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Classification of fine natural aggregates from Poland depending on alkali reaction according to the procedures of the General Directorate for National Roads and Motorways

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ABSTRACT

The alkali-silica reaction (ASR) poses a serious threat to the durability of concrete structures. Although the mechanism of this reaction was described as early as the 1940s, assessing the potential alkali reactivity of aggregates is still a research and engineering challenge. This article discusses the classification of the alkali reactivity of fine natural aggregates based on the results obtained by methods PB/1/18 (accelerated) and PB/2/18 (long-term), developed in accordance with the guidelines of the General Directorate for National Roads and Motorways. In the experimental part, the authors analysed fine natural aggregates previously used in concrete structures that showed no signs of damage due to ASR. The study confirmed the presence of reactive forms of silica, including deformed and polycrystalline quartz. Significant differences in alkali reactivity classification results were shown depending on the method used, confirming the limitations of the accelerated method and the possibility of erroneous results. It emphasised the need for careful interpretation of short-term test results and the need for further refinement of aggregate reactivity assessment methods to achieve better correlation with long-term methods, which more accurately reflect the service conditions of concrete.

Keywords: alkali-silica reaction, fine aggregates, concrete durability, reactivity classification.

INTRODUCTION

The reaction between the reactive forms of silica in the aggregate and the alkalis from concrete components (ASR) was first described by Stanton in the 1940s. He discovered that certain minerals in the aggregate, sourced from the California coast, reacted with sodium and potassium hydroxides in the cement, leading to concrete expansion[1]. Although more than 80 years have passed since the first description of the alkali-silica reaction in concrete, the complex mechanism of the processes involved means that ASR-induced concrete corrosion is still an ongoing challenge for modern concrete production. In order to minimise the risk of ASR in concrete, it is critical to properly classify aggregate reactivity. To this end, a number of methods have been proposed and standardised around the world. Of particular note are the methods developed by the American Society for Testing and Materials (ASTM), which has introduced several test procedures, including: ASTM C295: Standard Guide for Petrographic Examination of Aggregates for Concrete [2], ASTM C289: Standard Test Method for Potential Alkali-Silica Reactivity of Aggregates (Chemical Method) [3], ASTM C1260: Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method) [4], ASTM C227: Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method) (Withdrawn 2018) [5], ASTM C1293: Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction [6].

Although these procedures are widespread and used in many countries, they have important limitations. The test methodology proposed in ASTM C1293 is considered to be the most accurate for classifying the potential alkaline reactivity of aggregates due to its greatest degree of correspondence to real-world conditions, but the one-year testing time for concrete bars made from the tested aggregate makes it unsuitable for ongoing quality control. For this purpose, an accelerated test procedure was proposed, as described in ASTM C1260, involving measurements of the linear changes of bars made of mortar that are stored in 1M sodium hydroxide (NaOH) solution at 80 °C. The mortar is prepared from aggregate of a given grain size. The classification of the alkaline reactivity of the aggregate is based on the linear changes of the bars after 14 days of storage. The method is particularly useful for aggregates that react slowly [7]. Studies have shown that the fineness of the analysed aggregate can alter its reactivity. Increased surface area of the reaction and access to sodium and potassium ions may have an impact on false-positive results [8]. It is also recognised that the high temperature of the ASTM C1260 test does not reflect actual conditions. Douglas analysed the composition of the pore fluid at 80°C and found an increased proportion of SO_4^{2-} sulphate ions, and it was also noted that the concentration of OH^- ions decreased at this temperature and that the concentration of Ca^{2+} was the highest, which significantly altered the reaction system. This can translate directly into the value of the measured expansion [9]. In turn, Feng [10] analysed 40 fine and coarse aggregates from the USA in his study. He showed that the ASTM C1260 method could lead to false-positive or false-negative results in up to 72% of cases.

