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Research paper

ONE-YEAR SULFATE ATTACK: INFLUENCE OF CERTAIN FACTORS ON THE CONCRETE PROPERTIES

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Abstract

A large amount of aggressive ions, especially sulfate ions, is present in the soil and groundwater as well as in marine environment, which could lead to the deterioration of underground and offshore concrete structures in those areas. In the last few decades, the problem of the sulfate attack on building materials and structures has attracted considerable attention and is considered very important issue. Regardless of the increased interest and research, there is always a need for further research both because of the complexity of the sulfate attack and the long-term effect itself. The aim of the research presented in this paper is to try to determine the impact of the selected factors and their combination on certain properties of concrete using a mathematical-statistical analysis "Factorial Experiment", which evaluates its sulfate resistance after one year of exposure to 5% sulfate solutions (Na_2SO_4 and $MgSO_4$). The considered parameters included in this are the type of cement (CEM I and CEM III), the water-cement ratio (0.55 and 0.38) and the type of aggregate (river and aggregate from recycled concrete), while the selected properties through which sulfate attack is monitored are capillary absorption, length change and change in compressive strength. Based on the results of the analysis, it is difficult to draw a clear conclusion about the influence level of the key factors of selected properties for evaluation the sulfate resistance of concrete mixtures for the observed sulfate exposure period of one year, but it provides a contribution to further studies in this topic.

Key words: *sulfate attack, compressive strength, length change, capillary water absorption, factorial experiment*

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1. INTRODUCTION

The rapid development of the construction industry has accelerated the consumption of natural resources and generated large amounts of construction and demolition waste (CDW). As a consequence, the enormous amount of construction waste has become a burning issue in energy and the environment [1]. Although several countries recycle around 80 % of CDW such as Japan, the Netherlands, and Germany, there are some developing countries with an average recycling rate of 20 % to 40 %. Accordingly, promoting the use of recycled aggregate (RA) from the CDW into new concrete as a replacement for natural aggregate is an essential priority [2]. Using recycled concrete aggregate (RCA) has significant environmental benefits, in addition to conserving natural resources, reducing the need for disposal of this waste and achieving sustainable development. The use of supplementary cementing materials (SCMs) also may reduce the environmental impact and energy consumption of concrete used for construction.

In recent years, researchers have begun to focus on the degradation of concrete containing RCA due to external sulfate attack. Concrete structures such as basement walls, bridge piers, ground slabs, and tunnel linings often exhibit severe scaling and spalling [3] due to the action of sulfate ions from soil and water, thereby reducing the durability of concrete. This damage due to sulfate attack can be viewed as chemical sulfate attack and physical sulfate attack. The degradation of concrete subjected to sulfate attack in the fully immersed condition is largely attributed to the chemical reactions between the ingress of sulfate and the unreacted phases and hydration phases of cements. The creation of these products is associated with the volume expansive products of ettringite and gypsum [1]. Concretes with recycled concrete aggregate have been shown to be more sensitive to the effects of sulfates than conventional concretes due to the high water absorption and the presence of adhered mortar, but this can be improved by adding, for example, SCMs, by mixing with cements that already contain mineral additives, such as CEM III or using of a lower w/c ratio [4].

Although research in the field of sulfate attack has been very intensive in the last few years, due to the large number of factors that influence the entire problem, not much is still known and more work needs to be done on this problem. This paper attempts to help in this and to contribute to the understanding of this topic and extend the life of concrete structures exposed to the aggressive attack of sulfates. Namely, an attempt was made to show, by using the mathematical tool of a factorial experiment, which of the adopted parameters (cement type, water-cement ratio and type of aggregate-natural or recycled) contributes the most to the properties through which the resistance of concrete to sulfate attack is monitored. The factors that were chosen are the change in compressive strength, the length change and capillary water absorption.

2. EXPERIMENTAL

Eight concretes were prepared and their resistance to sulfate attack was determined by immersing specimens after the specified initial curing in 5% Na_2SO_4 and 5% $MgSO_4$ solutions. The concrete specimens were completely immersed in the solutions and the sulfate solutions were changed every 90 days. Lime-saturated water was used as the reference solution. The specimens were tested according to previously determined testing program.

