Scanning electron microscopy in the tests of fibre-cement boards

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Abstract. The subject of this article is research on fiber-cement boards, which are currently used in civil engineering as cladding for ventilated facades, but also as internal claddings. When these boards are used on elevations, they are exposed to changing weather conditions, and therefore they are given the appropriate requirements for strength, mass moisture, absorbability and, above all, durability in accordance with the relevant regulations. The paper presents a proposal for using a non-destructive microscopic method for testing fiber-cement boards using a scanning electron microscope (SEM) with an EDS analyzer. Fiber-cement boards subjected to various environmental factors (moisturizing, freezing-thawing) and exceptional factors (burning at 230°C and setting on fire for 5 and 10 minutes) were tested. Interesting research results were obtained, which allowed to observe changes occurring in the microstructure of the tested boards under the influence of various factors.

1 Introduction

Building materials made of fibres and cement have been usedin the construction for more than a hundred years, and they are also called cellulose fibre cement products. Their usage was initiated by Ludwig Hatschek, who in 1900 developed and patented the technology of producing light, strong, durable and non-flammable asbestos cement sheet, which was called an eternit by him. This sheet became one of the most popular roof coverings in the world in the 20th century. It was one of the most popular roof covering up to the moment when it was stated that asbestos is harmful. As a result of activities of numerous international and domestic medical associations and administrative authorities and institutions, their production was stopped and these products were gradually removed from being used, and the ingredient that was dangerous to the health was replaced by safe fibres. Such change was initiated in the world in 1970s, and in Poland at the end of 1980s. [1,2].

Currently, fibre cement boards are building products made of pure and harmless raw materials. These boards are made of natural ingredients, such as cement, cellulose fibres, PVA and water. A major part of the production mixture is cement, responsible for binding

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the material and its final durability. Cellulose is a filler of slots and an addition that ensures that there is appropriate amount of water in the process of binding the cement. Fibres made of polyvinyl alcohol (PVA) are used for reinforcing the material and making it strong enough. In order to optimize the features and to accelerate the process of binding, neutral fillers are added, such as, for example, limestone or mica.

Fibre-cement boards are most frequently used as building and finishing material for internal walls, but also as roofs and as sidings of ventilated facades assembled on the substructure. When used as facade sidings, the elements are exposed to wind load (pressure or suction). Additionally, variable climatic conditions have influence on them. This is also why this material has to be thoroughly controlled before being used, and most of all, when it is produced. It is then essential to analyse the composition of the mixture and the amount and the way of distribution of cellulose and PVA fibres. Incorrectly distributed fibres do not create spatial mesh reinforcement, and this causes the board not having the required strength parameters, and consequently it does not meet the criteria of durability.

As it was already mentioned, because of the fact that fibre-cement materials are currently widely used in construction, they are exposed to the influence of environmental conditions, such as the rain and changes of temperatures, and in particular, frequent freeze-thaw events in day and night cycle (regular freezing-thawing). Moreover, fibre-cement materials, most of all those used as siding elements, are exposed to extreme conditions, and these include high temperature caused by the fire.

In connection with the above, it is essential to conduct the research on the construction materials, taking into consideration environment and extreme conditions to which these materials will be exposed during exploitation. This research should ensure the possibility to control the microstructure of given material, and this is enabled by scanning electron microscopy [3,4]. It is worth highlighting that so far, the majority of research on fibre-cement materials has been limited to determining standard physical and mechanical parameters [5,6]. It is also possible to see the results of research conducted with the use of non-destructive methods, which as a diagnostic method is widely used and simultaneously developed worldwide with reference to steel and concrete constructions as well as composite materials [7-23].

2 Materials and methods

2.1 Materials

The analysis was carried out on two fibre-cement boards of different composition and use, marked in the following part of the article by letters A and B respectively. At the moment of the research, the boards were in the air-dried state and they were the subject of various processes, the purpose of which was to reflect the environmental conditions (soaking in water for 24 hours and 25 cycles of freezing-thawing) and extreme conditions (burning for 3 hours in laboratory furnace in the temperature of 230°C and torching for 5 minutes and 10 minutes).

Table 1. Comparison of tested boards.

