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The Alkali and Temperature Resistance of Some Fibres

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Porous concrete needs to be reinforced to improve its strength, frost-resistance, durability etc. An alkaline media (pH = 12.6) is formed during the production of porous autoclave concrete, so the reinforcing fibres should be stable to alkali and temperature influence (up to 180 °C). The resistance to these factors of four different fibres (mineral and kaolin wool, carbon and polypropene fibres) was investigated using X-ray diffraction and scanning electron microscopy (SEM). It was determined that kaolin wool cannot be recommended for use as a reinforcing agent during the formation of porous concrete. The others investigated fibres might be used for these purposes.

Keywords: mineral and kaolin wool, carbon and polypropene fibres, autoclave conditions, alkaline corrosion, porous concrete.

INTRODUCTION

Porous concrete articles in comparison to high-density concretes possess relatively low flexural and tensile strength also increased fragility. Due to this, various cracks and defects can appear during the production and transportation stages [1].

The porous concrete strength can be increased by introducing more strict specifications for raw materials and new production regulations. Several authors have investigated properties of concretes (mainly high-density) and ways to improve them by adding various fibre additives to the formation mixture [2 – 4]. However the issue of porous concrete reinforcement has not be yet fully investigated.

The concrete investigations have shown that the use of fibrous reinforcements can increase the flexural strength [5 – 7] and tensile strength [7], impact strength [8], reduce contraction deformations [7, 9 – 12], increase frost-resistance and durability [5]. Despite that all the structural changes of these fibrous additives are similar to those existing in porous concrete formation mixtures. The influence of temperature and pressure on the fibres under autoclave treatment have not been investigated.

An alkaline media is formed in the autoclave porous concrete formation mixture, so the fibrous material should be alkali and temperature (up to 180°) resistant.

The aim of this paper was to investigate different fibres suitable for porous concrete reinforcement in an alkaline media. Mineral and kaolin wool, carbon and polypropene fibres were used. It is well known that the first three fibres are temperature resistant from 700 °C to 1200 °C, while polypropene, carbon and basalt fibres are resistant to alkali [13 – 16]. The chemical composition of that mineral wool is close to basalt wool.

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RAW MATERIALS AND INVESTIGATIONS METHODS

The following materials were used to produce an alkaline media: JSC "Naujasis kalcitas" Ltd. milled lime correspon-ding to the CL 90 grade according to EN 459-2 requirements, the lime's main fraction particle size was from 20 μm to 32 μm; JSC "Akmenės cementas" CEM II/A-L 42.5 N type Portland cement corresponding to LST EN 197-1:2001 requirements; solid chemical reagent NaOH.

The chemical composition of Portland cement and lime is given in Table 1.

Fibrous materials used on the experiment: JSC "Paroc" Ltd. mineral wool fibres with acidic module – 2.88. The fibres were taken from the mineral wool conveyer and mechanically treated in a special equipment unit; polypropene fibres produced by "FORTA Corpo-ration" (Germany); synthetic carbon fibres produced in Russia; kaolin wool fibres produced in Ukraine.

The fibres technical specifications are given in Table 2.

The chemical composition of mineral and kaolin wool is given in Table 3.

To create an alkaline media similar to that which arises during porous concrete production, a lime and cement aqueous suspension (5 g solids and 50 ml H_2O) was prepared in a 100 ml glass beaker. After sedimentation, the saturated $Ca(OH)_2$ solution was formed above the settled lime and cement layer. The pH value of this solution, 0.044 M and 0.12 M NaOH solutions were measured and are given in Table 4.

Bags made from filter paper (e.g. paper tea bags) containing 1 g of fibres were put into the beaker with alkaline solutions. To eliminate the influence of CO_2, the glass beakers were covered with a 0.02 mm thick aluminium foil. Prepared samples were treated according to the different regimes. The thermal treatment parameters are given in Table 5.