Currently in Poland, the methodology for assessing potential reactivity is not standardised. In the past, alkali-silica reactivity analysis of aggregates was carried out according to standards PN-92/B-06714/46 [11] and PN-91/B-06714/34 [12], but these have been withdrawn, without replacement. In 2016, the Institute of Ceramics and Building Materials in Kraków worked together with the Institute of Fundamental Technological Research of the Polish Academy of Sciences to develop the criteria for assessing the reactivity of domestic aggregates and indicate technical guidelines for the design of long-lasting concretes, with particular attention paid to protecting structures from damage caused by the alkali-aggregate reaction. The potential alkaline reactivity of aggregates is determined based on tests performed according to the following procedures: PB/1/18 – accelerated method based on ASTM C1260 standard guidelines together with RILEM AAR-2, PB/2/18 – long-term method based on ASTM C1293 standard guidelines together with RILEM AAR-3, PB/3/18 – petrographic analysis [13].

The aforementioned project analysed, among other things, the reactivity of 17 fine aggregates of different origins: 7 of these were classified as R1 – moderately reactive aggregate, the remainder as R0 – non-reactive aggregate. Jóźwiak-Niedźwiecka [14] carried out alkali-silica reactivity tests on 18 natural sands mined in Poland, according to procedures PB/1/18 and PB/3/18 of the General Directorate for National Roads and Motorways. Of the sands tested, 33% were classified as moderately reactive, while 67% were considered non-reactive. The authors emphasise the impact of the origin of the sand on its susceptibility to alkaline reaction, indicating the need to take local geological conditions into account when designing durable concretes. Naziemiec [15], in turn, carried out tests on domestic fine aggregates using the accelerated method, in accordance with the ASTM C1260 standard. The results showed that more than half of the aggregates tested showed moderate alkaline reactivity. The author draws attention to the considerable variability in the quality of aggregates in terms of their mineral composition and the low reliability of the results obtained using the ASTM C1260 method in relation to actual service conditions.

It should be noted that there are few papers available in the literature that present the results of studies of the alkaline reactivity of fine natural aggregates from Poland using long-term methods. In this article, the authors classified fine aggregates with regard to their susceptibility to ASR using the methods indicated in the Technical Guidelines of the General Directorate for National Roads and Motorways and categorised them. The research focuses on demonstrating possible differences in the reactivity classification of aggregates with consideration of the fact that all aggregates have previously been used for concrete under real conditions and have not shown signs of destructive alkaline corrosion. The study was extended to include analyses of the mineral composition of the aggregate, which was referred to the extent of corrosion processes, defined by the degree of expansion measured using the accelerated and long-term methods.

RESEARCH MATERIALS AND METHODS

Cement

CEM I 52.5R cement with an alkali content expressed as Na_2O_{eq} equivalent of 0.86% was used in the tests. The alkali content meets the requirements of the test procedures. The chemical composition of the cement is presented in Table 1.

Aggregate

Granite fractions 2/8 and 8/16 were used as non-reactive coarse aggregate. Reactivity category R0 was confirmed by tests according to the procedures of the General Directorate for National Roads and Motorways No. PB/1/18, PB/2/18 and PB/3/18. The analysed fine aggregates were four natural aggregates from Poland, with a grain size of 0/2 mm whose suitability for the production of road and bridge concrete has been confirmed by many years of experience.

Petrographic analysis of aggregates

The mineralogical composition of the fine aggregate and the type of reactive silica forms and their percentage is presented in Table 2. The petrographic analysis of the aggregate was carried out in accordance with the guidelines of procedure PB/3/18. The tested specimens were reduced in size and used to prepare microscopic preparations for observation under polarised transmitted light according to PN-EN 12407 [16]. Aggregate observations were carried out using an Olympus BX-51 polarising microscope. A x5 lens and a x10 lens were used during the study. A planimetric analysis was performed in a grid with a pitch of 0.4 mm (number of counts >1500).

