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Impact of Natural Pozzolan And The Degree of Circularity of Aggregates on The Thickness, Strength and Durability Of ITZ in Concrete (Modeling and Experimentation)

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Abstract –The interfacial transition zone (ITZ) significantly negatively impacts the structural performance of concrete. This negative impact can be ameliorated by introducing mineral additions into the concrete. In this study, different forms (degree of circularity) of coarse aggregates and three contents (10%, 15%, and 20%) of mineral addition (natural pozzolan) were used to prepare ordinary concrete. This article aims to determine the optimal and effective mixture (the best form of aggregate and addition). This article aims to determine the optimal and efficient mixture (the best form of aggregate and addition) based on mechanical (compressive strength) and chemical (immersion in 3.5 % H_2SO_4 solution) tests. Furthermore, the effect of the interaction between the aggregate and the modified cementitious matrix was analyzed and discussed using the scanning electron microscope (SEM). The results show that adding 20% pozzolana improves the aggregate/matrix transition zone, making it possible to develop a maximum compressive strength at 60 days (51 MPa). In addition, the effect of mineral additions varies depending on the shape of the coarse aggregate. Thus, effective numerical modeling of the compressive strength is based on the full factorial design approach, considering the degree of circularity of the coarse aggregates and the pozzolan content.

Keywords: The interfacial transition zone (ITZ), natural pozzolan, degree of circularity, compressive strength, a form of aggregate, full factorial design.

Introduction

Concrete on a microscopic scale is a three-phase composite. It contains a mortar matrix, aggregates, and an interfacial transition zone (ITZ) between the two (Golewski, 2018). ITZ has a thickness of 10 to 50 µm (Akçaoğlu *et al.*, 2004). Its morphology, composition, density, and other characteristics are different from those of the matrix (Tasong *et al.*, 1999). The shape of the ITZ is influenced by the properties of the constituents of the concrete (coarse aggregates, cement, cementitious additions, and admixtures) and the w/c ratio of the composite. Thus, the ITZ is considered the weakest zone of concrete (Golewski, 2018). Moreover, it plays an important role in the mechanical behavior of concrete (Prokopski and Halbiniak, 2000, Ramesh *et al.*, 1996). Besides, aggregate is an important ingredient in concrete, which can be considered the skeleton of concrete. They occupy 70 to 80% of the total volume of concrete. Which strongly influences its mechanical and physical behavior (Belkadi *et al.*, 2022a, Hager *et al.*, 2016).

Researchers Xing *et al.*(2014) and Xing *et al.* (2011) showed that there is an accumulation of water at the matrix-aggregate interface. This large amount of water creates a more porous area and initiates the cracking of the concrete. Thus, the properties (size, shape, surface texture, mineralogy, and so on) of the aggregates have an important influence on the properties of ITZ (Akçaoğlu et al., 2004). Several studies have been conducted to evaluate the effect of aggregates with different shapes (rounded or irregular) and different surface textures (smooth or rough) on the properties of ITZ and concrete (Tasong et al., 1999, Li *et al.*, 2018).

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Akçaoğlu et al. (2004) observed that ITZ becomes critical for larger aggregates and low W/C ratio mortar matrices. Moreover, they concluded that the negative effect of the smooth surface texture of the aggregate and the large difference between the moduli of elasticity of the aggregate and the matrix on the properties of the ITZ is of paramount importance for concretes. Chen and Liu (2004) showed that aggregates could deflect cracks that propagate in the matrix and create a larger fracture process area.

Therefore, depending on the type of aggregate, the bond can be strengthened or weakened by chemical reactions between the cementitious matrix and the aggregate(Vázquez-Rodríguez *et al.*, 2020)(Karasin *et al.*, 2022)(Bensalem *et al.*, 2022). According to Zhou *et al.* (1995) (Kamali-Bernard *et al.*, 2014), the compressive strength of concrete is controlled by the quality of the mortar and the surface characteristics of the aggregates. Thus, the influence of the type of aggregate becomes important as the quality of the mortar is improved. Tasdemir *et al.* (1996) studied the effects of silica fume and aggregate size on the brittleness of concrete. They found that the fracture energy decreased significantly, especially for large grain sizes.

According to Yoo and Banthia (2016) (Yu *et al.*, 2016) to avoid the intrinsic strength limit of coarse aggregates, overcome the inherent weakness between coarse aggregates and cementitious matrix, increase homogeneity and eliminate stress concentration at contact points between these aggregates.The most concretes are carefully designed using good aggregates and mineral additions.Kozul and Darwin (1997) reported that concrete containing basalt aggregate gives higher mechanical properties than concrete containing calcareous aggregate, which is attributed to the intrinsic strength of the rock. Ma *et al.* (2004) reported that coarse aggregates could improve the modulus of elasticity and modify the workability of concretes more easily, as well as reduce the cost.