Symbol of board	**A**	**B**
Type of boards	fibre-cement, interior	fibre-cement, exterior
Thickness of boards [mm]	8	8
Class of bending strength	3	2
Bending strength [MPa]	12,76	26,54
Moisture content w_w [%]	5,05	2,41
Absorbability n_w [%]	14,71	7,54
Density [kg/m^3]	1100	1600
View of boards		

2.2 Methods

Integrating modern microscopy methods with other research techniques allows to obtain answers to the questions relating to the mechanism of the reaction, the diagnosis of destruction process, the relation between microstructure and performance characteristics [3]. The development of these methods that has been observed in the recent years aims at one hand at maximizing the magnifications and at the visualisation of the objects of nanometric size, and at the other hand - at the detection of any effects that may occur as a result of the influence of the factor that penetrates the analysed material [4].

For many years, an available and common tool in the research on the microstructure of the solids, including various building materials, is scanning electron microscope. Thanks to the use of scanning microscopy, there is the possibility to evaluate the shape and the size of the grains, surface morphology, the presence of joints, inclusions, cracks, the manner the space is structured or the shape of the pores. The main advantage of the method is very simple preparation - the samples are in the form of fractions or splits. The next important feature of scanning electron microscopy is the possibility to obtain extremely large magnifications, even hundreds of thousands times [24-26].

In the research presented in the article, a high resolution environmental scanning electron microscope Quanta 250 FEG, FEI with the EDS analyser was used, located at the Kielce University of Technology (Fig. 1). This equipment enables the research to be made in the conditions of high and low vacuum in ESEM pressure mode (in the presence of external gases). It is possible to make the analysis on any conducting and non-conducting materials. There is also a possibility of SE and BSE imaging in every mode of the work. Additionally, the device is also equipped with automatic trimmer Leica EM TXP and modular high vacuum sputtering system Q15OT ES (fibre-cement samples that were

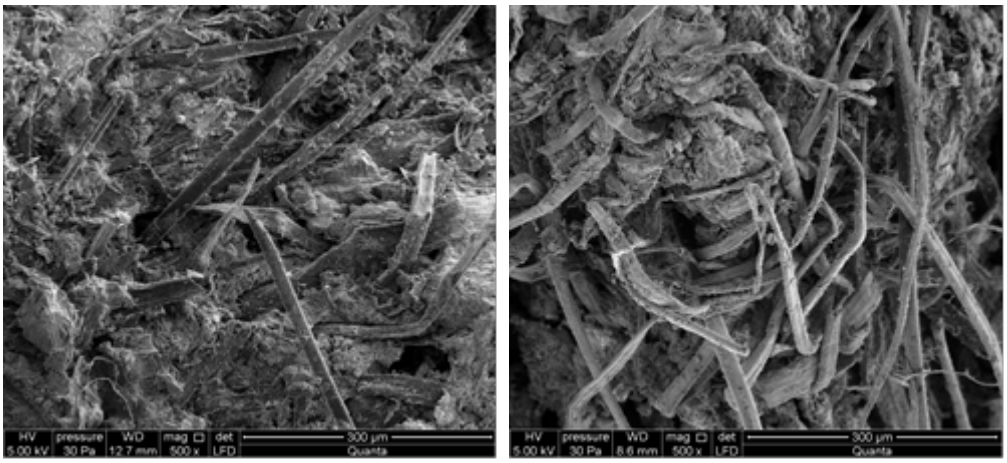
analysed were sputtered by gold and platinum). EDS (EDAX) analyser allows to make a quantitative analysis and to map the chemical composition of the sample.

Fig. 1. The view of scanning electron microscope with the EDS analyser.

3 Results

Figure 2 shows exemplary images obtained with the use of scanning electron microscope for the A board.

a) b)