Accelerated mortar expansion test method

Classification of the reactivity of the fine aggregate according to the accelerated method was carried out in accordance with procedure PB/1/18. The tested aggregate was used to produce mortar for which the w/c ratio was set at 0.47 and the cement content was 440 kg/m^3. Three bars with a size of 25 × 25 × 285 mm were then made from each batch. The specimens so formed were placed in a humidity chamber with a constant temperature of 20 ± 2 °C and a humidity of not less than 95% RH. After another 24 h, a baseline measurement was taken, and the specimens were subsequently immersed in a 1 molar (1 M) sodium hydroxide (NaOH) solution at 80 ± 2 °C for a period of 14 days. After 24h, the specimens were unmoulded, and an initial reading was taken, after which the specimens were placed in containers filled with distilled water in a thermostatic chamber, at 80.0 ± 2°C for a period of 14 days, with measurements of the linear changes in specimen length after 3, 7, 12 and 14 days. The changes in the length of the tested specimens, represented by the average expansion of the three mortar specimens, are presented in Figure 2a.

Concrete expansion test method

For the assessment of the reactivity of the aggregates according to the long-term method, the guidelines of procedure PB/2/18 of the General Directorate for National Roads and Motorways were followed. Three 75 × 75 × 285 mm bars were prepared for each of the analysed aggregates. The concrete mixture was made of CEM I 52.5R cement in an amount of 420 kg/m^3 and the tested fine aggregate combined with non-reactive coarse granite aggregate, maintaining the aggregate grain size required by the procedure. The w/c ratio was 0.43. In addition, to meet the requirements of procedure PB/2/18, an additional amount of NaOH was dissolved in the mixing water so that the Na_2O_{eq}. content per cement weight was 1.25%. A superplasticiser in a quantity of 4.5 kg/m^3 was used to ensure the concrete mix was workable. After the specimens had been stored for 24 hours at 20 ± 2 °C, RH ≥ 95%, an initial measurement was taken. The specimens were then placed in containers with distilled water (up to 25-mm high), in an atmosphere with 95% relative humidity, and stored for 365 days

Table 1. Chemical composition of cement

Type of cement	Oxide composition [%]								
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
CEM I 52.5R	19.2	5.05	3.01	64.0	2.1	3.48	0.26	0.92	1.98

in a thermostatic chamber at 38 ± 2 °C. The linear changes of the specimens were measured at 7, 14, 28, 91, 182 and 364 days.

RESULTS OF THE STUDY

Petrographic analysis

The petrographic results showed that the analysed aggregates were quartz sands with varying quartz content and form and some proportion of lithoclasts and feldspar grains. The full results of the analysis according to procedure PB/3/18 are presented in Table 2.

The main mineral ingredient of the tested aggregates likely to exhibit reactivity with alkalis are quartz grains where the deformed form is predominant. Aggregate P3 had the highest content of deformed quartz – 53%; the lowest content of deformed quartz, in turn, was found in aggregate P1 – 41%. The reactivity of polycrystalline quartz depends on the size of the crystallites, with quartz grains < 10 µm being the most reactive and crystallites with a size > 130 µm not reacting with alkalis [17]. Quartz with different crystallite sizes, i.e., 10–50 µm, was identified in the tested aggregates, with the highest proportion in aggregates P2 (< 10 µm – 36%) and P1 (10–50 µm – 13%).

Figure 1 shows microphotographs of the tested aggregates, together with an indication of potentially reactive ingredients, i.e., deformed quartz grains labelled Q_n and polycrystalline quartz Q_{pol}.