Tai *et al.* (2016) showed that cracks form and propagate through aggregates rapidly only at higher loading rates. Thus, the presence of coarse aggregates, Shrinkage was reduced by about 40% (Yoo and Banthia, 2016). Currently, the presence of the mineral additions is necessary to fill the voids between the aggregates in order to reduce the concentration of the contact stresses and to obtain a homogeneous distribution of the stresses through the matrix(Indrawati *et al.*, 2017)(Golewski, 2018, Nežerka *et al.*, 2019). Some research shows that the compressive strength of self-consolidating concrete (SCC) increases significantly with increasing powder content (Nikbin *et al.*, 2014).

Golewski (2018) concluded that the introduction of 20% fly ash improves the ITZ by decreasing the permeability of the concrete. Nežerka et al. (2019) showed that the presence of 20% of silica fume is effective in reducing the thickness of ITZ by increasing the mechanical performance of concrete. Khan and Alhozaimy (2011) concluded that concrete containing up to 15% natural pozzolana is equivalent to a mix containing 20% fly ash, which is very close to ordinary concrete. Kencanawati *et al.* (2020) concluded that the addition of 50% pozzolan increases the compressive strength of concretes in advanced agesDuan *et al.* (2013) showed that the addition of slag has a positive impact on the ITZ of concrete, especially for long-term hardened concrete.

Therefore, there is a need to better understand the effect of powder content and type and find an optimum amount for concrete when different coarse aggregates are used. In the present study, the effects of the shape of coarse aggregates (degree of circularity) with different surface textures and mineral addition (natural pozzolan) at different rates (10%, 15%, and 20%) on the formation of ITZ. on the mechanical performance and the durability of concrete composites have been studied experimentally and numerically. Compressive strength and durability (HCL attack) tests were carried out. To study ITZ, microstructural analyzes using SEM have been examined.

Materials and Methods

Materials

In the manufacture of concrete. Portland cement from the Ain-El-Kébira cement plant (Sétif-Algeria) is ground to a specific surface of 367 m^2/Kg. Natural pozzolan from BnieSaf was used as an addition. The chemical and physical properties of cement and natural pozzolan are given in Table 1.

In addition, to study the effect of ITZ paste-aggregates on the mechanical behavior and durability of concrete.Three sources of gravel (5/25) from different quarries in Algeria were chosen. These gravels vary mainly according to the morphological form. The Ain-touta (C1).Ziani (C2) and Tolga (C3) quarries were chosen as sources of the coarse aggregates (5/25). Silica river sand from Msila was selected (Figure 1).

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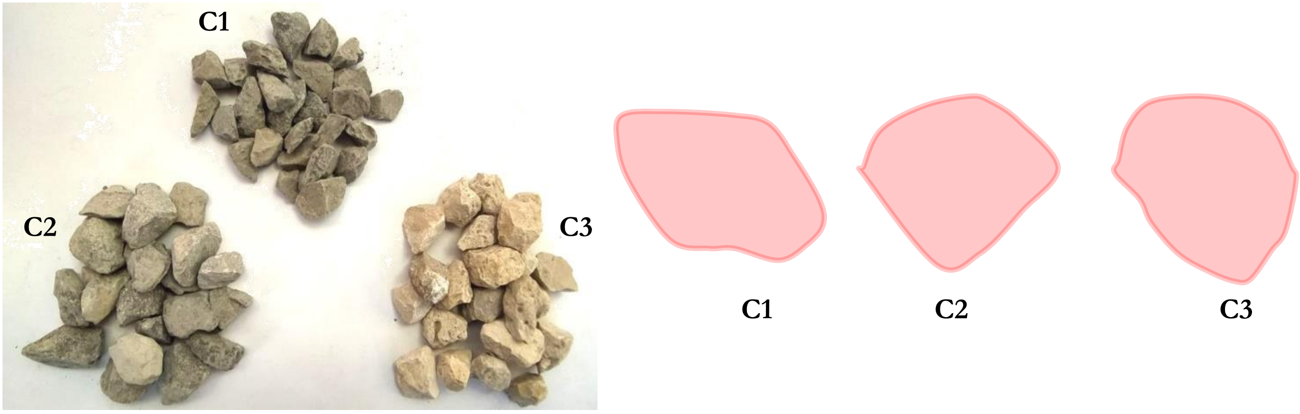

Figure 1. Different forms of coarse aggregates used.