2.1. Components materials and mixture proportion

To assess the influence of different parameters on sulfate resistance of concrete, the following component materials were used:

- Cement: Portland cement CEM I 42.5R (Lafarge-BFC Serbia, γ_{sc}=3100kg/m^3) and Low heat/Sulfate resistance cement CEM III/B 32.5N LH/SR (Lafarge-BFC Serbia, γ_{sc}=2650kg/m^3),
- Aggregate: fine aggregate (river aggregate, 0/4 mm) and coarse aggregate (river aggregate, 4/8 and 8/16mm and recycled concrete aggregate, 4/8 and 8/16mm),
- Admixture: HRWRA ("SikaViscoCrete 3070", γ_s=1090kg/m^3 and "SikaViscoCrete 5500HP", γ_s=1090kg/m^3 "Sika"- Switzerland,) and
- tap water.

Designed compositions of concrete mixtures and their labels are shown in Table 1.

Table 1: Mixture proportions of concrete in kg/m^3

Concretes	m_c (CEM I)	m_c (CEM III)	m_v	$m_{a,f}$	$m_{a,c}$	m_{spk}	w/c
NPC1	350	-	192.5	930	858	-	0.55
NPC2	423	-	161.0	936	864	5.9	0.38
NMC1	-	338	186.0	936	864	0.7	0.55
NMC2	-	416	158.0	937	865	2.5	0.38
RPC1	350	-	192.5	20.2	874	803	-
RPC2	425	-	162	20.5	880	813	3.4
RMC1	-	338	186	20.5	881	813	-
RMC2	-	414	157	20.5	881	814	3.3

m_c-quantity of cement; m_v-quantity of water; $m_{a,f}$-quantity of fine aggregate; $m_{a,c}$-quantity of coarse aggregate; m_{spk}-quantity of super-plasticizer; w/c-water-cement ratio

2.2. Curing, specimens preparation and labels

Initially all specimens were cured in lime-saturated water for 28 days. After that period, compressive strength was determined by testing three specimens from each mixture. One third of the remained specimens were transferred to containers with 5 % Na_2SO_4 and another third in 5 % $MgSO_4$ solution where they were stored until testing period of 180 and 365 days. The last third of specimens were submerged in lime-saturated solution for the same period.

For testing of compressive strength at the age of 180 and 365 days cylinders ∅100 mm and H=100mm were chosen. The length change was measured on prism-shaped specimens with dimensions 100x100x500 mm^3 until choosen periods while capillary absorption was determined on plate-shaped specimens, the dimensions 150x150x75 mm^3, for the same time. Specimens from each mixture were categorized in three series and labelling with five letters in the following way: those with the first letter "E" were cured in lime-saturated water solution, those with "N" were immersed in 5% Na_2SO_4 solution and those with "M" were stored in 5% $MgSO_4$ solution. The second letter in the label ("N" or "R") indicates the type of aggregate (natural or recycled). The third letter refers to the type of cement: "PC" stands for CEM I and "MC" stands for CEM III. And the last letter denotes w/c ratio:"1" is for w/c=0.55 and "2" is for w/c_2=0.38.

2.3. Methods

Compressive strength was tested according to EN 12390-3 [5] before the specimens were immersed in sulfate solutions and after storing them in these solutions for choosen period. For each compressive strength, three specimens were used. Length change was determined according to the procedure given in UNI 11307 [6]. For each mixture and each solution, three prisms were measured once a week until the above-mentioned observed periods. Testing of capillary water absorption were performed on specimens before were immersed in sulfate solutions and at chosen time. Determination of capillary water absorption was done by procedure given in SRPS U.M8.300 [7]. Before testing, samples were conditioned at standard laboratory air temperature and humidity for 14 days. Surface area subjected to water was cca 225cm^2. Measurements were conducted after 1, 5, 15, 30 minutes, 1, 4, 9 and 25 hours. The results were expressed as water absorption in kg/m^2 and presented values are average of three specimens.

