Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

24: 2 (2024) 155-163

COMPOSITES THEORY AND PRACTICE

ISSN (online): 2299-128X

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Received (Otrzymano) 09.05.2024

THE INFLUENCE OF VARIABLE TEMPERATURE CONDITIONS ON SELECTED PROPERTIES OF RESIN MORTARS MODIFIED WITH RECYCLED MATERIALS

https://doi.org/10.62753/ctp.2024.10.2.2

Polymer composites have a number of valuable properties that enable them to find a special place in the field of civil engineering. In response to current trends related to sustainable construction and the circular economy, effective ways of modifying these composites with recycled materials are being sought. This approach, in turn, makes these materials require systematic monitoring of their durability, especially in the context of variable operating temperatures. The article describes the results of the testing of concrete-like epoxy composites modified with waste materials, both within the resin matrix – by glycolysate obtained on the basis of propylene glycol and poly(ethylene terephthalate) derived from waste beverage bottles, and aggregates - polyethylene agglomerate from waste bags and waste rubber granules from car tires. After seven days of maturation in laboratory conditions, the mortar samples were exposed in a climatic chamber to cyclic (50 and 100 cycles) temperature changes ranging from +20 °C to +100 °C and from –10 °C to +10 °C. Then, strength tests, changes in the mass and adhesion to cement concrete were carried out. The proposed material solution, combined with the conducted set of tests, brings scientific novelty to the field of building composites. The test results confirm the beneficial effect of modification, especially when it took place in the resin matrix. Positive temperature cycles resulted in post-hardening of the samples and thus an increase in the strength characteristics. The impact of negative temperatures was more unfavorable for the material; after 100 cycles, in most cases, a slight decrease in the mechanical parameters was noted, while the adhesion remained at a very good level, twice as high as that required for repair systems.

Keywords: **polymer composites, epoxy mortars, strength parameters, pull-off strength, thermal degradation**

INTRODUCTION

Epoxy mortars consist of a mixture of synthetic resin (usually epoxy or polyester) as a binder, its hardener and fine aggregate, or other additives [1-5]. These materials can be classified as concrete-like polymer composites [6]. They are characterized by a number of very beneficial properties, including: excellent mechanical parameters, low shrinkage and water absorption, very good chemical resistance and adhesion to various materials, including cement concrete [7-11]. However, these composites are not without disadvantages, including: limited thermal durability, i.e. the ability of a material to retain its physico-mechanical properties during and after exposure to severe thermal conditions. This reduced heat resistance and susceptibility to the aging of polymer composites results mainly from the presence of a resin binder and depends on the type of resin and the hardener used [2, 12-14]. In the case of epoxy and polyester concretes, the maximum operating temperature for long-term exposure is approximately 60 °C, while for short-term exposure it is approximately 100-120 °C [15]. High sensitivity to temperature is related to the viscoelastic properties of polymers, the mechanical parameters of which may change significantly depending on the temperature, especially in the glass transition temperature (T_g) range [16-18]. These relationships are largely known for standard resin composite compositions, but modification of these materials, among others, by waste materials necessitates monitoring and re-characterization of their properties.

The available literature mainly contains information on comparative studies of the impact of thermal effects on the strength characteristics of resin composites, primarily polyester and epoxy [19]. It is worth noting that the most common approach to aging tests also concerns a limited range of temperatures, the lower range of which is usually ambient temperature. Although the data enabling the assessment of frost resistance indicate that this property in the case of polymer concretes is satisfactory and even after 500 cycles of freezing and thawing, the strength decreases by a maximum of 10-20 % [20]. However, it is worth conducting research also for temperatures lower or even much lower and much higher than ambient temperature [21, 22], if only because of the application possibilities of this type of composites, among others, for quick repairs, for the production of elements used in road and bridge drainage systems as well as surfaces, floors and coatings [23-28].

Ribeiro et al. [21] conducted comparative studies of the influence of the thermal effects on the flexural strength of mortars obtained using epoxy and polyester resin. The same work also cited the research results of other scientists who addressed similar topics. Based on them, it can be concluded that at temperatures close to room temperature, the compressive strength practically does not change, while the flexural strength decreases slightly. Nevertheless, in the case of much higher temperatures (60-200 °C), a significant decrease in these strength parameters is observed. Nonethless, these results were obtained for samples previously post-hardened at the temperature of 80 °C.