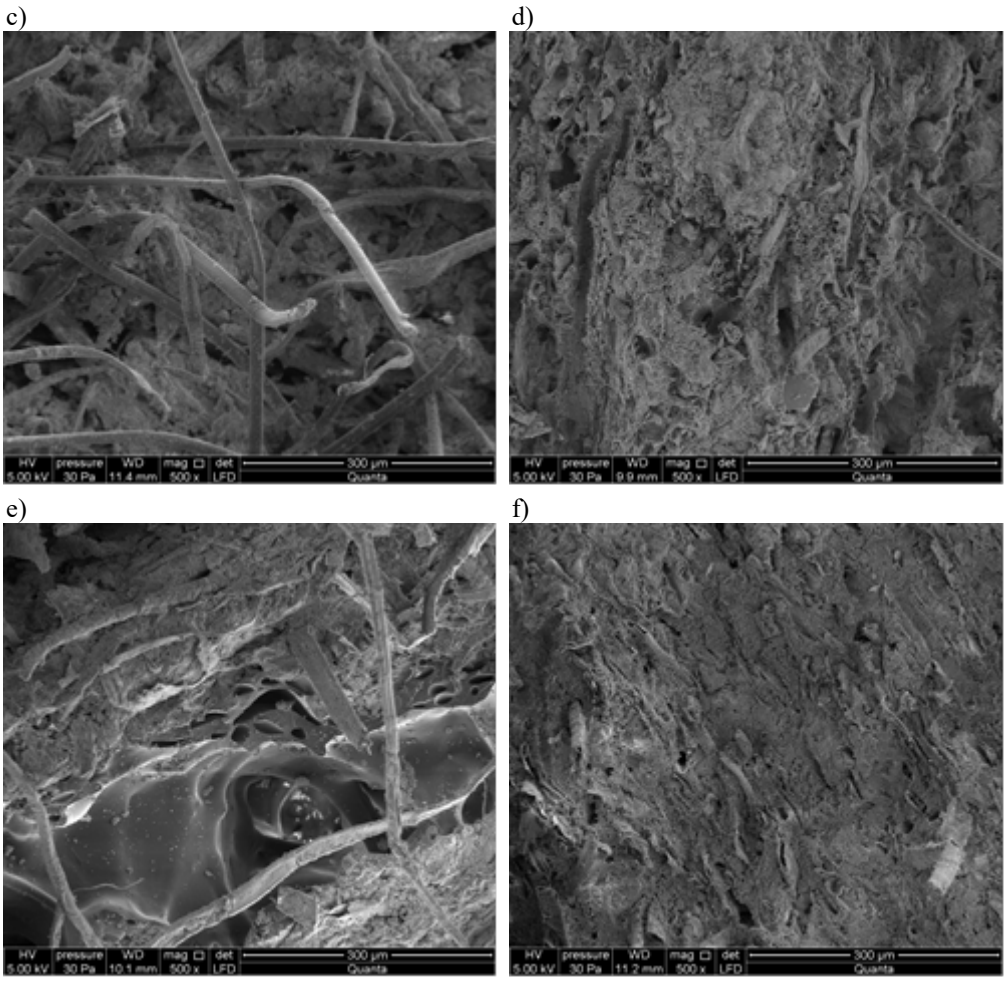
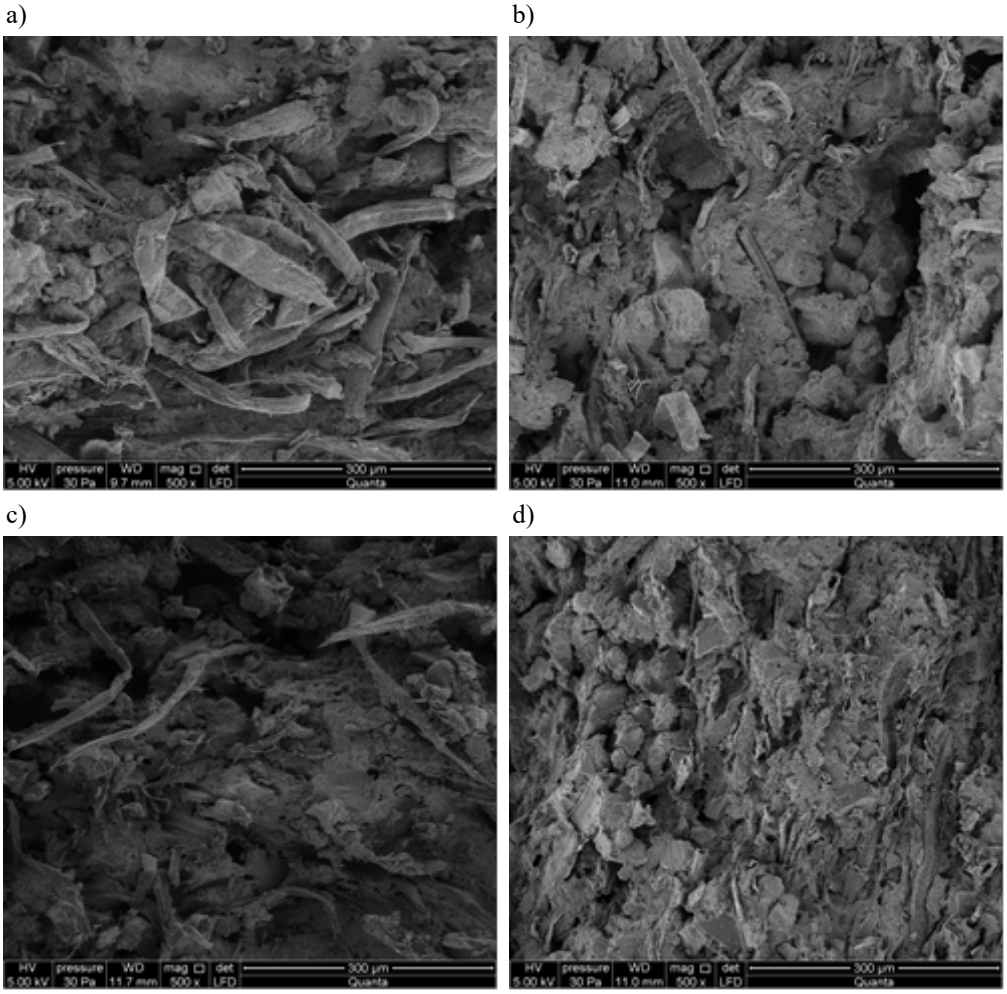