3. RESULTS AND DICSUSSION

3.1. Compressive strength

The average results of concrete compressive strength after 180 and 365 days of immersion in $MgSO_4$, Na_2SO_4 and lime-saturated water are presented in Figure 1a for mixtures with NA and in Figure 1b for the mixtures with RA.

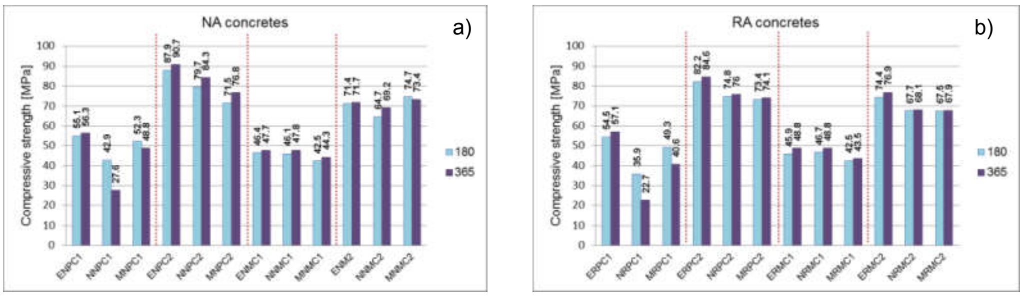


Figure 1. Compressive strength of concrete mixtures at 180 and 365 days a) NA concrete mixtures; b) RA concrete mixtures

Based on the results shown in the Figure 1 it can be concluded that almost all concretes have a decrease in compressive strength or a slight increase compared to the corresponding reference specimens, for both ages, but this is within the limits of up to 10%.

The exception is concretes with CEM I and w/c=0.55 for both types of aggregates, where is this difference greater.

3.2. Length change

Length change results of reference concrete specimens as well as specimens immersed to Na_2SO_4 and $MgSO_4$ solutions after 180 and 365 days are shown in **Error! Reference source not found.**2.

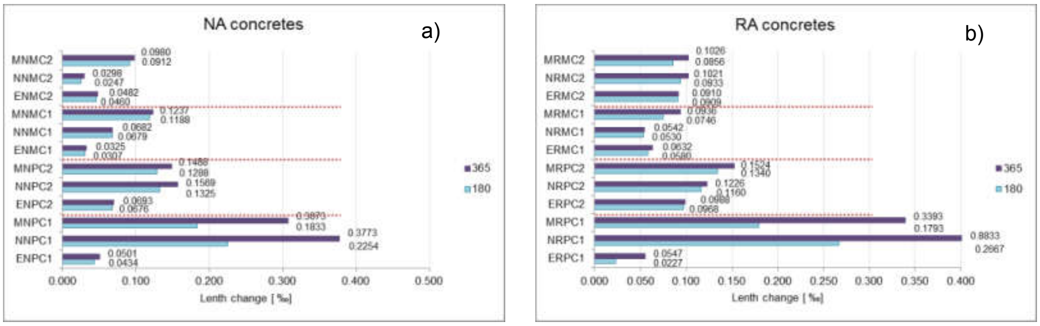


Figure 2. Length change of concrete mixtures at 180 and 365 days a) NA concrete mixtures; b) RA concrete mixtures

Based on the measured values of the length change of specimens exposed to sulfate solutions after 180 and 365 days, it can be concluded that the dominant length change is observed in concrete mixtures with CEM I and w/c=0.55 in both sulfate solutions. All other concretes have a length change within acceptable limits.

3.3. Capillary water absorption

Results of capillary water absorption at 25 hours in kg/m^2 on all observed specimens after 180 and 365 days immersed in sulfate solutions are presented in Figure 3.