Taking into account the fact that it is not always possible to harden the material at the place of production and use, it was decided to test samples of mortars that matured only in natural conditions. The research described in this article is part of a broader campaign of aging tests designed for and conducted on epoxy mortars modified with recycled materials. A series of control samples as well as samples in which 9 wt.% of the epoxy binder was replaced by a glycolysate based on propylene glycol and poly(ethylene terephthalate) derived from waste beverage bottles were prepared. Additionally, in two subsequent series of mortars, apart from the partial replacement of the binder, part (10 % by volume) of the quartz aggregate was also replaced, respectively, by a polyethylene agglomerate from waste plastic bags or waste rubber granules from car tires. The detailed method of obtaining the mortars was described in our earlier article [29], which presented the results of accelerated aging tests in a chamber simulating radiation and rain. This time, samples of the obtained mortars were exposed in a climatic chamber to cyclical (50 and 100 cycles) temperature changes ranging from +20 °C to +100 °C and from –10 °C to +10 °C. Then, strength tests, changes in mass and adhesion to the concrete substrate were carried out, as well as analyses using SEM. The proposed method of modifying epoxy mortars, combined with the investigated set of properties, allows the authors to supplement the literature base with new information on resin composites and expand the source database. Thus, it can also be a valuable source of information for designers and contractors using this type of composites.

MATERIALS AND METHODS

The main part of the binder in the mortars was Epidian 5 epoxy resin (CIECH Sarzyna SA, Nowa Sarzyna, Poland). Triethylenetetramine (Z-1) was used as the hardener, in each case in an amount of 10 % by resin mass. The aggregate was standard quartz sand, grain size 0-2 mm. A constant binder/aggregate volume ratio was assumed – B/A = 0.58. This basic composition was modified and, as a result, four series of mortars with different compositions were obtained:

c (control): binder – epoxy resin; hardener; aggregate – standard quartz sand.

PET: binder – 91 % (by mass) epoxy resin + 9 % (by mass) PET glycolysate; hardener; aggregate – standard quartz sand.

PE: binder – 91 % (by mass) epoxy resin + 9 % (by mass) PET glycolysate; hardener; aggregate – 90 % (vol.) standard quartz sand + 10 % (vol.) PE waste agglomerate.

R: binder – 91 % (by mass) epoxy resin + 9 % (by mass) PET glycolysate; hardener; aggregate – 90 % (vol.) standard quartz sand + 10 % (vol.) waste tire rubber granulate.

After demoulding, all the samples were aged for 7 days in laboratory conditions (21±2 °C, 35±5 % RH), then one batch was subjected to planned physical and mechanical tests, and four batches were subjected to two types of thermal aging cycles: freezing and thawing and positive thermal cycles, the detailed characteristics of which are presented in Figure 1.

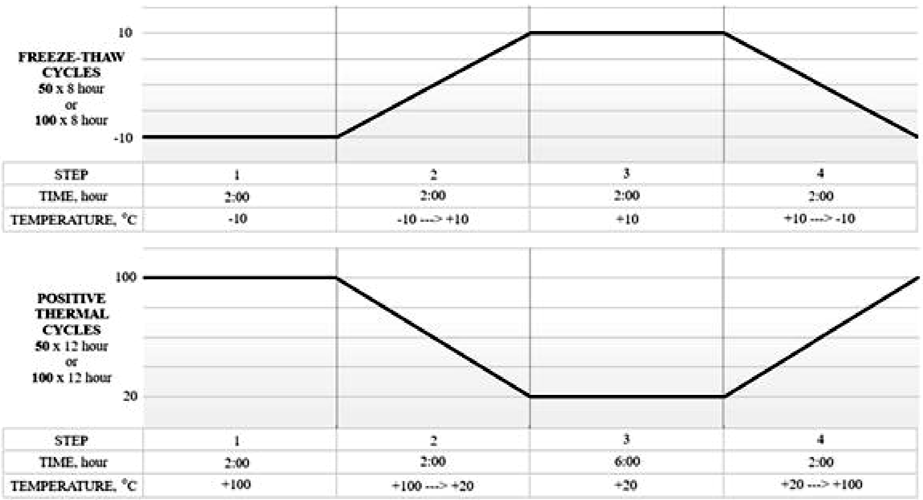

Fig. 1. Characteristics of performed thermal cycles

Analogous temperature ranges to those described in the works of Ribeiro et al. [21, 22] were used, which allowed comparison of selected research results. At the same time, contrary to the tests described in [17, 21, 30], no additional post-hardening of the samples was carried out after demoulding.