Fig. 2. Images obtained with the use of scanning electron microscope for the board A: a) in air-dry state, b) damp, c) frozen - thawed 25 cycles, d) burnt out in a furnace in the temperature of 230°, e) set on fire for 5 minutes, f) set on fire for 10 minutes.

On the basis of the analysis of the results presented on figure 2, it should be stated that the macrostructure of every sample of the A board was defined as dense. During the microscopic observations, the structure of the samples was determined as fine-pored, with the size of the pores of up to 50 μm. It was stated that there was the occurrence of deep gouges of the width of up to 100 μm on the surface of the splits of the samples that were soaked and frozen. It was noticed that there was great concentration of irregularly distributed cellulose fibres and PVA fibres on the analysed splits (Fig. 2a, 2b and 2c), with the exception of the burnt sample and the samples that were torched. It was noticed in the case of the torched sample that the majority of the fibres is burned down or blended in the matrix (Fig. 2d). Torching causes gradual burning of the fibres and the degradation of their structure, depending on how long the fire was active (Fig. 2e and 2f). It was noticed that there were caverns and gouges in the place of the fibres that were extracted. The structure of the matrix for dry sample was defined as dense. In the remaining cases, it is more granular, and in the case of the sample that was torched for 10 minutes, it was consisting of laminar structures in some places. Because few little spaces between the fibres

and the matrix occurred, it was stated that the bond between these elements was strong. Various forms of $Ca(OH)_2$ were detected in the structure of the matrix, as well as amorphous C-S-H phase and structured of closely adhering particles. Few crystals of ettringite were observed in the sample that was cyclically frozen-thawed.

Figure 3 presents the examples of images obtained with the use of scanning electron microscope for the B board.



e) f)

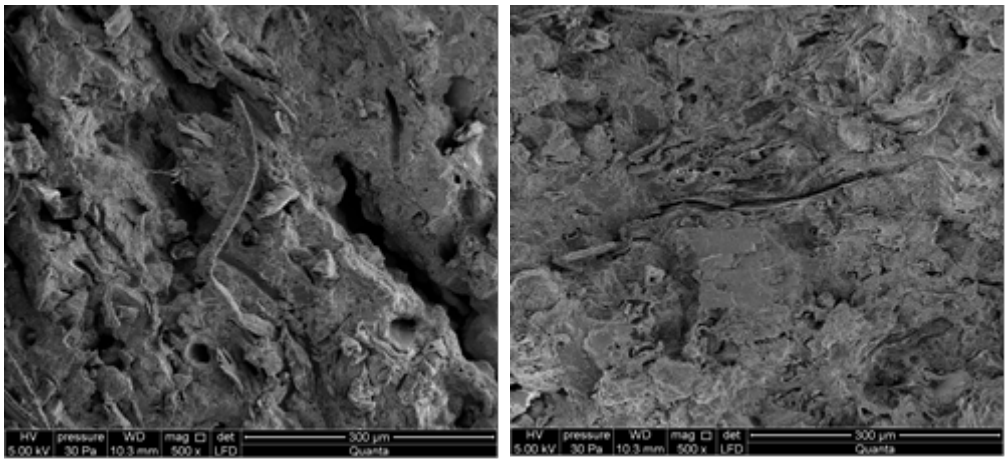


Fig. 3. Images obtained with the use of scanning electron microscope for the board B: a) in air-dry state, b) damp, c) frozen – thawed 25 cycles, d) burnt out in a furnace in the temperature of 230°, e) set on fire for 5 minutes, f) set on fire for 10 minutes.