The degree of circularity (C) is the deviation from a perfect circle calculated using the following equation: $C = (4\pi S/P*P)$. Where P and S are the perimeter and area of the two-dimensional projection of a particle, respectively(Kessal, 2017), a circular object will have a circularity value of C=1, and other shapes will have circularity values less than 1 (C<1). The morphological parameter (degree of circularity) of the aggregates is characterized by specialized image processing and analysis software called "Image j ©"(Abràmoff *et al.*, 2004) (figure 2). Thus, an average of the C values of 20 grains of each type of coarse aggregate was carried out.

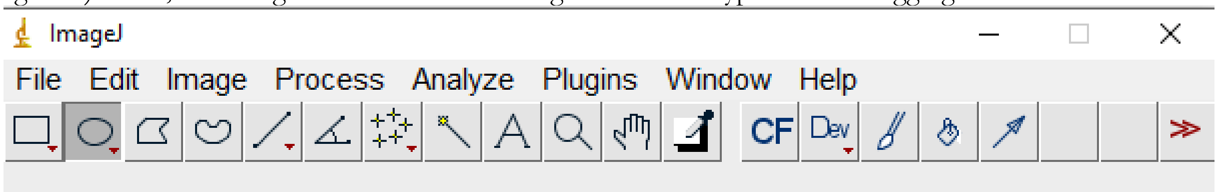

Figure 2. Main windows of Image J.

The chemical and physical properties of these aggregates are presented in Table 2. The particle size distributions of the raw materials have been illustrated in Figure 3.

Table 1. Chemical and physical characteristics of all powders used.

	Cement	Pozzolan
CaO	64.31	14.96
SiO2	21.57	39.72
Al2O3	4.37	15.76
Fe2O3	5.27	12.44
MgO	1.00	2.31
Na2O	0.24	-
K2O	0.38	1.49
SO3	1.62	0.36
P.A.F	0.98	12.95
SSB (m^2/kg)	367.0	441.70
Densité absolute kg/m^3)	3160	2700

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Table 2. Chemical and physical properties of aggregates.

	C1	C2	C 3	Sand
CaO	47.43	40.10	43.26	1.63
SiO2	7.20	13.4	10.30	90.46
Al2O3	1.29	1.02	0.09	1.38
Fe2O3	1.24	0.81	0.26	1.92
MgO	1.73	3.4	4.21	0.39
Na2O	0.58	0.09	0.05	-
K2O	0.23	0.1	0.07	0.22
SO3	1.01	0.09	1.12	0.2
P2O5	0.43	-	-	-
TiO2	0.07	-	-	-
P.A.F	38.79	40.9	41.43	2.44
Densité absolute (kg/m3)	2610	2710	2570	2550
Circularity (C)	0.72	0.73	0.76	-

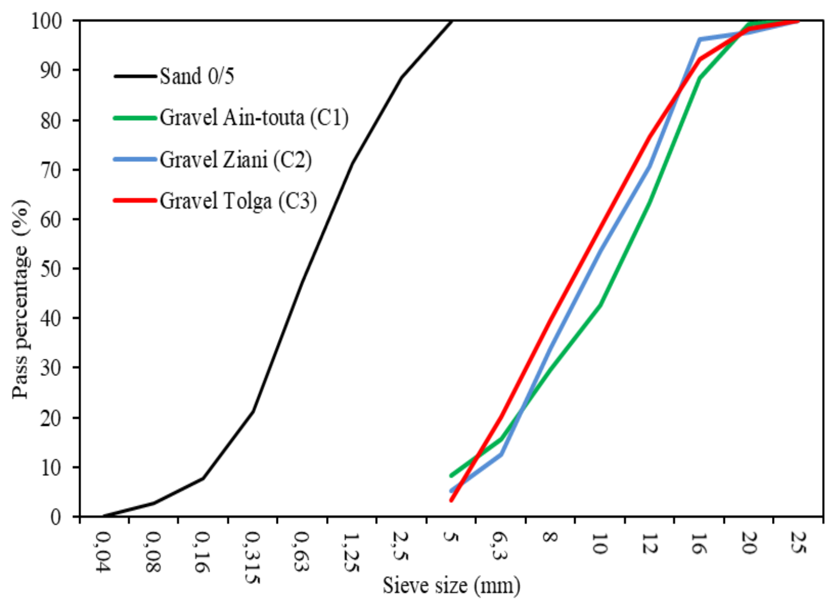

Figure 3. Sieving test results of aggregates: Particle Size Distribution.