The testing of the samples subjected to cyclic temperature changes was carried out using an Espec PL-2J climatic chamber, ESPEC CORP. Japan (Fig. 2) enabling programming of the assumed thermal cycles.

The change in mass for each type of mortar subjected to cyclic freezing and thawing was calculated as average values from 3 samples using Equation (1):

$$\Delta m\,(\%) = \frac{mf - mi}{mi} * 100 \tag{1}$$

where m_f – final mass – sample mass after completing 100 freeze-thaw cycles; m_i – initial mass – sample mass before the start of 100 freeze-thaw cycles.

The strength parameters of the samples were determined for each type of mortar after the end of a given seasoning cycle on testing machines equipped with appropriate test inserts (Fig. 3). The flexural strength was tested on a 150 kN testing machine, QC-505B1 COMETECH TESTING MACHINES CO., LTD Taiwan, with an assumed load increment of 0.25 mm/min, on 3 samples for each type of mortar and thermal load cycle. The compressive strength was determined using a 1500 kN hydraulic press, C6/4 MATEST, Italy with an assumed load increment of 2.4 kN/s on 6 samples for each type of mortar and thermal load cycle.

Pull-off adhesion fh was determined in accordance with the PN-EN 1542 standard [31]. The adhesion measurements were made by peeling off the resin mortar applied to concrete blocks of strength class C16/20. Before applying the mortar, the blocks were surface treated and cleaned with a wire brush. The thickness of the applied mortar was 5 mm. The test method involved direct detachment of steel discs glued to the surface of the epoxy mortar using a two-component epoxy adhesive. The test area was marked by appropriate drilling of the surface. The test was carried out by means of a Dyna Pull-off Tester (Fig. 4a) using steel disks with a diameter of 50 mm and a thickness of 20 mm (Fig. 4b, 4c).

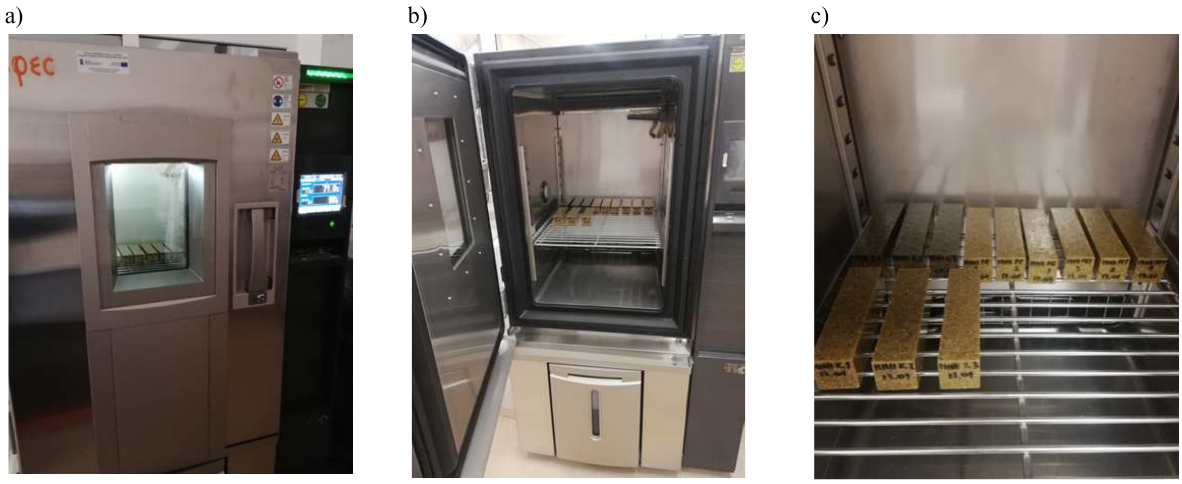

Fig. 2. Climatic chamber a) during work, b) after work, c) interior of chamber with mortar samples

