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A NEW APPROACH TO THE ACCELERATED METHOD FOR ASSESSING THE ALKALI REACTIVITY OF DOMESTIC AGGREGATES

NOWE PODEJŚCIE DO PRZYSPIESZONEJ METODY BADANIA REAKTYWNOŚCI ALKALICZNEJ KRUSZYW KRAJOWYCH

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Abstract

In Poland, the prevailing protocols for examining the alkali reactivity of aggregates are based on indirect methodologies such as petrographic appraisal, in addition to direct methodologies including the measurement of expansion in mortar and concrete specimens containing the aggregate under investigation. The available research methods exhibit certain deficiencies, which have been mitigated under the experimental conditions delineated in the novel accelerated approach for ascertaining the reactivity of aggregates, otherwise known as the MCPT – Miniature Concrete Prism Test. The methodology of MCPT has the potential to become an alternative for the existing procedures of quality assessment for both fine and coarse aggregates. This work presents the assessment results of the alkaline reactivity of indigenous fine quartz aggregate, examined in accordance with the protocols established by the Polish General Directorate for National Roads and Motorways along together the novel, expedited MCPT methodology.

Keywords: alkali-aggregate reaction, testing methods, mcpt method, correlation

Streszczenie

Obecnie stosowane w Polsce procedury badania reaktywności alkalicznej kruszyw oparte są na metodach pośrednich, takich jak ocena petrograficzna oraz metodach bezpośrednich, polegających na określaniu ekspansji próbek zapraw i betonów z badanym kruszywem. Dostępne metody badawcze wykazują pewne wady, które zostały ograniczone w warunkach badawczych ustalonych w nowej przyspieszonej metodzie określania reaktywności kruszyw, tzw. MCPT – Miniature Concrete Prism Test. Metoda MCPT może stać się alternatywą dla obecnego testowania jakości kruszyw drobnych i grubych. W pracy przedstawiono wyniki oceny reaktywności alkalicznej krajowego drobnego kruszywa kwarcowego, badanego zgodnie z procedurami Generalnej Dyrekcji Dróg Krajowych i Autostrad oraz nowej przyspieszonej metody MCPT.

Słowa kluczowe: korozja alkaliczna kruszyw, metody badawcze, metoda MCPT, korelacja

1. INTRODUCTION

The reaction of sodium and potassium hydroxides found in the pore fluid of concrete with specific types of aggregate silica (ASR) results in a reduction in the durability of the concrete, and, in severe instances, leads to its complete disintegration. The reaction yields a hygroscopic gel of sodium and potassium silicates, containing calcium ions and exhibiting

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expansion capabilities [1]. The implications of ASR, such as failures of civil engineering structures, the lack of ability or extensive costs of building repairs, render the assessment of the quality of aggregates used in concrete as a current and crucial matter in modern concrete technology.

The main method of preventing ASR-induced concrete damage is the use of aggregates that which are environmentally compatible. For this purpose, appropriate classification of aggregates is required, as it allows for potential additional limitations in the concrete mixture. Research methods for evaluating the reactivity of aggregates have been a focus of extensive, worldwide, and their development has been continuously ongoing since the 1940s [2].

The technical directives of the Polish General Directorate for National Roads and Motorways to establish the appropriateness of aggregate for application in structural concrete necessitate conducting a petrographic assessment (in accordance with PB/3/18 [3]) which considers the detection of reactive silica forms in the aggregate [4]. The classification of aggregate reactivity, which includes non-reactive (R0), moderately reactive (R1), highly reactive (R2), and extremely reactive (R3), is determined through direct methods by evaluating the elongation rate of mortar and concrete specimens. The accelerated testing method in accordance with procedure PB/1/18 [5], based on the guidelines issued by ASTM C1260 [6] and RILEM AAR-2 [7], allow for a fast assessment of the reactivity degree of aggregate based on measuring linear changes in the elongation of cement mortars stored for 14 days in a 1M NaOH solution at 80°C [5]. The harsh conditions of the research methodology, coupled with the necessity for mechanical fragmentation of coarse aggregates into requisite fractions for analysis, frequently lead to erroneous classification. In light of the considerable number of false positives and the relatively small number of false negatives, many researchers consider the faster method to be a reliable means of determining the absence of damaging reactivity in an aggregate [8, 9]. The study is particularly advantageous for aggregates that exhibit a low rate of reaction [10].