Table 1. Lime and Portland cement chemical composition

No.	Raw material	Composition, %							Loss on ignition, %
		SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	R_2O	SO_3	
1.	Portland cement	22.41	4.20	4.76	63.00	2.50	–	1.65	1.33
2.	Lime	4.04	2.02	0.87	89.23	2.37	0.6	–	0.86

Table 2. Fibres technical specifications

No.	Fibrous material	Fibre length, mm	Fibre diameter, μm	Tensile strength, GPa	Notes
1.	Mineral wool	5	4.6	2 – 3	Withstands up to 700 °C
2.	Polypropene	5	7.5	0.5	Alkali - resistant
3.	Synthetic carbon fibre	5	4.6 – 7.7	2 – 3	Alkali - resistant, withstands up to 700 °C
4.	Kaolin wool	5	3.3	–	Withstands up to 1200 °C

Table 3. Mineral fibre chemical composition

No.	Fibre	Chemical composition, %					Loss on ignition, %
		SiO_2	Al_2O_3	CaO	MgO	Fe_2O_3	
1.	Mineral wool	45.86	22.65	15.52	7.89	2.81	5.27
2.	Kaolin wool	50.33	49.02	0.12	–	0.23	0.3

Table 4. Alkaline solution pH values

Alkali	Solution conc., M	pH	Notes
$Ca(OH)_2$	0.02	12.6	Saturated solution above lime sediments
$Ca(OH)_2$	0.02	12.6	Saturated solution above cement sediments
NaOH	0.04	12.6	–
NaOH	0.12	13.1	–

Table 5. Fibre thermal treatment parameters in an alkali media

Temperature, °C	Isothermal duration, h	Treatment place	Number of samples
20	168 (7 days)	thermostat	16 (4 fibre types and 4 alkaline media)
50	168 (7 days)	thermostat	–"–
180	8	autoclave (P=1.0 MPa)	–"–

After the certain period of time the fibre was filtered, washed with distilled water and dried at 100 °C. The fibre surface morphology was investigated by a scanning electron microscope JSM 840 (SEM). The fibre was fixed on the specimen holder of scanning electron microscope, covered with vacuum evaporated carbon conductive layer (0.5 μm).

To determine the phase composition of the corrosion products formed during the alkaline solution attack, X-ray diffraction (XRD) investigations (equipment DRON- 1, Cu anode) were performed. XRD investigations were performed also with the crushed fibre.

RESULTS AND DISCUSSIONS

The SEM image of the surface of the non-alkali exposed fibre is presented in Fig. 1. The image of the fibre surface shows that the surface of polypropene, mineral wool and kaolin wool fibres (Fig. 1, b, c, d) is smooth. On the surface of carbon fibre (Fig. 1, a) the longitudinal grooves, formed during fibre fabrication, are seen evidently.

No changes in the surface area of all the fibre's types were noticed after 7 days exposure at the atmospheric pressure and 20 °C temperature, when the fibres were wetted by the different alkaline solutions: 0.04 M and 0.12 M NaOH, saturated $Ca(OH)_2$ and water extract of Portland cement.

After 7 days exposure at the atmospheric pressure, 50 °C temperature only the surface of the mineral wool fibre's wetted by the 0.12 M NaOH solution was changed (Fig. 2): a layer of fibre corrosion products was formed on the fibres surface. The thickness of this layer is about 0.1 μm.

The corrosion products layer was dense and unstable. In some places, the fibre surface was destroyed (Fig. 2, a, b).

The results of the SEM investigations of the surfaces of fibres, wetted by alkaline solutions and kept in auto-clave, showed that only the carbon fibres were stable at