Fig. 3. Machines for testing a) flexural and b) compressive strength

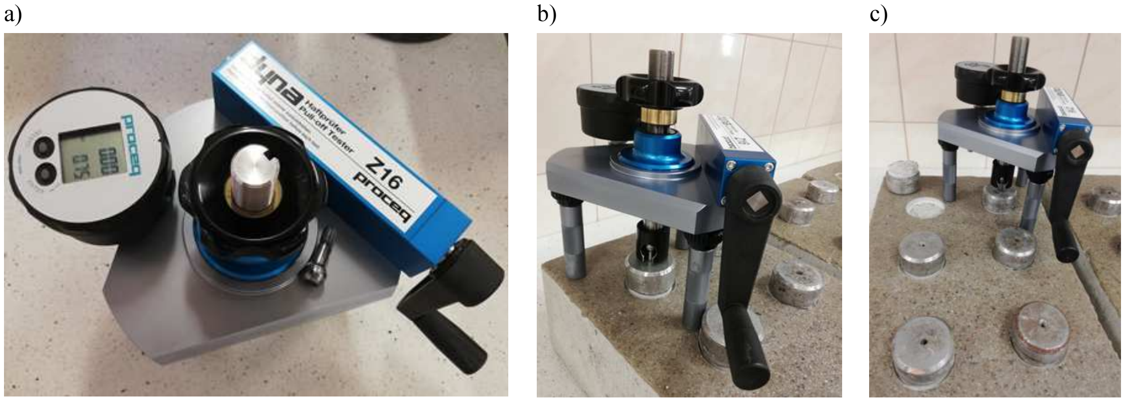

Fig. 4. Pull-off test device: a) general view, b) and c) during measurement

The microstructure of the samples after the freeze-thaw cycles was evaluated employing scanning electron microscopy (SEM) analysis with secondary electrons (SE). VEGA3 equipment from TESCAN was utilized for this purpose.

RESULTS AND DISCUSSION

Strength parameters

The results of the flexural strength tests (average values and standard deviation) obtained for individual types of mortars, taking into account the number of cycles and the method of thermal aging, are shown in Figure 5.

The average of the compressive strength test results with standard deviation obtained for individual types of mortars, taking into account the number of cycles and the thermal aging method, are presented in Figure 6. A summary of the calculated changes in flexural and compressive strength for each type of mortar, number of cycles and thermal aging method is included in Table 1.

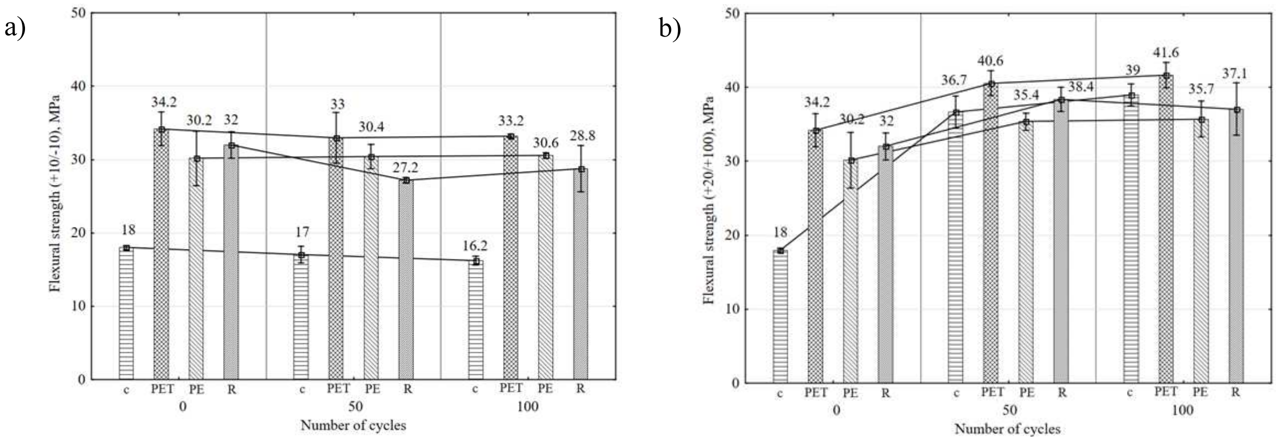

Fig. 5. Summary of flexural strength test results: a) cyclic freezing-thawing, b) positive thermal cycles