Mixture proportions

The concretes studied in this work are made in accordance with standard NF EN 206 (EN-206, 2014). A DreuxGorisse method (Dreux *et al.*, 1983)was chosen to formulate the different concrete mixes. Pozzolan powder was used as a cement additive. With contents of 10%, 15%, and 20% based on the weight of the cement. In addition, a fixed water/binder ratio is equal to 0.53. The concretes produced were kept in the molds for 24 h. Then all the specimens were placed in water at 23 ± 2 °C until the test time.

Table 3 shows the composition of all the mixtures according to the different pozzolan contents and the three degrees of circularity (C) of the coarse aggregates.

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Table 3. Mixtures proportions.

	Mixture (Kg/m³)	Cement	Water	Sand (0/5)	Gravel (5/25)	PZ
1	C1	393	210	479	1208	
2	C2					
3	C3					
4	C10PZ1					39.3
5	C10PZ2					
6	C10PZ3					
7	C15PZ1					58.9
8	C15PZ2					
9	C15PZ3					
10	C20PZ1					78.6
11	C20PZ2					
12	C20PZ3					

Compressive strength

The compressive strength test was carried out using a device with a maximum capacity of 3000 KN. These mechanical properties were measured according to the standard (EN 12390-3)(EN-12390-3, 2009). The dimensions of the molds used are 10 × 10 × 10 mm. Three samples were taken from each mix and tested at 28 and 60 days after casting.

Chemical attack

The samples were removed from the mold after 24 hours and kept in water for 28 days, then they were kept in an air-conditioned room (50 % relative humidity and 20 °C) until a constant mass was obtained. Then they were immersed in an aggressive H_2SO_4 solution with a concentration of 3.5 %. After 60 curing, mechanical tests were used to examine the durability of the different concretes with respect to chemical attack.

Scanning electron microscope (SEM)

In order to understand the microstructure and the paste-aggregate ITZ of different formulated concretes at 60 days, scanning electron microscopy (SEM) was carried out using Hitachi S-3400N SEM apparatus.

Results

Compressive strength

Figure 4 presents the evolution of the compressive strength as a function of the C coefficient of the gravel and the content of addition of pozzolan.

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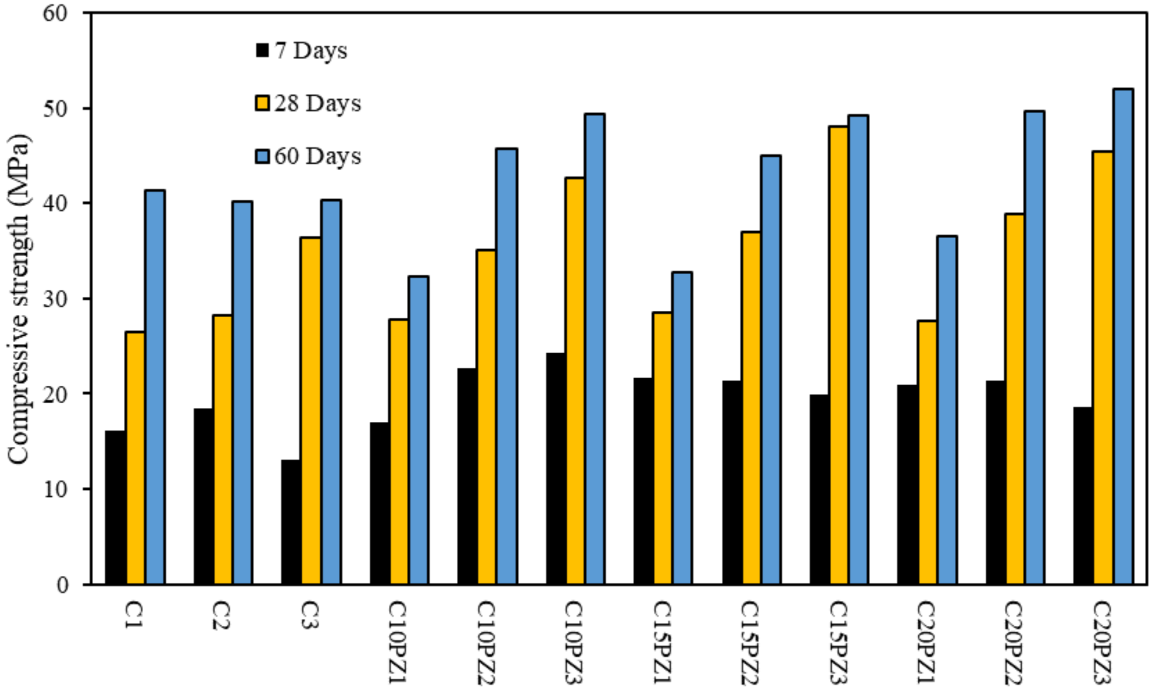

Figure 4 Compressive strength of different formulated concretes.