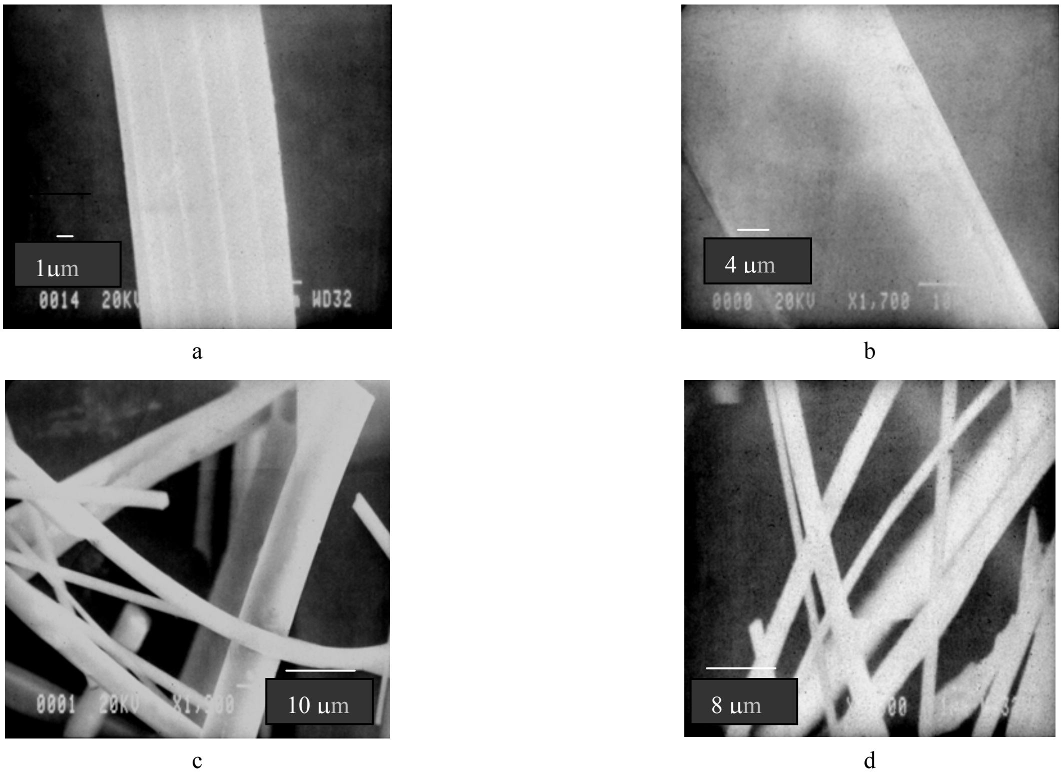


Fig. 1. Non-alkali exposed fibres: a – carbon fibre, b – polypropene fibre, c – mineral wool fibre, d – kaolin fibre

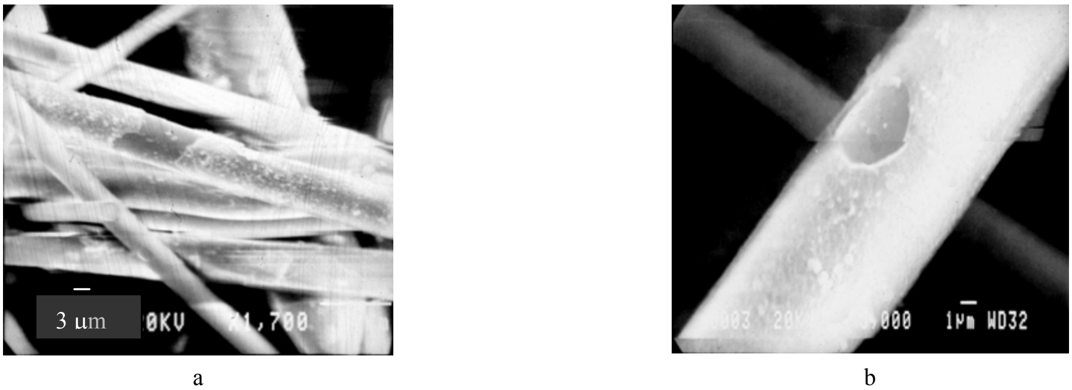


Fig. 2. Mineral wool fibre wetted and exposed by the 0.12 M NaOH solution for 7 days at atmospheric pressure, 50 °C temperature: a – general view, b – corroded fibre

these conditions in spite of the composition of the alkaline solution.

The surface of these fibres did not change in comparison with non-alkali exposed carbon fibre (Fig. 1, a).

No corrosion products were noticed on the layer on the surface of the polypropene fibres wetted by the each of the alkaline solution (0.04 M and 0.12 M NaOH, saturated $Ca(OH)_2$ and water extract of Portland cement) and kept in an autoclave.

But their shape (Fig. 3), in comparison with the shape of as produced fibres (Fig. 1, b), was changed under the temperature attack. It was determined that the change of the shape of these fibres doe's not depend on the composition of the alkaline solution.

The surface area of mineral wool fibres wetted by the alkaline solutions and kept in an autoclave changed significantly (Fig. 4, Fig. 5, Fig. 6) in comparison with the as produced fibres (Fig. 1, c). They became very brittle and decomposed. No long fibres were found in the mineral wool samples. The fibres surfaces were covered by the corrosion products.