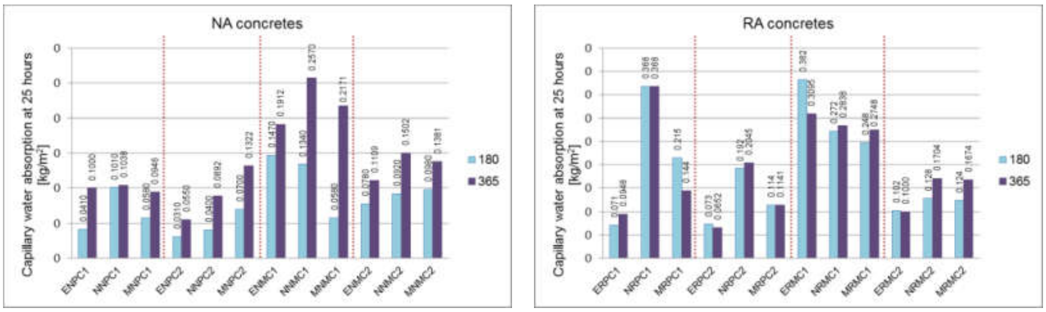


Figure 3. Capillary water absorption of concrete mixtures at 180 and 365 days a) at NA concrete mixtures; b) at RA concrete mixtures

It is difficult to find any regularity in this property, but it can be seen that all concretes with NA have higher capillary absorption values after 365 days, while this is not the rule for concretes with RA.

3.4. Factorial experiment

The influence of selected factors such as the type of cement, water-cement ratio and type of aggregate used in this experiment, as well as their combination, on certain properties of concrete determined after 180 and 365 days immersed in sulfate solutions was carried out using the "multi-parameter" analysis "Factorial experiment", which basically consists of appropriate mathematical and statistical procedures with the aim of obtaining a mathematical model of the process.This type of analysis enables the verification of the hypothesis about the existence of a relationship between the observed properties of concrete and various influencing factors. Factorial analysis can be used to define the relative size of the influence of various parameters, and based on their "significance" the simplest analytical form can be formulated. Each influential factor (type of cement, water-cement ratio and type of aggregate) is assigned a level of consideration that includes values of +1 or -1[8].

The results of the analysis of the influence of the mentioned parameters on the compressive strength are given in Figure 4 according to time exposure and solutions type.

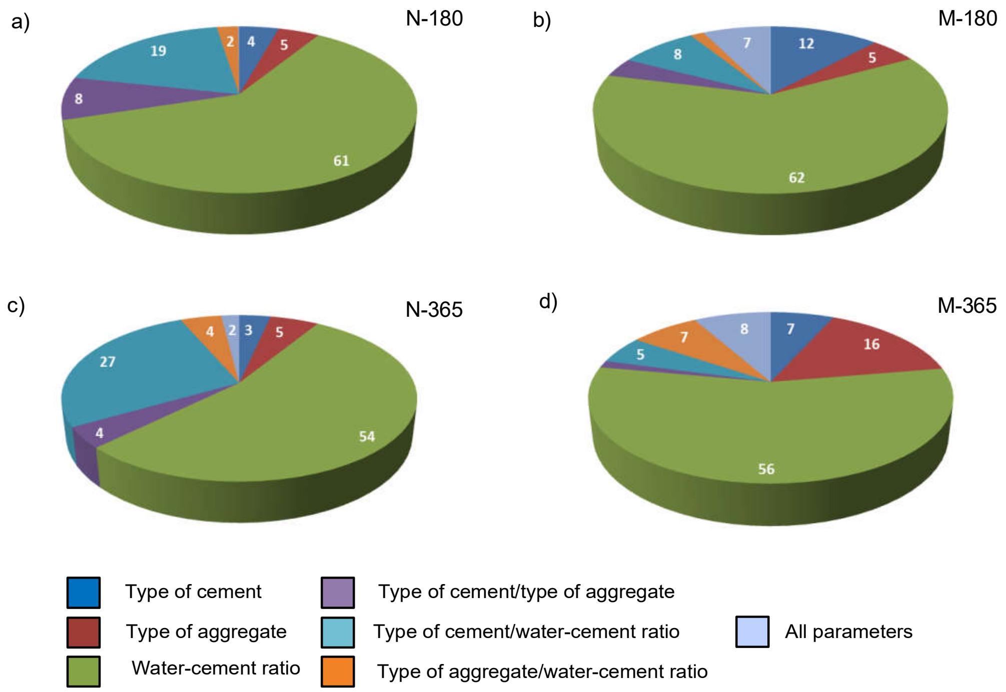


*Figure 4. Results of analysis using a factorial experiment in the case of **compressive strength** a) 180 days immersed in Na_2SO_4, b) 180 days immersed in MgSO4, c) 365 days immersed in Na_2SO_4, d) 365 days immersed in MgSO4,*