The research method that most closely approximates actual conditions is that outlined in the PB/2/18 procedure [11], which is based on the methods set forth in the ASTM C1293 [12] and RILEM AAR-3 [13] standards. The concrete specimens are maintained at a temperature of 38°C and a relative humidity exceeding 95% for a period of 12 months. Although the method is considered to be dependable, the one-year assessment period makes it unsuitable for continuous monitoring of aggregate quality. A further limitation of the long-term method is the considerable degree of alkali leachability, which may lead to an underestimation of the expansion results of the tested specimens. It has been demonstrated that approximately 35% of the original alkalis present in the concrete samples examined are leached over the course of a year, with approximately 20% being leached after just 90 days. These findings are supported by the literature, with sources [14-16] providing similar evidence.

The weak correlation between the results of mortar expansion and the expansion of concretes with aggregates of varying reactivity, investigated by accelerated and long-term methods, prompted the development of a new research method, MCPT, by Latifee and Rangaraju [17]. This novel approach to the study of fine and coarse aggregates represents a modification and adaptation of the methodologies outlined in ASTM C1260 and ASTM C1293. The elevation of the testing temperature from 38°C, as utilised in the ASTM C1293 standard method, to 60°C in the MCPT method has the effect of accelerating the reaction and consequently the expansion of concrete containing reactive aggregate. Nevertheless, the utilisation of an immersion solution (1M NaOH) effectively inhibits the leaching of alkalis from concrete. The MCPT method does not include any stipulations regarding the fragmentation of the tested aggregates for the purpose of adjusting their grain size grading. The investigation period has been protracted from 56 days to 84 days for slowly reactive aggregates. A notable correlation exists between the results obtained via the MCPT method and those procured through the long-term method (ASTM C1293). Conversely, a weaker correlation is observed between MCPT and the accelerated method (ASTM C1260) for non-reactive, moderately reactive and highly reactive aggregates [18]. Moreover, the MCPT method is regarded as a reliable and precise technique for assessing the reactivity of aggregates and the effectiveness of ASR preventive measures in comparison to the AMBT method, particularly for aggregates with high reactivity [19]. Furthermore, it is essential to confirm a better correlation between the expansion results obtained through MCPT techniques and the outcomes achieved through the long-term method for coarse aggregates in comparison to fine aggregates [17]. The universality of the new method for evaluating aggregate reactivity necessitates further investigation – currently, the data concerning determining correlations, particularly for slowly reactive or moderately reactive aggregates, still remains insufficient. The objective of the studies presented in the article is to evaluate the reactivity of fine aggregate based on the results of mortar and concrete expansion tests obtained using research methods PB/1/18, PB/2/18, and MCPT. The work was based on an examination of an aggregate containing cryptocrystalline quartz as a reactive component and deformed quartz, which are minerals that are considered to react more slowly with alkalis [20-22].

2. MATERIALS AND RESEARCH METHODS

2.1. Cement

The mortar and cement samples were prepared using CEM I 52.5R cement with an alkali content expressed as equivalent Na_2O_{eq} at a level of 0.87%. The chemical composition is delineated in Table 1.