Results of aggregate reactivity determination based on expansion tests

Figures 2a–b show the course of expansion of the bars of mortars and concretes made from the analysed aggregates. The red line indicates the limit of expansion below which the aggregate is considered non-reactive. For the mortars tested according to PB/1/18, there is a large difference in the measured elongation of the specimens. Aggregate P3 showed the smallest expansion of all the tested specimens, i.e., 0.14% after 14 days, while aggregate P2 showed the largest expansion – 0.29%. There are also noticeable differences in the expansion curve itself, where for aggregates P1 and P2 showed a steeper increase in the period up to day 12 than for aggregates P3 and P4. For all the mortars

Table 2. Classification of the minerals in the aggregate under study

Material name	Mineral ingredients	Content (%)
P1	Quartz exhibiting homogeneous light attenuation	13
	Deformed quartz (with wavy attenuation)	46
	Polycrystalline quartz (with crystallite size 10–50 µm)	13
	Lithoclasts	28
	Feldspar grains	1
P2	Quartz exhibiting homogeneous light attenuation	10
	Deformed quartz (with wavy attenuation)	41
	Polycrystalline quartz (with crystallite size < 10 µm)	36
	Lithoclasts	7
	Feldspar grains	9
P3	Quartz exhibiting homogeneous light attenuation	8
	Deformed quartz (with wavy attenuation)	53
	Polycrystalline quartz (with crystallite size < 50 µm)	5
	Lithoclasts	27
	Feldspar grains	7
P4	Quartz exhibiting homogeneous light attenuation	25
	Deformed quartz (with wavy attenuation)	52
	Polycrystalline quartz (with crystallite size < 30 µm)	6
	Lithoclasts	12
	Feldspar grains	5