Based on the results of the compressive strength analysis (Figure 4), the water-cement ratio is by far the dominant parameter in all cases, regardless of the time of exposure to the sulfate solution or the type of solution. The next parameter that has an impact on this observed characteristic is the combination of cement type/water-cement ratio, in specimens exposed to Na_2SO_4, while in the case where the specimens are exposed to $MgSO_4$ cement (at 180 days) or aggregate type (at 365 days).

The results of the analysis of the influence of the choosen parameters on the length change of the specimens are given in the Figure 5.

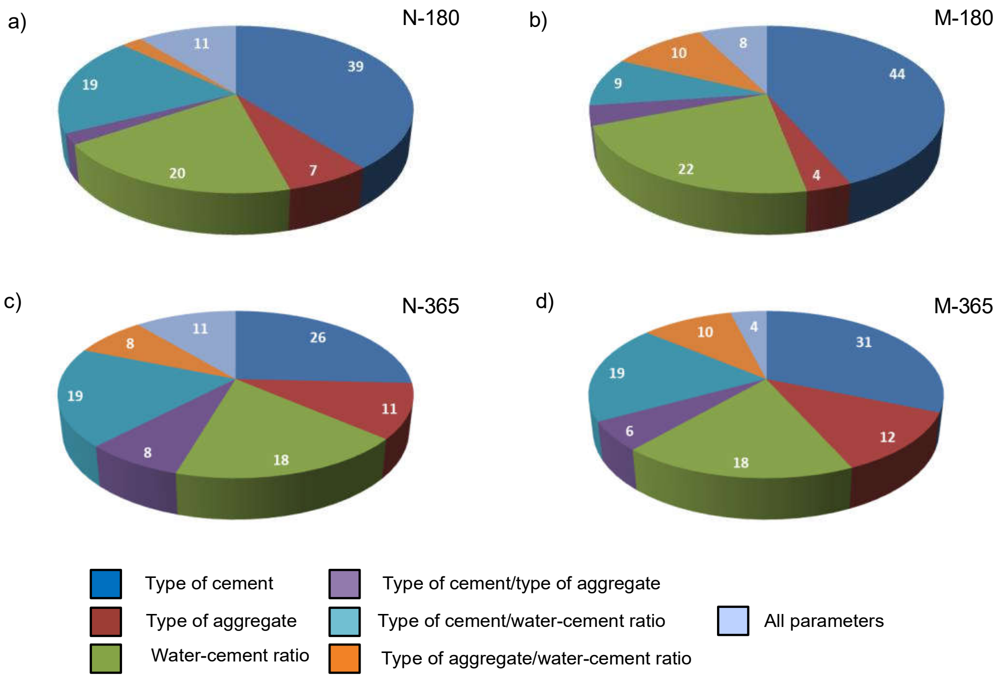
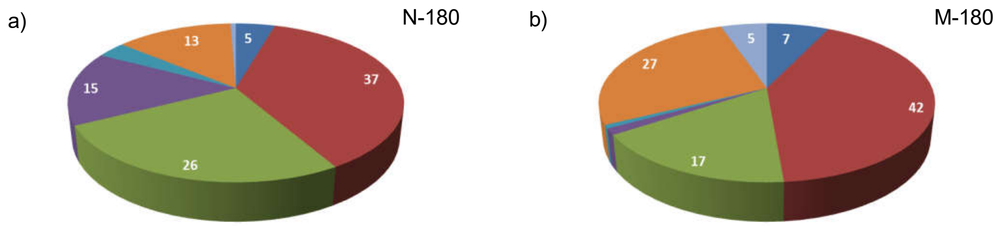


*Figure 5. Results of analysis using a factorial experiment in the case of **length change** a) 180 days immersed in Na_2SO_4, b) 180 days immersed in MgSO4, c) 365 days immersed in Na_2SO_4, d) 365 days immersed in MgSO4,*

The results obtained and presented in the Figure 5 indicate that in the case length change, the dominant parameter is the cement type (more in the case of 180 days), followed by the water-cement ratio or the combination of cement type/water-cement ratio (at 365 days).