2.2. Aggregate

The studies used domestic, medium-grained quartz sand (0/2 mm) with a significant proportion of carbonate lithoclasts. Granite was employed as the non-reactive coarse aggregate, with fractions of 2/8 mm and 8/16 mm. The findings of the studies conducted in accordance with PB/1/18, PB/2/18 and PB/3/18 substantiate the non-reactive nature of the aggregate. The mineralogical composition of the fine aggregate, together with the determination of the types and percentage share of reactive silica forms, is presented in Table 2 and Figure 1. The petrographic analysis of the aggregate was conducted in accordance with the procedures set forth in guideline PB/3/18 issued by the Polish General Directorate for National Roads and Motorways. The samples under investigation were subject to reduction, following which they were meticulously prepared for observation under the conditions of transmitted light polarisation, as per the guidelines laid out in the PN-EN 12407 standard [23]. The visual inspection of the aggregate material was executed through an Olympus BX-51 polarising microscope. In the course of the research, two lenses were employed: a 5× and a 10× lens. The images were obtained using an Olympus SC180 camera. The planimetric analysis was conducted in a grid with a 0.4 mm pitch, with a total of over 1500 counts.

Table 1. Oxide composition of cement

Cement type	Oxide composition [%]									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K20	LOI	Na_2O_{eq}
CEM I 52.5R	19.2	5.05	3.01	64.0	2.1	3.48	0.26	0.92	1.98	0.87

Table 2. The particulate constituents present in the fine-grade aggregate were identified

Component	Description	Percentage rate [%]
Quartz	The lithoclastic constituents were composed of cryptocrystalline and polycrystalline quartz fragments	5
Deformed quartz (with wavy attenuation)	The grains exhibited a range of shapes, ranging from semi-angular to rounded, and a corresponding range of diameters, from 0.1 to 1.6 mm (see Fig. 1)	41
Polycrystalline quartz	Particles with a crystal size <0.010 mm (cryptocrystalline in nature) typically exhibit a well-rounded morphology, frequently culminating in the formation of larger grains with dimensions reaching up to 1 mm (see Fig. 1)	36
Quartz exhibiting homogeneous light attenuation	Well-rounded, with an average diameter of 0.4 mm	10
Feldspar grains	The particles of potassium feldspar are manifested as microcline, Carlsbad law-compliant orthoclase twinning, and perthitic intergrowths. They are exceptionally rounded with markedly smaller dimensions, not exceeding 0.3 mm	9
Lithoclasts	A small number of grains of semi-rounded fragments of metamorphic rocks (such as sericite schists)	7