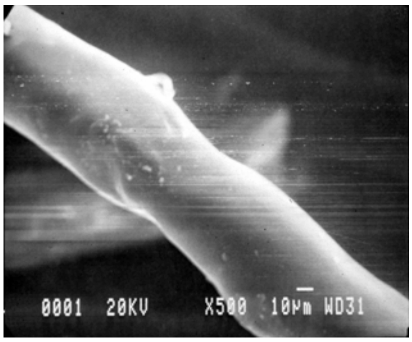
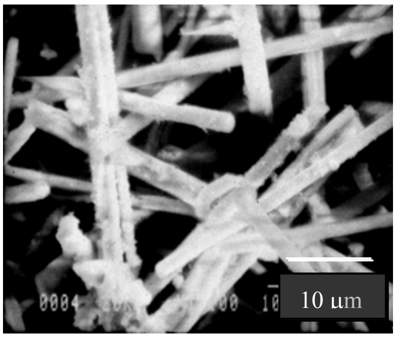


Fig. 3. Polypropene fibre wetted by 0.12 M NaOH after keeping it in autoclave conditions

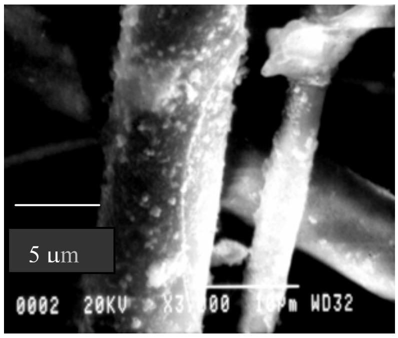
The surface image of mineral wool fibres, wetted by 0.04 M and 0.12 M NaOH solutions and kept in an autoclave, is presented in Fig. 4, wetted by saturated $Ca(OH)_2$ and kept in autoclave – in Fig. 5, wetted by water extract of Portland cement and kept in autoclave – in Fig. 6. One can see that the fibre surface images, presented in all three figures are very similar. The corrosion products, in spite of the alkaline solution composition, are gel-like. A layer of these products is formed on the fibre's surface.

Fig. 7 – Fig. 9 show the SEM images of the kaolin wool fibre's wetted by the alkaline solutions and kept in an autoclave. The surface image of kaolin wool fibres, wetted by 0.04 M and 0.12 M NaOH solutions and kept in an autoclave, is presented in Fig. 7, wetted by saturated $Ca(OH)_2$ and kept in autoclave – in Fig. 8, wetted by water extract of Portland cement and kept in autoclave – in Fig. 9. The images show that the kaolin fibres are not stable to the alkaline solutions attack at the autoclave conditions. The corrosion products layers are formed on a fibre surfaces.

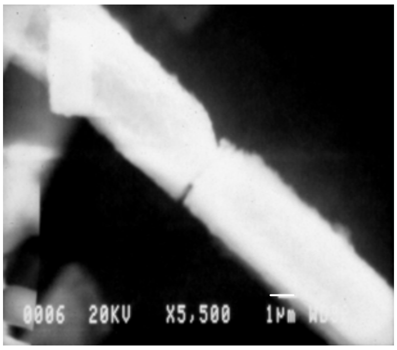
Although there was no possibility for the quantitative determination of the difference in the degree of kaolin wool and mineral wool fibre destruction, it was only noticed that kaolin wool fibres after autoclaving in the alkaline solutions will be very brittle.



a

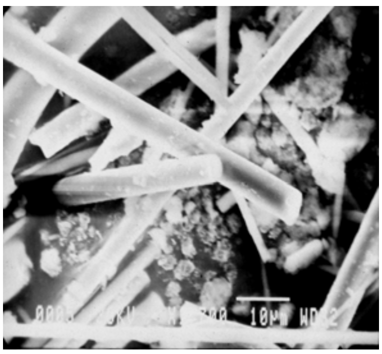


b

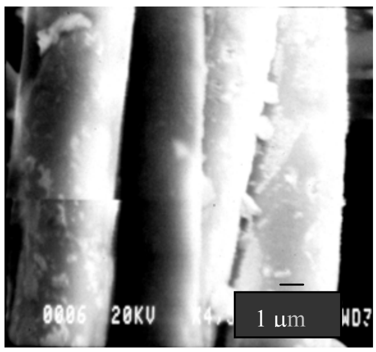


c

Fig. 4. Mineral wool fibre affected by NaOH solution at autoclave conditions: a and b – 0.12 M NaOH, c – 0.04 M NaOH

