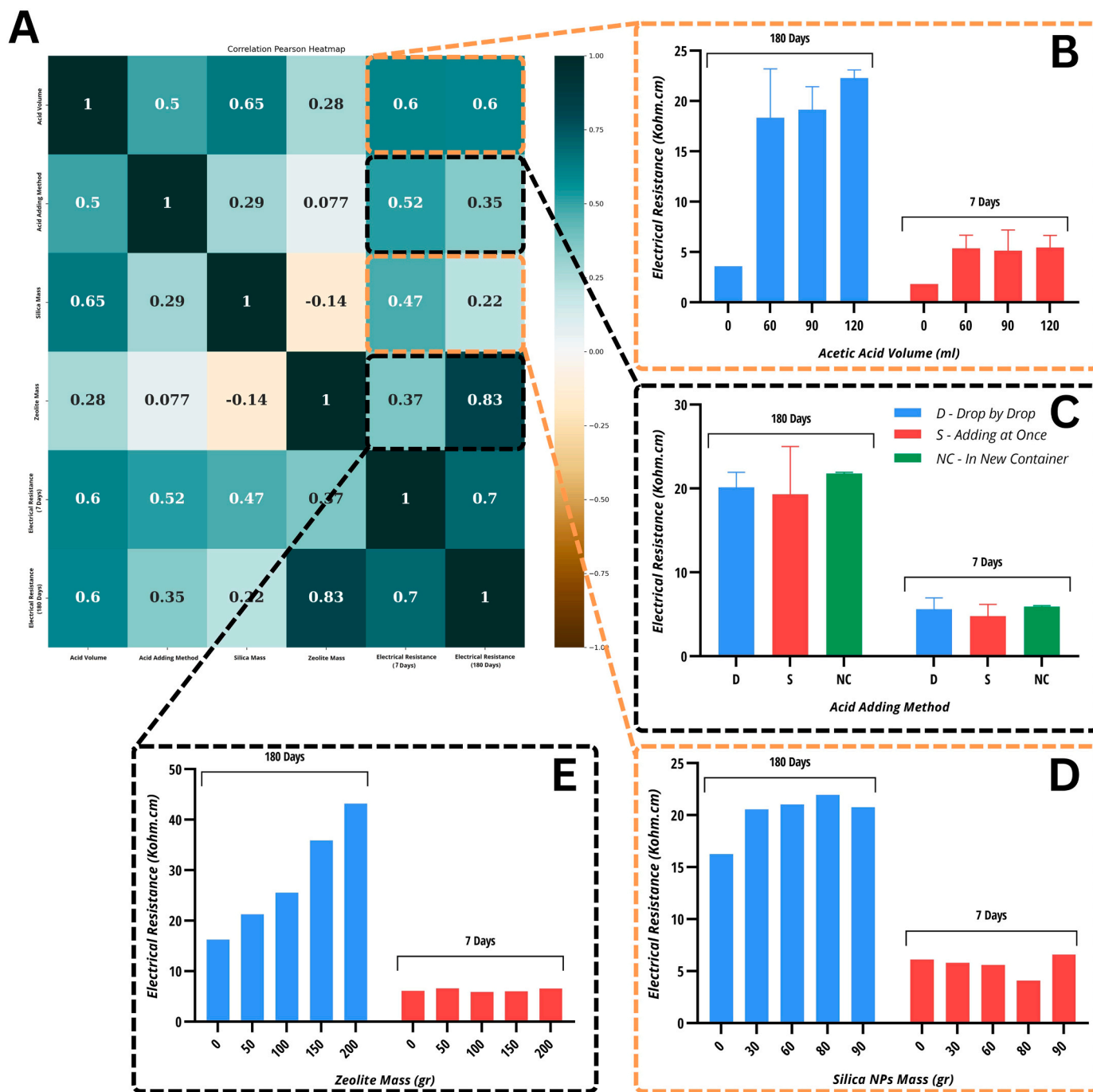


Fig. 5. Results of the correlation analysis performed between the controlled variables and performance indicator (electrical resistance) at 7-day and 180-day concrete, A) Pearson's correlation coefficient heatmap, B) Electrical resistance vs acetic acid volume, C) Electrical resistance vs acid adding method, D) Electrical resistance vs silica nanoparticles mass, E) Electrical resistance vs zeolite mass.

CRedit authorship contribution statement

Chamack Masoumeh: Writing – review & editing, Supervision, Project administration, Investigation, Formal analysis, Data curation. **Ghorbanzadeh Sadegh:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Zoghi Nasim:** Supervision, Resources, Project administration. **Mohammadi Amir Malek:** Writing – review & editing, Supervision, Formal analysis, Data curation.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.conbuildmat.2024.134962](https://doi.org/10.1016/j.conbuildmat.2024.134962).

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