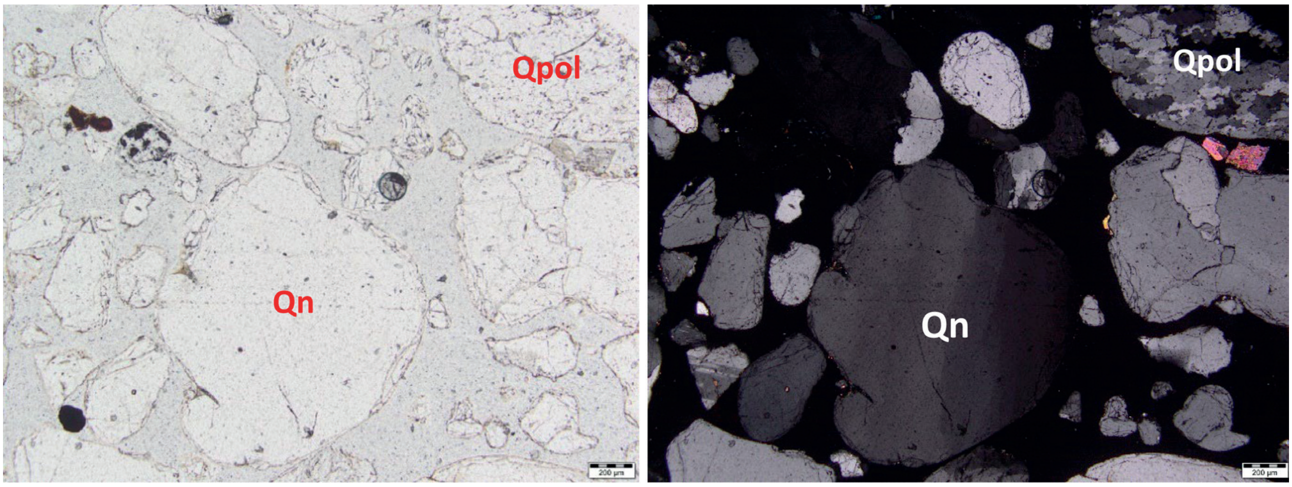


Fig. 1. The sample's microscopic representation exhibits prominently spherical grains of quartz with wavy attenuation (known as deformed Qn quartz), in conjunction with polycrystalline quartz (Qpol) (refer to figures 1N and XN)

2.3. Research procedures

The categorisation of the reactivity of the examined fine aggregate was conducted through the measurement of linear elongation of samples utilising three methodologies: the procedures PB/1/18 and PB/2/18, as well as the modified MCPT method. The modification entailed alterations in the granularity and proportion of individual fractions. The fine aggregate of the 0/4 mm fraction constituted 40% of the total aggregate mass, while the coarse aggregate of the 4/22 mm fraction accounted for 60%. This methodology was developed as a means of calibrating the granulometry of aggregates to align with the standard aggregate blend employed in the PB/2/18 protocol. Table 3 presents a comparison of the employed methods for assessing the potential alkali reactivity of aggregates, along with an indication of the differences in relation to the original MCPT method.

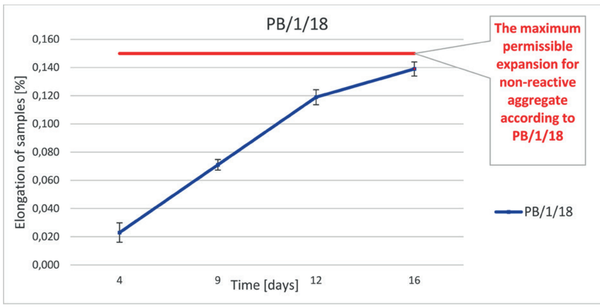
In accordance with the specifications of the MCPT method, three concrete specimens of dimensions 50 × 50 × 285 mm were prepared. The specimens were stored in moulds under conditions of high humidity (relative humidity >95%) for a period of 24 hours, after which they were transferred to water at a temperature of 60°C. Two days following the moulding process, the initial length of the specimens was measured. Thereafter, the concrete bars were placed in a 1M NaOH solution at 60°C. The linear changes of these specimens were measured at intervals of 3, 5, 7, 10, 12, 14, 21, 28, 42, and 56 days using a Graf-Kaufman device.

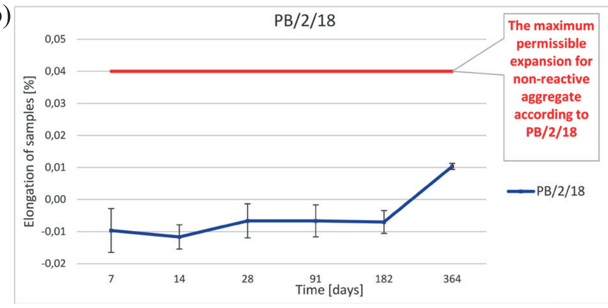
3. RESULTS

The expansion trajectory of mortar and concrete bars utilising the aforementioned fine aggregate is illustrated in Figures 2a-c. The delineation of aggregate reactivity, indicated by red lines,

Table 3. Analysis of methodologies for evaluating potential reactivity of aggregates