The results of the analysis of the influence of the mentioned parameters on the coefficient of capillary absorption are given in the Figure 6 for both ages and both solutions.



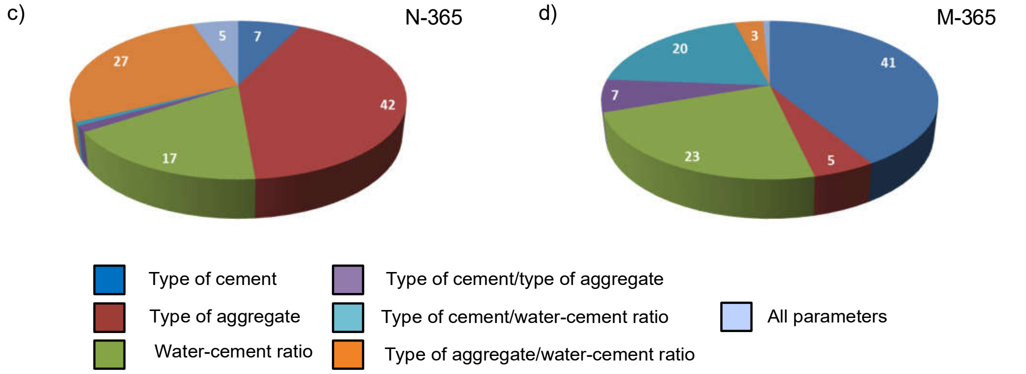


*Figure 6. Results of analysis using a factorial experiment in the case of **coefficient of capillary absorption** a) 180 days immersed in Na_2SO_4, b) 180 days immersed in MgSO4, c) 365 days immersed in Na_2SO_4, d) 365 days immersed in MgSO4,*

Based on the results shown in Figure 6, it is difficult to draw a single conclusion, but some rules can be observed. Namely, in the case of exposing specimens to the both solutions for 180 days and the Na_2SO_4 solution for 365 days, the parameter that has the greatest influence on the observed property is the type of aggregate, followed by the water-cement ratio in the case of Na_2SO_4 or the combination of aggregate/water-cement ratio. When we consider a period of 365 days of exposure in the $MgSO_4$ solution, the dominant parameters are completely different. Thus, the type of cement is dominant, followed by the water-cement ratio.

5. CONCLUSION

Based on the analysis conducted on the magnitude of the influence of selected parameters on certain properties of concrete exposed to sulfate attack, the following conclusions were drawn:

- **Compressive strength**: in all observed cases (both sulfate solutions, Na_2SO_4 and $MgSO_4$, and both ages of 180 and 365 days), the water-cement factor has by far the dominant influence. After it is the type of cement in the case of Na_2SO_4 solutions, regardless of age, while in the case of $MgSO_4$ solutions, it is the type of aggregate or the type of cement.
- **Length change**: in all observed cases, the dominant influence on the observed characteristic is the type of cement. It is followed, with a small difference, by the combination of parameters cement/water-cement factor in all cases except for the $MgSO_4$ solution for 180 days.
- **Coefficient of capillary absorption**: the parameter that has the greatest influence on this observed property in all cases is the type of cement except in the case of $MgSO_4$ and age of 365 days. The parameter that follows this dominant one is variable for all observed cases.

As can be seen, it is difficult to make a single attitude and conclusion about the dominant influences of certain parameters in the case of observing the resistance of concrete under the attack of 5% Na_2SO_4 and $MgSO_4$ after 180 and 365 days and that they depend on both the observed characteristic and the type of solution and the exposure time. This is certainly a move towards understanding the behavior of concrete under sulfate action and further work should be done to study and increase the resistance and durability of concrete exposed to this aggressive influence.

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