It is noted that the presence of aggregates with a shape close to circularity presents higher compressive strengths of concretes compared to those based on irregular aggregates. This is due to the improvement of the compactness of the mixtures by decreasing the voids in the mixture.

In addition, the addition of pozzolan with 10 % considerably increases the compressive strength for mixtures based on gravels with a high circularity coefficient. An example, the compressive strength of C10PZ2 and C10PZ3 increases by 35 % and 38.8 % compared to C2 and C3, respectively, at 7 days. This is due to the physical effect of the additions filling the voids between the aggregates and the paste. Therefore, a development of the properties of the material was noted.

At 28 and 60 days, the introduction of pozzolan causes an improvement in the strength of the concretes compared to the concretes without addition. This can be explained by the pozzolanic reaction transforming the portlandite to C-S-H (Ammar and Zeghichi, 2017). The latter fills the capillary pores in the concrete. Moreover, this improvement in strength is a function of the degree of circularity of the aggregates. The coefficient of circularity is high and presents a firmer and more solid ITZ paste aggregate. Therefore an increase in the strength of the concrete was observed(Nežerka et al., 2019).

Compressive strength modeling

In order to model the response (compressive strength), the following steps must be carried out: The first step consists in identifying the influencing factors and determining the model allowing the experimental values to be described(Belkadi *et al.*, 2022b). Thus, an analysis of variance will make it possible to dissociate the different effects studied from the variance of the response obtained(Kessal *et al.*, 2022). In addition, the results of this plan will be presented by mathematical models. These will be used to plot the iso-response curves. A factor equation based on the second-order model with a two-factor interaction was used in the form:

$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_{12} AB$ ……(1) where:

Y: is the predicted response;

β_0: *is* the coefficient constant for the offset term;

A and B: are independent factors: (degree of circularity) and addition content (%)) respectively;

β_1 and β_2: are coefficients for the linear effects of factors;

β_{12}: is the coefficient for interaction effects.

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Model variance analysis

The analysis of variance makes it possible to evaluate the contribution of the factors to the response. The model fitted in this study has a relatively high correlation coefficient of 0.94 (figure 5) and Table 4. This shows a good correlation between the response obtained by the tests and the values predicted by the model found. Thus, it can be expected that the established models are sufficiently accurate to correctly predict the effect of the presence of pozzolana powder (PZ%) and the degree of circularity (C) on the mechanical behavior of concretes. The adopted model will allow us to plot the iso-response curve according to different study factors. The analysis of variance (ANOVA) of modeled response is presented in Table 5. The total degrees of freedom (DF), the associated Sum of Squares (SS) means square, F-ratio, as well as the probability, are summarized in ANOVA.

The significance of the patterns established in this study can be assessed using the Fisher distribution test. For a 90% confidence interval, the F ratio is 45.6. From Table 5, it can be seen that the F (Fisher) ratio of the response is very high, moreover, in this context. The probability values (Prob. > F) for all models are less than 5% (p-value allows us to confirm that there is at least one significant effect of a factor in the model).
Table 6 shows the contribution of each factor (C) and (PZ%) and their interaction on the response. The $P \leq 0.05$ criteria were used to verify the significance of each coefficient. It can be seen that the effect of the factors PZ% and C and their interaction on the response is slightly significant.

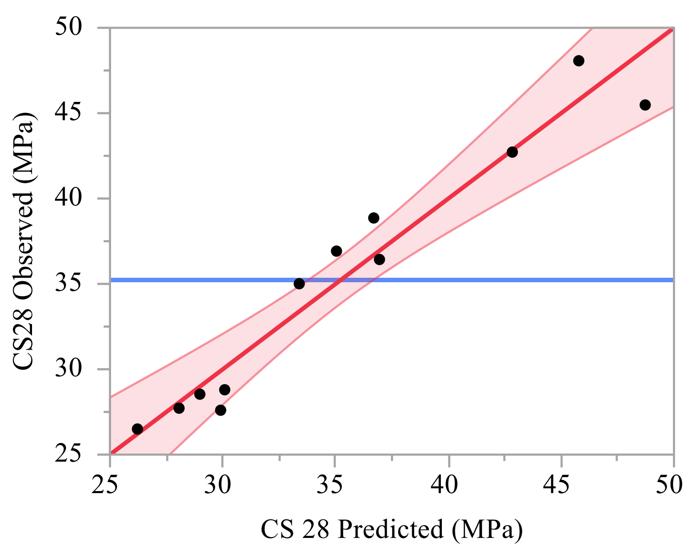

Figure 5 Correlation between the observed and predicted responses CS_{28}.