On the basis of the analysis of the results presented on figure 3, it should be stated that the macrostructure of every sample of the B board was defined as dense. During the microscopic observation, the structure of dry and soaked samples was defined as fine-pored, the size of the pores to 50 μm, and in the case of the samples that were cyclically frozen-thawed, burnt and torched, the size of the pores was up to 100 μm. It was stated that there was irregular distribution of cellulose fibres and PVA fibres on the analysed splits. What is more, in some places there are aggregations of fibres, especially cellulose ones, and in other places there was lower amount of fibres on the splits. It may implicate that the stage of mixing during the process of production was not accurate. In the case of the burnt sample, it was stated that the majority of the fibres is burnt or blended in the matrix (Fig. 3d). However, torching causes gradual burning of the fibres and the degradation of their structure, depending on how long the fire was active (Fig. 3e and 3f). It was noticed that there were caverns and gouges in the place of the fibres that were extracted. In all analysed samples, there are empty spaces less the rim built by the external product. The structure of the matrix for dry sample and soaked sample was defined as extremely dense (Fig. 3c). In the frozen, burnt and torched samples, the structure of the matrix is more granular. Additionally, it was stated that there were very dense structures in every sample (the analysis indicates $Ca(OH)_2$). It was noticed that there were spaces between the fibres and the matrix, and this fact indicates that the bond between them is weak. Various forms of $Ca(OH)_2$ were detected in the structure of the matrix, as well as amorphous C-S-H phase and structured of closely adhering particles. The occurrence of the crystals of ettringite was proved in the sample that was frozen, burnt and torched for 5 minutes. During the analysis of the burnt sample and the samples that were torched (Fig. 3d and 3f), slight micro-cracks were noticed.

Figure 4 presents examples of the results of elemental composition obtained with the use of the EDS analyser for the analysed A board.

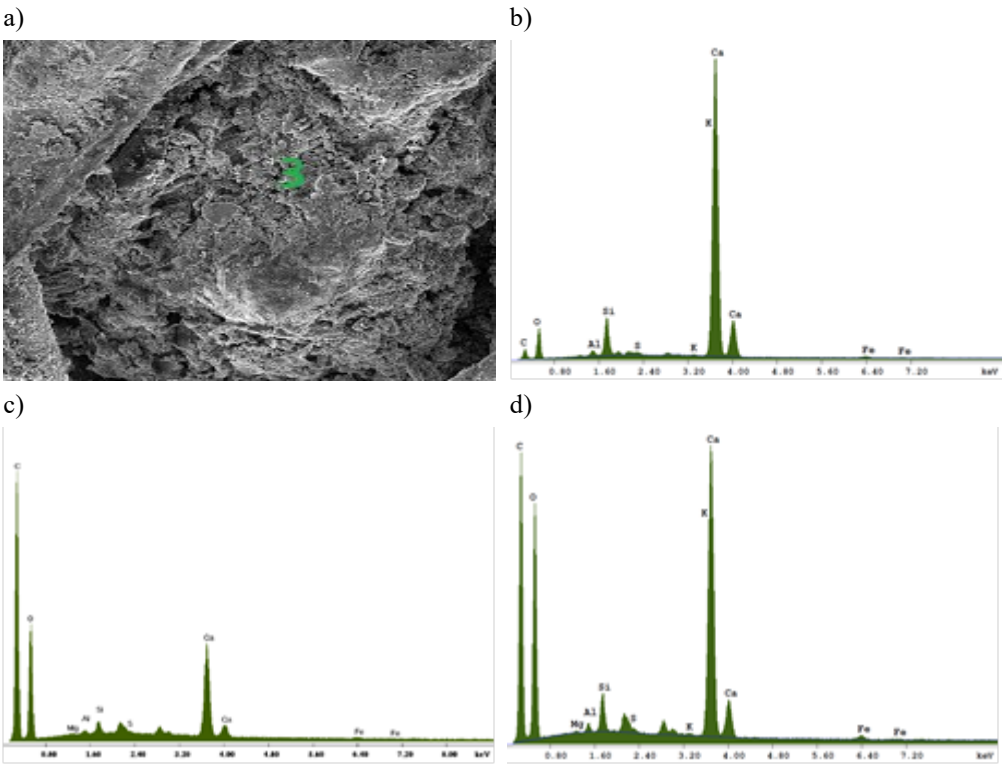
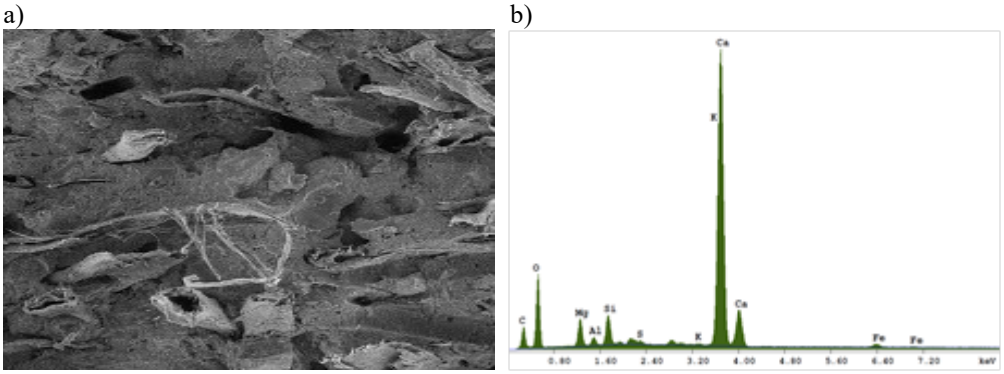