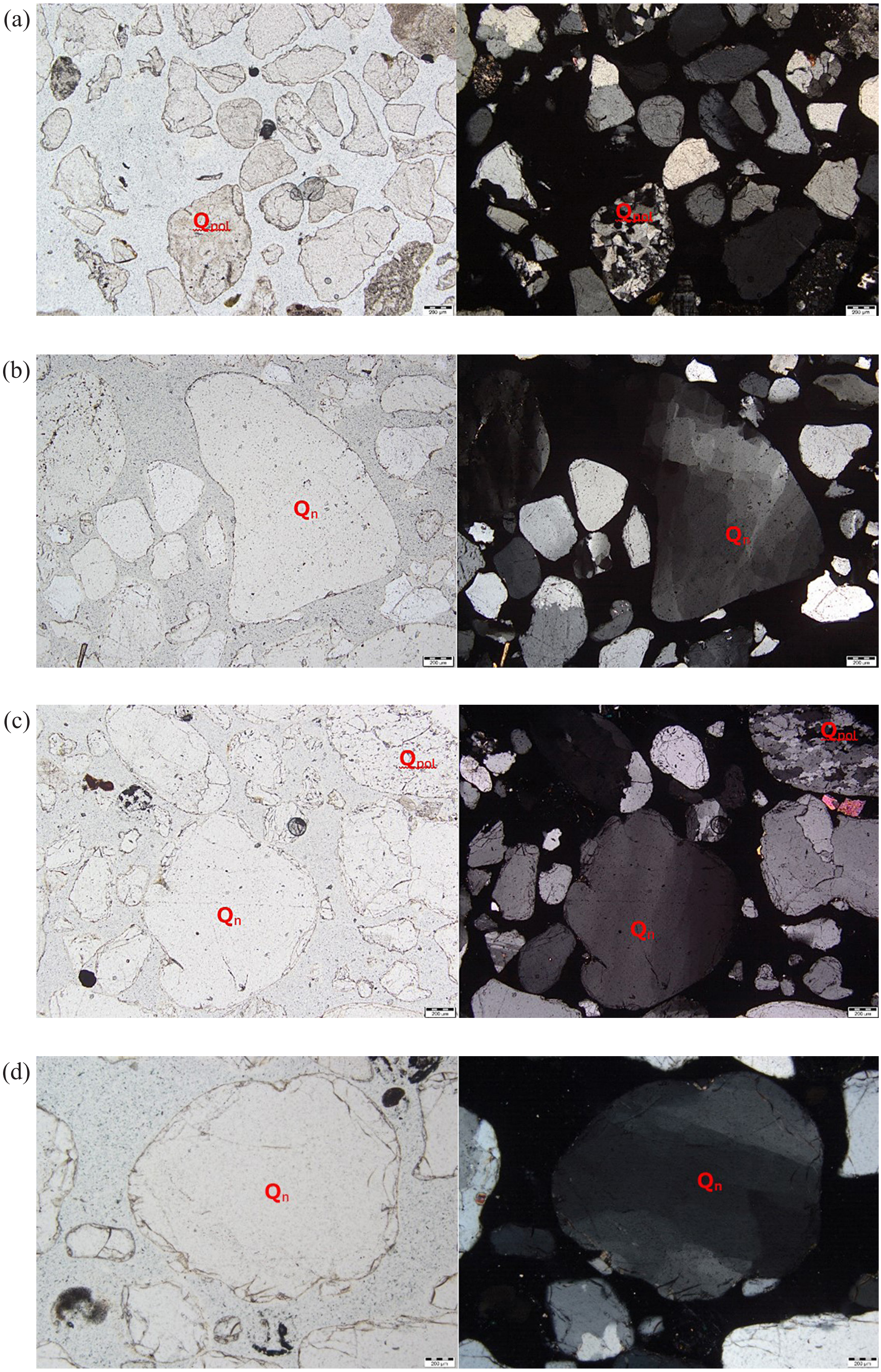

Fig. 1. Microscopic images of specimens in transmitted light with plane-polarised and cross-polarised dipoles; Q_n – deformed quartz Q_{pol} – polycrystalline quartz; (a) P1, (b) P2, (c) P3, (d) P4

analysed, the increase in elongation of the specimens slowed down significantly after 12 days.

The expansion of the concrete specimens tested according to PB/2/18 for aggregates P1, P2, P4 was close to linear throughout the test period, while for aggregate P3, no significant increase in bar elongation was recorded until the middle of the test period; an intense increase in length occurred between 182 and 364 days of testing. The magnitude of change in elongation of the specimens for all of the analysed aggregates did not exceed 0.04% after 12 months of testing.

DISCUSSION OF THE RESULTS

The reactivity of the aggregates was assessed on the basis of the mortar and concrete expansion results obtained using the accelerated method PB/1/18 and the long-term method PB/2/18, assigning them to the corresponding reactivity categories according to the Technical Guidelines for the Classification of Domestic Aggregates [18]. The results are given in Table 3.

The expansion results showed differences in the reactivity categories of the individual fine aggregates for the test series using the accelerated procedure PB/1/18. The measured expansion values are in the range of 0.14% to 0.29%, which means that most of the tested materials are classified as R1 – moderately reactive aggregate. In contrast, the recorded expansion values according to PB/2/18 are significantly lower (0.02–0.04), leading to their classification as R0, indicating a non-reactive aggregate. Particularly significant differences are found for sands P1, P2 and P4, which were classified as R1 when tested according to procedure PB/1/18 and as R0 when tested according to procedure PB/2/18. Only in the case of the P3 aggregate, the classifications according to the accelerated and the long-term methods were consistent.

Petrographic analysis provides important information on the potential reactivity of the