Table 4 Summary of Fit.

	SC_{28}
R^2	0.944751
Adjusted R^2	0.924033
RMSE	2.070044
Mean of response	35.22083

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Table 5 Analysis of variance (ANOVA) for derived models.

	Source	Degree of freedom	Sum of squares	Mean square	F -ratio
CS_{28}	Model	3	586.19883	195.400	45.6000
	Error	8	34.28066	4.285	Prob. > F
	Total	11	620.47949		<.0001*

Table 6 Effect test.

	Model term	Estimation	Standard Error	t ratio	Prob. > |t|
CS_{28}	Intercepts	35.447538	0.609863	58.12	<.0001*
	C	7.3867153	0.732817	10.08	<.0001*
	PZ (%)	3.8670712	0.81315	4.76	0.0014*
	C × PZ (%)	2.0228744	0.977089	2.07	0.0722

Mathematical model

Indeed, the independent variables obtained [C] and [PZ (%)] and the response [CS28] are included in the JMP 14 software(Takahashi, 2010). The model developed uses the full-factorial design approach. It is valid for a wide range of mixtures with a degree of circularity (C) ranging from 0.72 to 0.76 and a pozzolan powder filler dosage of 10 to 20%. The model provides an efficient way to calculate average responses, evaluate the effect of factors and their interactions, and provide a good prediction of the responses found. However, the models developed are based on a certain limited set of data. They can be useful in understanding the influence of each term ((C) and (PZ%)) on the behavior of concretes.

The proposed model for the prediction of studied responses CS can be written as follows:

$$CS28 = 35.447 + 7.386\left(\frac{C - 0.73}{0.02}\right) + 3.867\left(\frac{PZ\,\% - 10}{10}\right) + 2.022\left(\frac{C - 0.736}{0.02}\right)\left(\frac{PZ\,\% - 10}{10}\right)$$

With:
PZ (%): pozzolan content
CS: compressive strength
C: degree of circularity

Discussion

The different types of plots generated by analyzing the results of the compressive strength (CS) at 28 days, taking into account the factors of the content of PZ (%) and the degree of circularity (C), are presented in Figure 5. Figure 5 shows the iso-response curve of the compressive strength at 28 days of concretes made from different PZ (%) and (C) contents. Compressive strength at 28 days increases from 40.1 to 51 MPa with

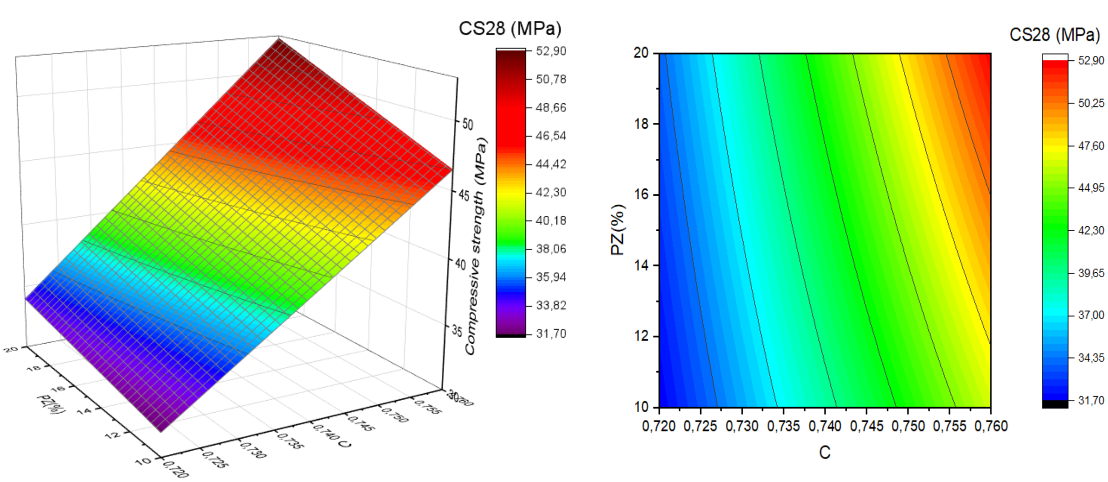

Figure 6 Iso-response curves and iso-response surfaces of the 28-day compressive strengths.