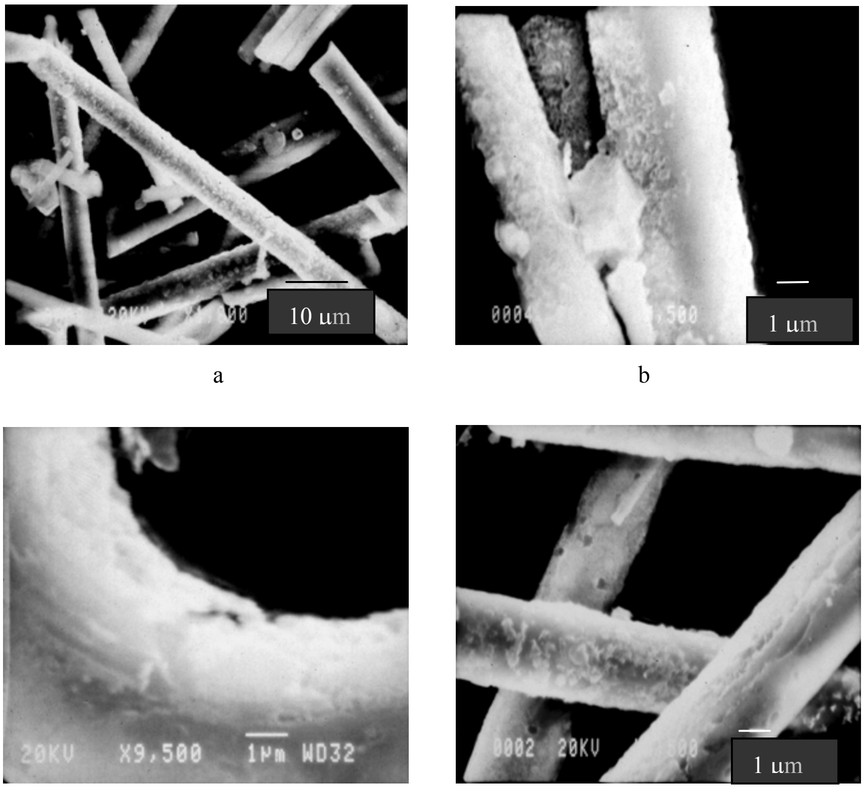


a



b

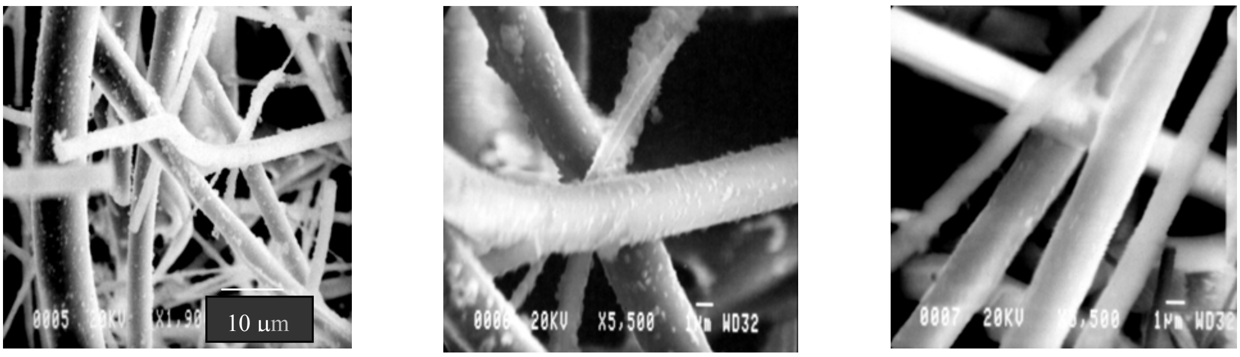
Fig. 5. Mineral wool fibre affected by saturated $Ca(OH)_2$ solution at autoclave conditions: a – general view, b – corroded fibres