Research method	PB/1/18 [5]	PB/2/18 [11]	MCPT [17]	MCPT (modified)
Dimensions of the specimens [mm]	25 x 25 x 285	75 x 75 x 285	50 x 50 x 285	50 x 50 x 285
Dimensions of the aggregates [mm]	0.125 – 4	<22	<12.5	<22
Cement content [kg/m^3]	440	420±10	420	420
W/c (water-cement ratio)	0.47	0.42-0.45	0.45	0.45
Increase in Na_2O_{eq} [%] content	None	1.25	1.25	1.25
Storage conditions	1M NaOH, 80°C	RH>95%, 38°C	1M NaOH, 60°C	1M NaOH, 60°C

a)


b)


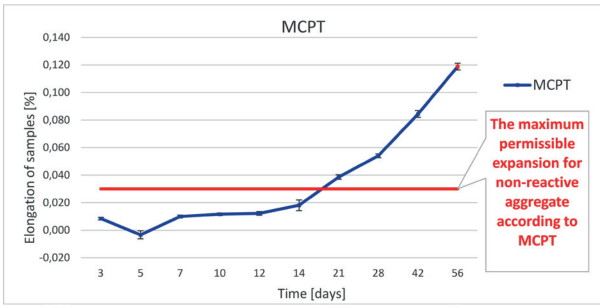
c)


Fig. 2. The alteration of elongation in samples subjected to studies using the following methods: a) PB/1/18, b) PB/2/18, c) MCPT

Table 4. Categorisation of aggregate reactivity for concrete [24]

Research method	PB/1/18 [5]	PB/2/18 [11]	MCPT [17]	MCPT (modified)
Upper limit of expansion	NR: 0.15% after 2 weeks MR: 0.30 after 2 weeks HR: 0.45% after 2 weeks	NR: 0.04% after 52 weeks MR: 0.12% after 52 weeks HR: 0.24% after 52 weeks	NR: 0.03%5 after 8 weeks[1] SR: 0.04 % after 8 weeks MR: 0.12% after 8 weeks HR: 0.24% after 8 weeks	NR: 0.03% after 8 weeks[1] SR: 0.04 % after 8 weeks MR: 0.12% after 8 weeks HR: 0.24% after 8 weeks

where: NR – non-reactive aggregate, MR – moderately reactive aggregate, HR – highly reactive aggregate, SR – slowly/weakly reactive aggregate.

[1] or 0.04%, if the expansion rate from 8 to 12 weeks is ≤0.010% /2 weeks

categorises the aforementioned aggregates into distinct classifications in accordance with the data presented in Table 4.

In accordance with the established expansion criteria, the quartz aggregate was categorised as follows:

- non-reactive aggregates according to procedure PB/1/18 issued by the GDDKiA (General Directorate for National Roads and Motorways) due to an expansion value after 14 days ≤ 0.15;
- non-reactive aggregates according to procedure PB/2/18 issued by the GDDKiA (General Directorate for National Roads and Motorways) due to an expansion value after 365 days ≤ 0.04;
- aggregates with a medium degree of reactivity, in accordance with the new accelerated MCPT method, due to the expansion value after 56 days ≤ 0.120;

4. DISCUSSION OF RESULTS

The results of petrographic aggregate examinations indicated the presence of potentially reactive forms of silica, including cryptocrystalline quartz and distorted quartz. Small quartz crystals had dimensions of <10µm. Scientific literature suggests that the reactivity of specific silica forms is influenced by the extent of structural imperfections and the dimensions of the crystallite grains [21]. The process of reducing the dimensions of quartz grains, which concurrently expands the surface area susceptible to alkali reactions, results in an amplification of the reactive properties of the material. In accordance with the classification outlined in the Norwegian Standards, crystallites with a size of <60 µm are deemed capable of reacting with alkalis, exhibiting high reactivity within the grain size range of <10 µm, and a lesser degree of reactivity within the range of 10-60 µm.

Nonetheless, it is recognised that the reactivity of granules measuring 60-130 μm is uncertain [25]. Furthermore, it has been established that the quartz content within the boundary layer with a grain size of 10 μm, is approximately 2.6% by volume. However, it is advised to reduce the permissible value to 2% [20]. An investigation of the grains of siliceous aggregate revealed that they contain over 36% of minerals that are capable of reacting with alkalis.