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increasing pozzolan powder content (10 % to 20 %). Thus, we can notice that the increase in C causes an increase in the resistance of concretes containing content of 10 to 20% of the PZ. These results are consistent with the estimated coefficients (Table 6) and equation (1), where the coefficients of C and PZ content are (7.38) and (3.86), respectively.

The interaction diagrams of the 28-day CS response of the mixtures taking into account the selected factors of the PZ content and the degree of circularity of different gravels (C), are presented in Figure 7. It can be noticed that the interaction graphs of the two factors are crossed, indicating that the interaction effects of the two factors on the response are significant. In other words, the effect of the C factor on the response is modified with the change in the level of the factor (PZ%).

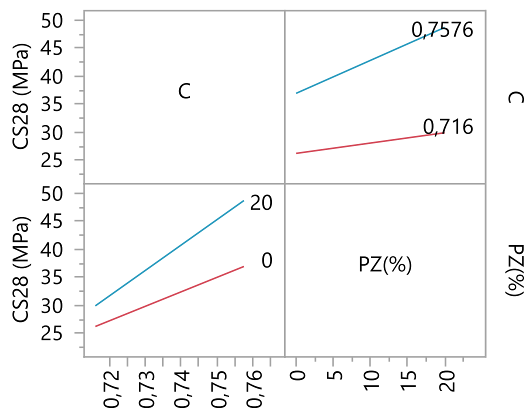
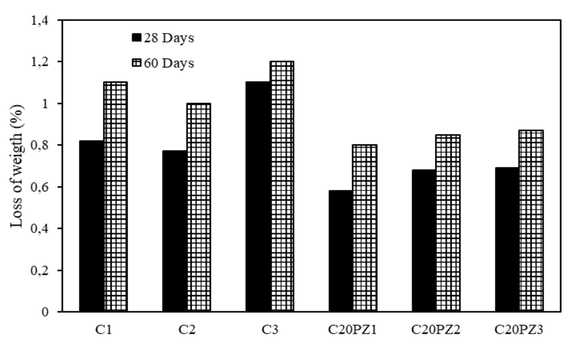


Figure 7 Interaction plot of the 28-day compressive strengths.

Durability

Figures 8 a and b show, respectively, the loss of compressive strength and mass of the different concretes immersed in H_2SO_4.



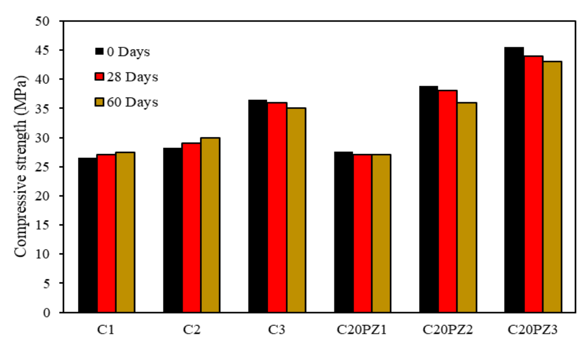


Figure 8 (a) Decrease in mass (b) Decrease in the strength of different concrete.

From Figure 8 (a and b), it can be seen that the C3 mixture had good resistance to chemical attack (H_2SO_4) compared to C1 and C2. This is probably due to the good compactness of concretes based on aggregates having a round shape. In addition, the presence of pozzolan causes a slight decrease in compressive strength for concrete immersed in the chemical attack. Knowing that the effect of the PZ is linked to the degree of circularity (C). Round aggregates containing 20 % PZ significantly improve the resistance of concretes to H_2SO_4. Microscopic tests confirm this observation (figure 9). According to Duan et al. (2013), the microstructural analysis has shown that the Si/Al-rich residues generated at the surface by the pozzolanic reaction of the slag have the potential to inhibit corrosion by acting as a barrier to acid ions.