a b

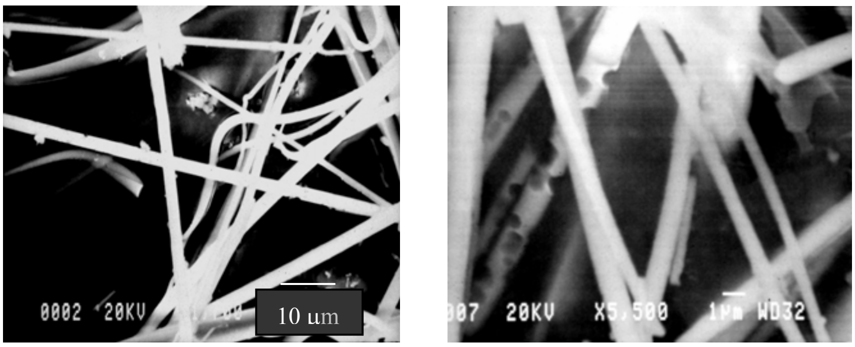
c d

Fig. 6. Mineral wool fibre affected by saturated water extract of Portland cement at autoclave conditions: a – general view; b, c, d – corroded fibres



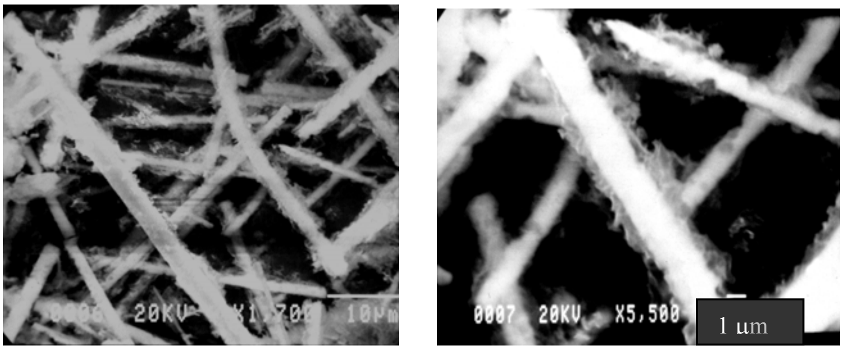
a b c

Fig. 7. Kaolin wool fibre affected by NaOH solution at autoclave conditions: a and b – 0.12 M NaOH, c – 0.04 M NaOH



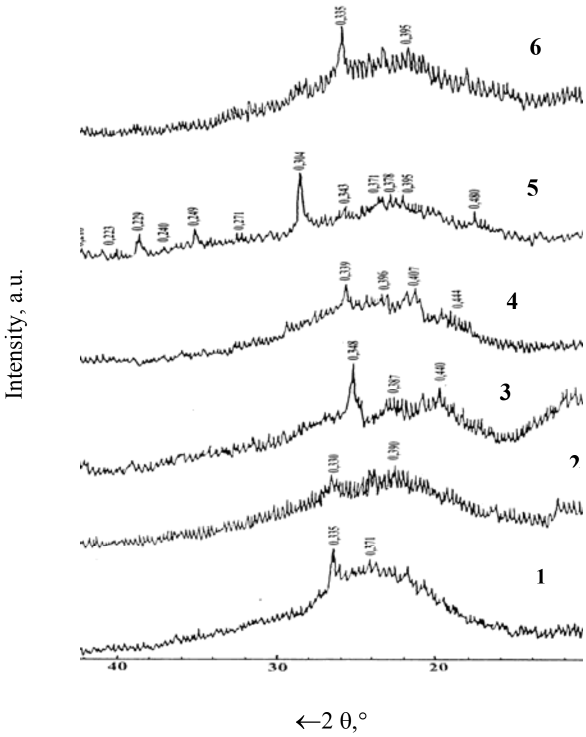
a b

Fig. 8. Kaolin wool fibre affected by saturated $Ca(OH)_2$ solution at autoclave conditions: a – general view, b – corroded fibres



a b

Fig. 9. Kaolin wool fibre affected by saturated water extract of Portland cement at autoclave conditions: a – general view, b – corroded fibres