The trajectory of expansion curves ascertained by the three methods being examined exhibits significant deviations between one another. The elongation of samples tested by the accelerated method in accordance with the PB/1/18 procedure demonstrates a uniform increase up to the 12th day of the test. Following a 14-day period, the expansion value reached 0.139%, which is situated close to the lower boundary and thus categorises the aggregate as moderately reactive. In accordance with the aggregate reactivity classification per ASTM C1260, the examined aggregate should be classified as moderately reactive (range $0.101 \leq X \leq 0.200$). The expansion curve for concrete samples tested in accordance with the MCPT method exhibits a distinct trajectory. In the initial period, up to the 12th day, the expansion is observed to increase to a small extent, however after that period, it does amplify. The expansion value of the samples after 56 days is 0.119%, which is similar to the upper limit for classifying the aggregate as moderately reactive (range $0.041 \leq X \leq 0.120$). The values of linear length changes in concrete samples, as determined by the PB/2/18 procedure, indicate the occurrence of concrete shrinkage up to the 180th day. The final value of the concrete sample expansion, i.e., after 360 days, is 0.01%, which is markedly lower than the threshold value for aggregate classification as moderately reactive ($X>0.04$). It must be noted that an elevated test temperature and the provision of an external source of alkalis facilitate the acceleration of the reaction between the examined quartz sand and alkalis. The elongation of the concrete bars was examined according to the methodology specified in procedure PB/2/18, thus under conditions most closely corresponding to real conditions, which indicates the absence of a harmful aggregate reaction with alkalis. The observed increase in expansion at a later time may be indicative of a delayed reaction of deformed quartz forms that are contained within the aggregate. Moreover, the elevated test temperature and the availability of an external NaOH source in the MCPT method provide an environment conducive to the reaction of silica with alkalis, in comparison to the conditions of the long-term method. The more lenient conditions specified in procedure PB/2/18, when considered alongside the potential for increased alkali leaching from concrete samples, may ultimately result in a reduction in the values of measurable concrete expansion [18].

5. CONCLUSIONS

The findings of the study have enabled to formulate the following conclusions:

1. The examined siliceous aggregate was found to contain potentially reactive forms of silica, including cryptocrystalline quartz with grains measuring less than 10 μm and deformed quartz.
2. The investigative techniques for aggregate reactivity, in compliance with PB/1/18, PB/2/18 and MCPT, denote a disparity in the sensitivity of the aggregate towards the alkali – silica reaction. The linear elongation values obtained for concrete samples were used to classify the fine aggregate as moderately reactive in accordance with the MCPT method. However, the results of tests conducted in accordance with the PB/1/18, and PB/2/18 procedures indicated the absence of a harmful aggregate reaction with alkalis.
3. The results of the expansion studies conducted in accordance with the procedures specified in PB/1/18 and PB/2/18 categorise the fine aggregate into various reactivity categories, rendering it suitable for use in R1 and R0. The recorded final values of expansion obtained through the MCPT and PB/1/18 methods are found to be similar. Following an analysis of the findings from the Miniature Concrete Prism Test, the aggregate was categorised as belonging to the SR grouping, which denotes a slow and weak reactivity.
4. The parameters of the reaction such as temperature, humidity, presence of antagonistic ions in relation to silica, and the period of investigation, exert an impact on the progression of corrosive processes.
5. The study results demonstrate the necessity for further enhancement of the existing methodologies for the determination of alkali reactivity of aggregates in Poland. The proposal of an alternative method for examining concrete sample expansion (MCPT – Miniature Concrete Prism Test) represents a potential avenue for improvement, addressing some of the shortcomings of the current procedures. However, further research is required to confirm the effectiveness and accuracy of this proposed method.

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