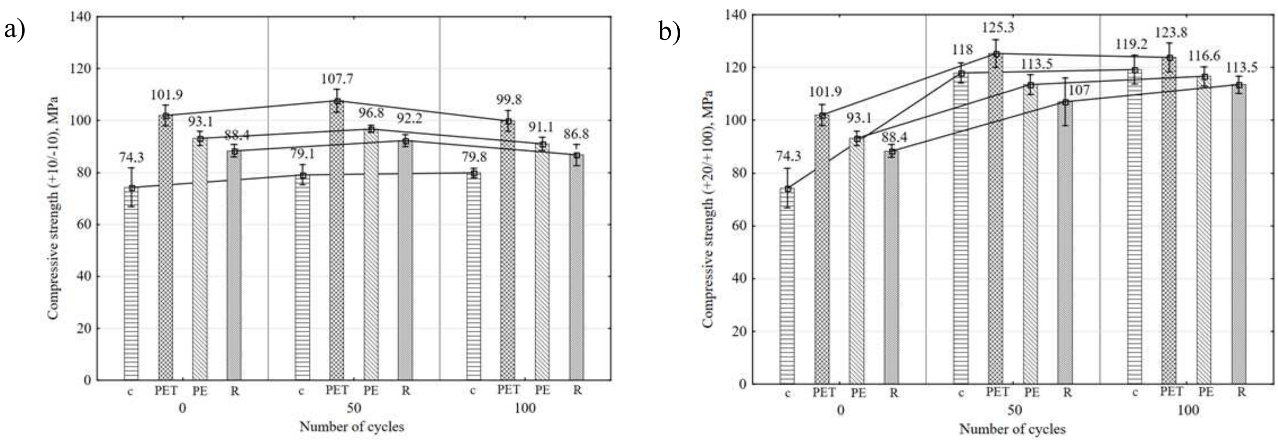

Fig. 6. Summary of compressive strength test results: a) cyclic freezing-thawing, b) positive thermal cycles

TABLE 1. **Summary of changes in flexural and compressive strength obtained for each type of mortar, number of cycles and thermal aging method**

Mortar type	Flexural strength change Δf_f [%]				Compressive strength change Δf_c [%]			
	50 cycles		100 cycles		50 cycles		100 cycles	
	–10/+10	+20/+100	–10/+10	+20/+100	–10/+10	+20/+100	–10/+10	+20/+100
c	–5.6	103.9	–10.0	116.7	6.5	58.8	7.4	60.4
PET	–3.5	18.7	–2.9	21.6	5.7	23.0	–2.1	21.5
PE	0.7	17.2	1.3	18.2	4.0	21.9	–2.1	25.2
R	–15.0	20.0	–10.0	15.9	4.3	21.0	–1.8	28.4

Based on the test results presented in Figures 5 and 6 and in Table 1, it can be concluded that the type of mortar and the method of thermal load differentiate the strength values. For the samples subjected to positive temperature cycles, an increase in flexural and compressive strength was noted both after 50 and 100 thermal cycles. This initial improvement in strength results from the continuation of hardening processes and is most visible for control mortars (c) – after 100 cycles, the increase in flexural strength was over 116 %, although these parameters are lower than those obtained for the mortar modified by glycolysate obtained on the basis of waste PET, which proves the beneficial effect of modifications within the resin matrix. At the same time, by using cyclic freezing and thawing in the assumed temperature range of –10°C/+10°C, it is not possible to achieve such an effect, and only for the compressive strength determined after 50 cycles is there a noticeable rise in this parameter in the range of 4.0-6.5%. Damage caused by cyclic freezing and thawing may be caused primarily by an increase in the volume of water freezing in the pores of the material. However, for damage to occur locally, at least a critical degree of water saturation must be achieved, and resin mortars have low water absorption. Nevertheless, it is possible for water to be absorbed during exposure to freeze-thaw cycling.The flexural strength of epoxy mortars obtained before thermal aging by Ribeiro et al. [21] is at the level of 40 MPa and is higher than the flexural strength of the epoxy mortars shown in Figure 5. This behavior results from the preliminary post-hardening of the samples by Ribeiro et al. As the curing temperature increases, the strength grows and the curing time becomes shorter, which can be explained by the fact that the raised temperature in the epoxy resin molecule increases its functionality. The chemical reaction between the epoxy group and the hydroxyl group occurs slower at lower temperatures. The reason for this is the inability to further react with the second epoxy chain, the so-called a diepoxy group formed in the reaction of an amine hardener with an epoxy group. The use of increased temperature leads to a situation in which the same molecule is theoretically bifunctional and becomes tetrafunctional, and the increment in temperature allows growth in the rate of reaction progress [32]. The curing temperature of the epoxy resin affects the final mechanical properties of the composite in a non-monotonic manner. In particular, raising the hardening temperature to the glass transition temperature (T_g) may lead to greater strength and stiffness, but also to an increase in T_g [9]. At the same time, after exceeding T_g, the mechanical properties begin to deteriorate owing to the increment in the randomness of adhesive cross-linking, oxidative cross-linking and degradation of the polymer structure [18]. With long-term exposure to increased thermal energy, the matrix may undergo oxidation and thermal decomposition, which results in increased brittleness and thus reduced strength of the composite. This behavior can be observed in the work of Ribeiro et al. [21], where with positive thermal cycles the flexural strength decreased by 14 % for 50 cycles and by 75 % for 100 cycles.