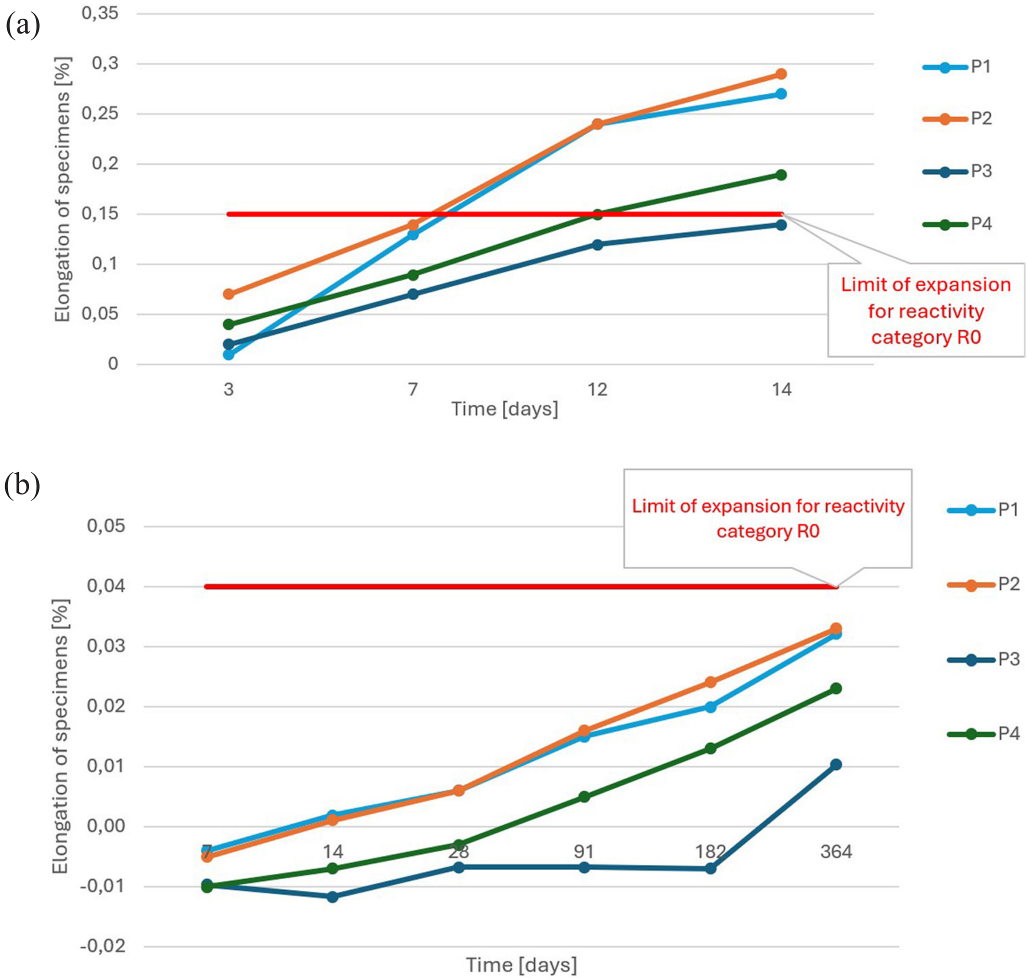

Figure 2. Measurement of linear changes in bar length: (a) according to Pb/1/18, (b) according to Pb/2/18

Table 3. Comparison of the reactivity classifications of the tested fine aggregates

Material	PB/1/18	Reactivity category	PB/2/18	Reactivity category
P1	0.27	R1	0.03	R0
P2	0.29	R1	0.03	R0
P3	0.14	R0	0.04	R0
P4	0.19	R1	0.02	R0

aggregate, enabling a better explanation of the results. Potentially reactive ingredients of the tested aggregates include deformed quartz and polycrystalline quartz [19, 20, 21]. Aggregates containing deformed quartz are categorised as slow-reacting or late-reacting [22], while those containing microcrystalline quartz react faster. In addition, as indicated in the literature, the higher the deformed quartz content, the higher the expansion values should be recorded when using the short-term method [23]. The content of the various forms of silica also affects the recorded expansion values – the less reactive the form, the greater the permissible limit of its content in the aggregate. Permissible limits for microcrystalline quartz and deformed quartz content range from 0.5% to 30% [24]. Deformed quartz content (with wavy attenuation) is dominant in aggregates P3 (53%) and P4 (52%). Despite this, specimens with P3 and P4 aggregates show low expansion when tested using both PB 1/18 and PB/2/18 methods. Polycrystalline quartz, which can also exhibit reactivity [25], is present in the greatest amount in aggregates P2 (36%) and P1 (13%), and it is for the mortar bars prepared from these aggregates that the highest values of linear elongation were recorded when using the short-term method. For polycrystalline quartz, as mentioned above, the size of the crystallites is the most important factor. The literature indicates that crystallites < 10 µm are highly reactive, crystallites 10–60 µm are considered reactive, for crystallites 60–130 µm, reactivity is uncertain, and quartz crystallites > 130 µm do not react with alkalis [17]. It can therefore be assumed that this translates directly into the magnitude of the observed expansion in tests performed using the accelerated and long-term methods, where the largest linear change was observed for P1 and P2 sand. It is noteworthy that for the tested fine aggregates characterised by the lowest polycrystalline quartz content, the expansion values were the lowest for tests performed using both the accelerated and long-term procedure.