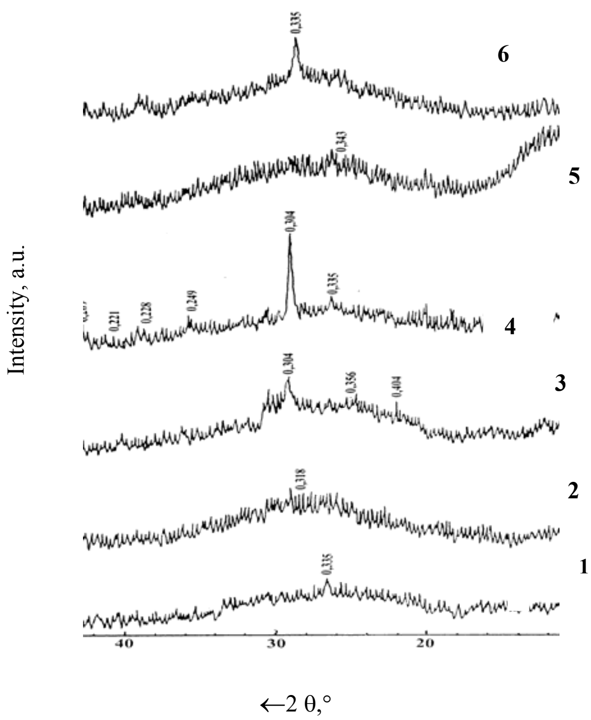


Fig. 10. XRD patterns of kaolin fibre (peaks are noted by the interplanar distance expressed in nm): 1 – as produced, 2 – 6 after interaction with the alkaline solutions: 2 – 0.12 M NaOH (t = 50 °C; τ = 7 days); 3 – 0.12 M NaOH; (after autoclaving t=180 °C; P = 1.0 MPa, τ = 8 h); 4 – 0.04 M NaOH (after autoclaving); 5 – saturated $Ca(OH)_2$ solution (after autoclaving); 6 – extracted from Portland cement (after autoclaving)

Fig. 11. XRD patterns of mineral wool fibre (peaks are noted by the interplanar distance expressed in nm): 1 – as produced, 2 – 6 after interaction with the alkaline solutions: 2 – 0.12 M NaOH (t = 50 °C; τ = 7 days); 3 – 0.12 M NaOH; (after autoclaving t = 180 °C; P = 1.0 MPa, τ = 8 h); 4 – 0.04 M NaOH (after autoclaving); 5 – saturated $Ca(OH)_2$ solution (after autoclaving); 6 – extracted from Portland cement (after autoclaving)

The results of SEM investigations show that silicate fibre is not stable to the alkaline solution attack. As the result of its interaction with this solution a layer of the corrosion products is formed on the fibres surfaces.

The XRD results confirmed the results of SEM investigations. The main products formed during mineral wool and kaolin wool fibres chemical interaction with the alkaline solutions are amorphous. (Fig. 10 and Fig. 11).

In the case of the kaolin wool fibre interaction with NaOH (Fig. 7 and Fig. 10) at autoclaving conditions the purely crystallized products which may be identified are similar to $Na_2Al_2Si_2O_8 \cdot H_2O$ (0.295 nm; 0.340 nm and 0.430 nm).

In the case of the products formed during kaolin wool interaction with 0.04 M NaOH the values of interplanar distances are 0.339 nm and 0.444 nm, in the case of interaction with 0.12 M NaOH – 0.348 nm and 0.440 nm.

The results of the investigations show that the intensity of silicate wool corrosion in the alkaline solutions is extremely increased at autoclave conditions.

CONCLUSIONS

1. The investigated fibres after exposure for 7 days ($t = 20$ °C), in different alkaline media (pH up to 13.1) remained intact. After similar condition only at $t = 50$ °C, it was noticed that only destruction of mineral wool began in 0.12 NaOH solution.

2. Autoclave treatment ($t = 180$°C, $P = 1.0$ MPa, $\tau = 8$ h) of fibres, intensified the caustic corrosion process – destruction of kaoline and mineral wool was noticed in all alkaline media (pH from 12.6 to 13.1).

3. It was determined that the kaolin wool fibres during autoclave treatment react with NaOH solutions and form sodium alumosilicates with a composition similar to $Na_2AL_2Si_2O_8 \cdot H_2O$. The fibres become fragile and upon touching breaks-up. That is why it is not recommended for use as an additive for porous concrete mixtures.

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