The nature of changes in strength during heat aging does not always have to be negative from the beginning of exposure to elevated temperatures, which is confirmed by the results of our research. Initially, growth in strength is observed, and only after exceeding the limit number of cycles may the strength decrease. The heat resistance characteristics of composites reheated during curing, as a consequence of better polymer cross-linking density parameters, are usually more favorable than those hardened longer, but at ambient temperature. Nonetheless, taking into account the difficulties with initial hardening in some applications, it is worth considering the possibility of using the modified mortars proposed by our team in such situations. PET mortars seem particularly interesting, with results similar to or even higher than those presented by Ribeiro et al. After 100 positive cycles, the flexural strength of these compositions continued to rise and reached an average value of 41.6 MPa, while for the mortars tested by Ribeiro et al. a decline in strength was recorded to 30.8 MPa. Mortars modified simultaneously by PET glycolysate and waste PE or rubber granulate after 50 and 100 positive thermal cycles did not obtain such high values of flexural strength as the PET and C mortars, but they were also higher than those presented in the work of Ribeiro et al. This fact is extremely important in the environmental context; it confirms the possibility of the disposal of burdensome waste while maintaining the required durability of the composite.

In [13] it was indicated that, depending on the sample exposure temperature, at temperatures lower than 150°C an increment in the residual compressive strength can be observed, which can be explained by subsequent hardening and an increase in cross-linking density. In turn, above a temperature of 150 °C, thermo-oxidative degradation of the polymer binder and separation of the binder from the aggregate may occur. If changes are visible, it is for samples subjected to flexural strength tests rather than compression tests. Cycles of heating and cooling cause the polymer to harden and increase the compressive strength. With a greater number of positive thermal cycles, yellowing of the mortars is also noticeable.

Mass change and pull-off test

The average results of determining the change in sample mass and adhesion to the substrate before and after 100 freezing and thawing cycles are shown in Figures 7a and b, respectively.

After 100 freeze-thaw cycles, slight positive changes in the mass of the samples can be observed, not exceeding 0.0033 %, and in the case of control mortars – 0.002 %. In the work of Ribeiro et al. with a similar number of cycles, a change in the mass of the epoxy samples was recorded at the level of 0.017%, which is approximately 10 times higher. The mass gain can be associated with the adsorption of moisture in the pores of the material and on the aggregate particles [33]. The average adhesion was calculated based on six results obtained for each type of mortar.

In each case, a cohesive failure model was observed in the concrete substrate (Fig. 8). Therefore, it can be concluded that the adhesion is higher than the marked value, which, depending on the mortar used, ranges from 2.87 to 3.7 MPa, and is much higher than the value of 1.5 MPa required in repair systems. The cyclic freezing and thawing could have had an impact on the weakening of the repaired material itself, i.e. cement concrete.

SEM

Microphotographs showing the fractures of the mortar samples obtained in the flexural strength test after 100 freeze-thaw cycles are presented in Figure 9. It can be seen that despite the cyclic temperature changes in samples C and R, the aggregate particles are well bonded to the resin matrix; most of the contact surfaces are in good condition. For the PET samples, there are gaps in some places between the matrix and the aggregate, which could have resulted in a recorded decrease in compressive strength after prolonged (100 cycles) exposure to variable (positive and negative) temperatures.