Fig. 4. Results obtained with the use of the EDS analyser for the board A: a) the places of elemental composition analysis, b) results of EDS in point 1, c) results of EDS in point 2, d) results of EDS in point 3.

Figure 5 presents the examples of the results of elemental composition obtained with the use of the EDS analyser for the analysed B board.



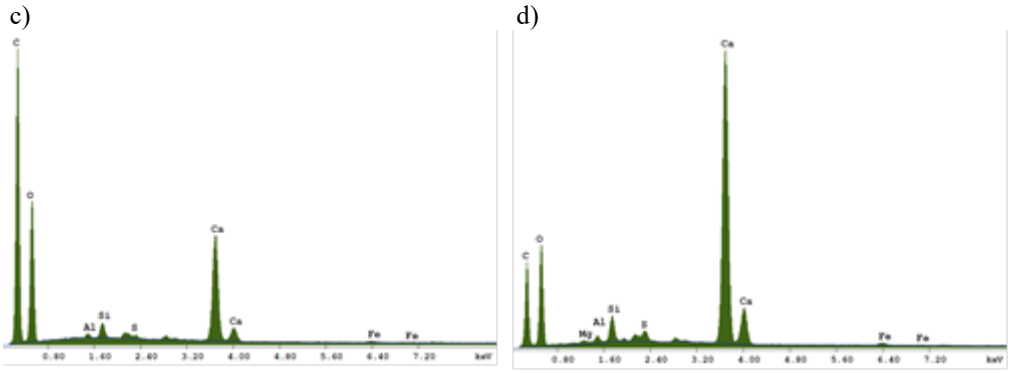


Fig. 5. Results obtained with the use of the EDS analyser for the board B: a) the places of elemental composition analysis, b) results of EDS in point 1, c) results of EDS in point 2, d) results of EDS in point 3.

On the basis of the analysis of elemental composition of the matrix for all samples from both groups (Fig. 4,5), it was stated that there were the elements that are in the composition of cement (Ca, Fe, Al, Mg, S, C, Si, K) (Fig. 4b, 5b). During the analysis of the fibres (Fig. 4c, 5c, 4d and 5d), it was stated that there were elements that are in the composition of fibres (increased amount of elements C and O) or that are partially in the composition of the cement, because the surface of the fibres is covered with thin layer of cement matrix and products of hydration. Based on the analysis of the results obtained for all samples, it was found that environmental and exceptional conditions do not affect the change in the elemental composition of materials.

4 Conclusions

The article presents the proposition for using non-destructive microscopic method for the analysis of fibre-cement boards with the use of scanning electron microscope (SEM) with the EDS analyser. The analysis was conducted on 2 types of fibre-cement boards that were subject to various environmental conditions (soaking, freezing-thawing) and to extreme conditions (burning in the temperature of 230°C and torching for 5 and 10 minutes).

The obtained results of the research allowed to notice changes that occur in the microstructure of the analysed boards under the influence of various factors. The indicated method allows to evaluate the level of degradation of the microstructure of such material after years of exploitation in variable climatic conditions, and mainly after the occurrence of extreme situations, e.g. fire. According to the authors, this method is particularly useful for the construction. In relation to the above, this method should be further developed in the context of analysing fibre-cement materials, in particular boards that are subject to various environmental and extreme conditions.

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