SEM

The images captured were images showing morphology and distinct cracks in the ITZ zone. SEM observation of the cracks in the samples tested made it possible to evaluate the average width of the cracks for each type

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of concrete. Figure 9 shows the examples of representative SEM images taken for all types of composites analyzed after 90 days of curing. At this age, heterogeneous ITZ zones can only be observed in C1 concrete. The durability of concrete directly depends on the internal crack system, which decides when the material fails. The most important are the cracks appearing at the contact between the coarse aggregate and the cementitious matrix.Since it is in this zone of the composite that the destructive processes begin. In the present tests, it was shown that the largest cracks in the ITZ zone were observed in the C1 concrete. As shown in Figure 9. with the addition of pozzolan. the composition of ITZ changes. specifically, the C–S–H hydration product forms into a gel and the $Ca(OH)_2$ content decreases due to pozzolanic reactions. In addition, the fine particles fill the gaps between the matrix and the aggregates, the ITZ becomes denser and the microcracks gradually decrease, the connection between the matrix and the aggregate is improved. The porous structures (tortuosity and constriction or disconnection) have been improved with the hydration process incorporating pozzolanic additions. In addition, concretes based on large round aggregates and 20% PZ (C20PZ3) reveal a microstructure and a more uniform and compact ITZ than those based on irregular aggregates.

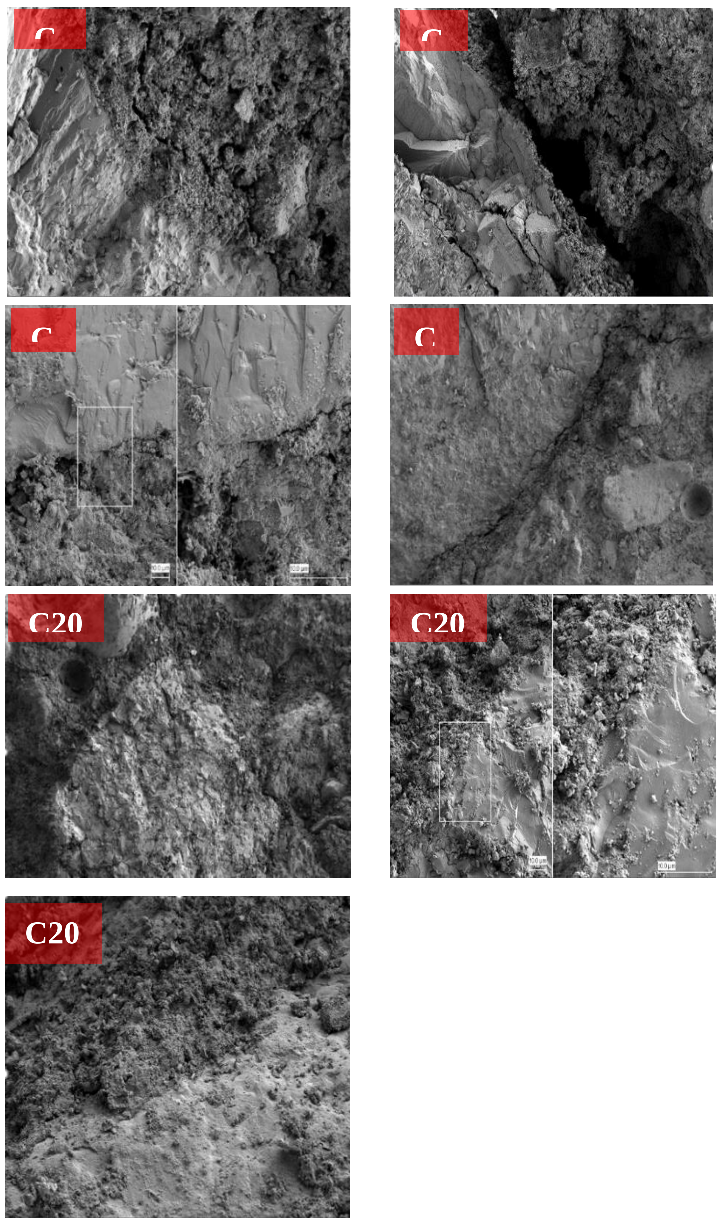

Figure 9. SEM observation.

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Conclusion

The evaluation of the microstructure. Compressive strength and durability of hydrated phases of concrete with pozzolan and 3 degrees of circularity of aggregates were presented in this article from the results obtained in this study. The following conclusions can be drawn: with the addition of pozzolana, the ITZ becomes denser, the pore structure of concrete is optimized and the pore size distribution is more reasonable. The addition of pozzolan can improve the microstructure as well as the compressive strength. 3- SEM analysis shows that the fine particles fill the gaps between the matrix and the aggregates, the ITZ becomes denser and the microcracks gradually decrease, the connection between the matrix and the aggregate is improved. 4- Concretes based on round calcareous aggregates and 20% Pozzolan show good resistance to chemical attack (H_2SO_4).

At the end in this study it is concluded that the efficiency of the reactions of the pozzolanic additions linked to the degree of circularity of the coarse aggregates used.

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