The dependence of the expansion value on the reactive form of silica and its content was also observed by Alaejos and Lanza. In presenting this issue in greater detail, the authors pointed out a correlation between mortar expansion and total content of quartz crystallites with a size of < 10 µm and 10–60 µm, also finding that grains with a size of > 60 µm did not affect expansion. The designated equivalent reactive quartz (ERQ), which is the sum of the volumetric fraction of quartz with crystallites with a size of < 10 µm (HR) and quartz with crystallites with a size of 10–60 µm (*R*), is 2.6% by volume for aggregates containing these forms of reactive silica. The R/HR ratio was determined to be equal to 0.31, excluding extremely reactive cryptocrystalline quartz. Applying these findings to the quartz sands analysed in this article, it could be concluded that the dependence of the expansion recorded in the accelerated test on the size of the quartz crystallites was preserved.

The lack of correlation between the classification results obtained in the accelerated test and long-term test is probably due to the lower reactivity of these aggregates in the conditions specified in the PB/2/18 method. However, it should be noted that the trend in the course of expansion for the tested aggregates itself has been preserved and is the same as that found in the short-term test. The delayed response of the aggregate containing slow-reacting deformed quartz may be indicated by the delayed increase in linear elongation of concrete bars made using the P3 aggregate (deformed quartz content: 53%, polycrystalline quartz content: 5%). It appears that in the analysed aggregates, the key factors are the conditions of the tests, i.e., the availability or non-availability of alkalis and temperature, which determine the rate of reaction of the different forms of$_{SiO2}$, i.e. deformed and polycrystalline quartz, with alkalis. This finding requires further in-depth analyses, enhanced by studies of the microstructures of grains subject to alkali corrosion.

CONCLUSIONS

The following conclusions were drawn from the research. Potentially reactive forms of silica were identified in the studied aggregates. The main ingredient of the analysed natural fine aggregates analysed was deformed quartz, making up 46% to 53% by volume. Polycrystalline quartz occurred in smaller quantities, i.e., 5% to 36% vol.

The most reactive quartz sand (P2) contained the highest amounts of polycrystalline quartz with a grain size of < 10 µm and a significant amount of deformed quartz (41%).

Based on testing procedure PB/1/18 of the General Directorate for National Roads and Motorways, three of the analysed aggregates (P1, P2, P4) were shown to exhibit harmful expansion due to ASR, and based on the categorisation of reactivity of aggregates for concrete, they are classified as moderately reactive (R1). Reactivity tests according to procedure PB/2/18 indicated that all analysed aggregates should be classified as non-reactive (R0). The classification of reactivity according to the accelerated method PB1/18 is not always consistent with PB/2/18, which can lead to different categorisation. This means that the accelerated method PB/1/18 does not always provide results that are consistent with the long-term method PB/2/18, leading to the misclassification of aggregates in terms of their alkaline reactivity, which could limit their use despite the fact that these aggregates may have other desirable physical and mechanical properties. This confirms the need for careful interpretation of the results of accelerated tests.

The differences in the classification of potential alkaline reactivity according to PB/1/18 and PB/2/18 indicate an impact of the test conditions on the reactivity assessment. Further refinement of accelerated methods for assessing the alkaline reactivity of aggregates is needed to improve correlation with the long-term method results, which is considered to be the most reliable with regard to the behaviour of aggregates in the structure.

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