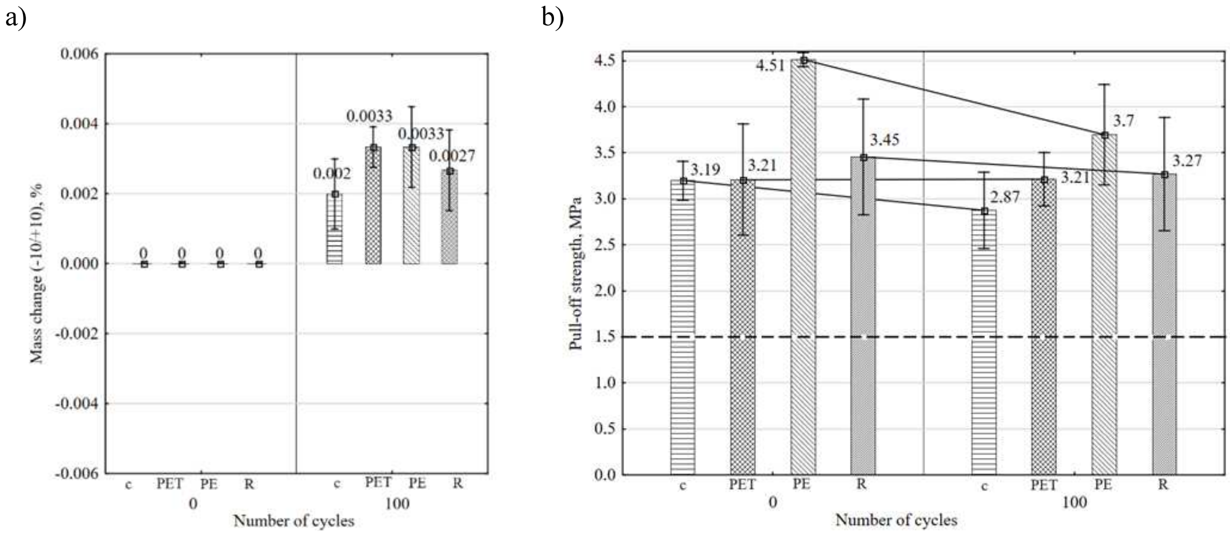

Fig. 7. Comparison of test results of samples before and after 100 freeze-thaw cycles: a) change in mass, b) adhesion to concrete

Fig. 8. Adhesion test to concrete: a) sample during test, b) sample after test, c) enlargement – visible cohesive model of detachment in concrete substrate

a) b) c)

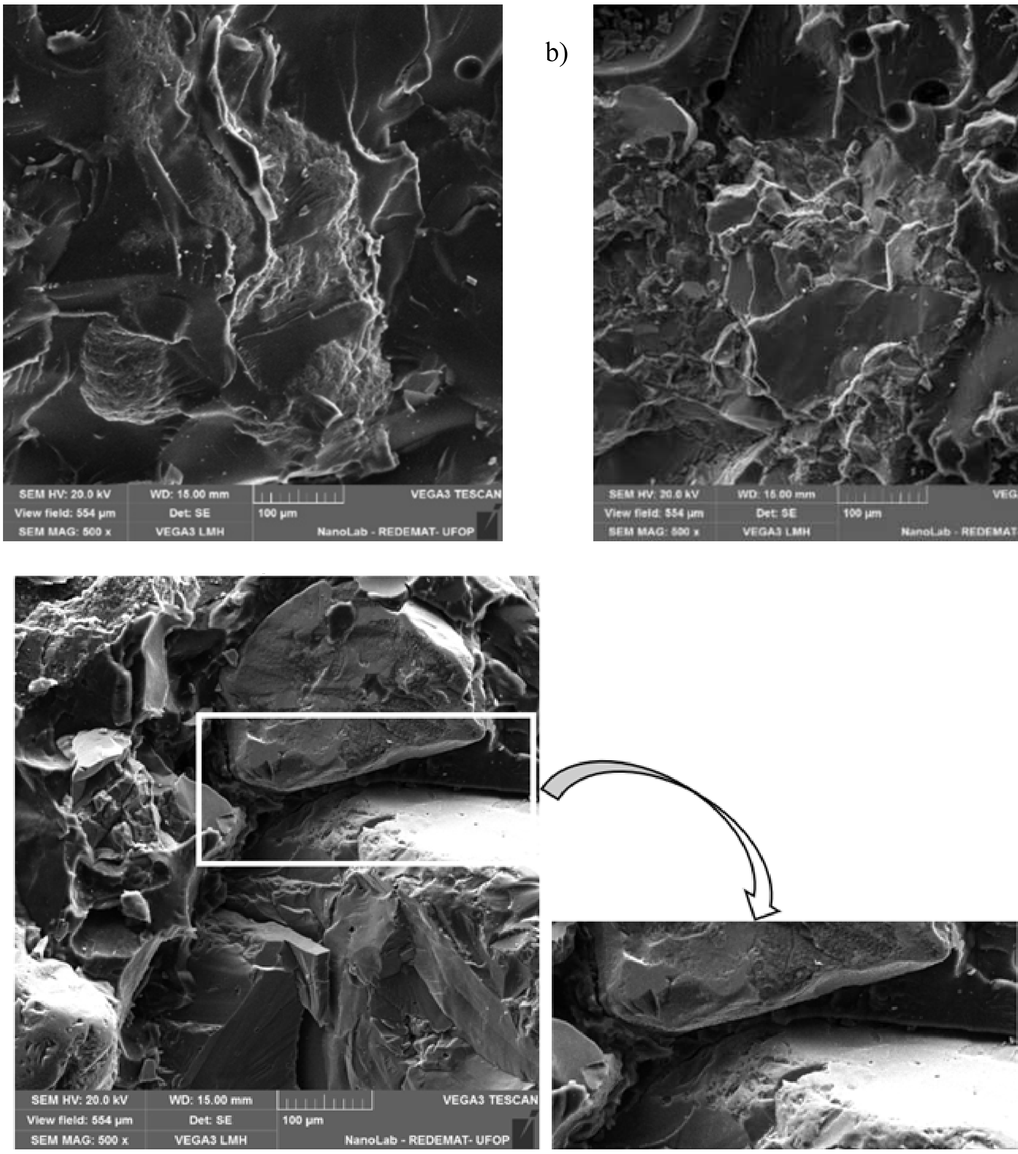

Fig. 9. Microphotographs of mortar samples after 100 freeze-thaw cycles a) C, b) R, c) PET

CONCLUSIONS

Polymer composites used in civil engineering are exposed to periodic environmental loads during operation, which can be simulated using thermal cycles. The article describes the test results of four types of epoxy mortars modified with recycled materials, subjected to cyclical temperature changes, both in the area of positive, high temperatures and negative temperatures. Based on these results, the following conclusions can be drawn:

- In the case of the epoxy mortars cured only in natural conditions (without additional post-hardening), modification of their composition by waste materials allows much higher strength parameters to be to obtained compared to unmodified mortars.
- The cyclical impact of the positive temperatures in the range of +20/+100 °C causes an increment in the flexural and compressive strength of all the types of mortars obtained, especially the unmodified ones, which is related to the continuation of hardening processes.
- For the designed mortars, even after exposure to 100 positive thermal cycles, the strength parameters are higher than before exposure, and in the case of the PET mortars, the flexural strength of 41.6 MPa is also higher than the strength of 40.7 MPa obtained before exposure by other authors conducting additional post-curing.
- Cyclic freezing-thawing causes a slight reduction in the strength of the designed mortars, but for the composites containing waste, these properties are still at a high level in the range of 27.2-33.2 MPa for flexural and 86.8-107.7 MPa for compression, depending on the type of modifier and the number of cycles.
- Mortars exposed to 100 temperature cycles in the range of –10/+10 °C are characterized by a negligible change in mass and very good adhesion to concrete.
- The changing technological and production conditions of polymer composites constitute the basis for continuing research on these materials. Due to the presence of a resin matrix in this type of mortars, it

seems particularly important to extend the research on these composites to include parameters related to brittle fracture.

Acknowledgement

The research was funded by ***the National Science Center Poland*** *as part of the* ***MINIATURA 5 competition****, the project entitled "Study of the impact of accelerated aging on the physical and mechanical properties of resin composites containing waste materials", application registration number 2021/05/X/ST5/00325.*

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