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Monitoring the long-term durability and microstructure of polymer concrete patching materials with high content of secondary raw materials using advanced methods

J Hodul, L Meszarosova and R Drochytka

Institute of Technology of Building Materials and Components, Faculty of Civil Engineering, Brno University of Technology, AdMaS Centre, Veveri 331/95, Brno 602 00, Czech Republic

Abstract. For commercially produced polymer systems, only primary raw materials are used as fillers. These fillers are predominantly pure silica sand of various grading. The substitution of primary materials with secondary raw materials, and their efficient use in the manufacture of polymeric repairing substances, is an indisputable advantage of the use of secondary raw materials. There is currently no general methodology for monitoring the long-term durability and microstructure of repairing substances on a polymeric basis. In this article, long-term durability was assessed by subjecting developed substances with a high content of secondary raw materials (60% fly ash, 75% glass recyclate) to freezing cycles and subsequent cohesion testing. Additionally, through exposure of a concrete element reprofiled by the developed substance, the weather effects for approximately one year. The microstructure of polymeric repairing substances cannot be observed by the same methods used for silicate substances. With regard to the type of tested substance and the filler used, advanced CT tomography and high resolution optical microscopy were chosen for monitoring the microstructure. Based on the results of the tested substances cohesion with the underlying concrete following the freezing cycles, and the evaluation of the taken images, it can be stated that the test repairing substances have excellent long-term durability and can be used in different applications. Furthermore, it was confirmed that replacing the primary filler with the secondary raw material does not reduce the long-term durability of the polymeric repairing substances on an epoxy basis. It was confirmed that only slight degradation occurs due to the photooxidation of epoxy resin by UV radiation, but this does not have a significant effect on physical and mechanical properties.

1. Introduction

In recent years, the use of polymer substances in the field of repairing substances has become widespread in the building industry. All industrially manufactured repairing substances use primary raw materials. The use of a polymer binder, together with secondary raw materials, is therefore an important topic.

Vipulanadan and colleagues concluded that the optimal amount of polymer binder in a mixture ranges from 14% to 16% [1-3]. Regarding the use of secondary raw materials in the epoxy matrix, Singl and Chawl traced a positive contribution alongside the use of fly ash. They found that with an increasing addition of fly ash (in the range of 30% to 54%), the strength of the resulting composite increased [1, 4]. Fly ash is a by-product of coal combustion in thermal power plants and is rich in silicon dioxide (SiO_2), iron oxide (Fe_2O_3), and aluminium oxide (Al_2O_3) [5]. Its use in polymer matrix (comprising polyethylene terephthalate, polyester, or polyoxomethylene composites) was addressed by



Chand, Li, Koszkul, Kwiatkowski and Baheti [1,6-8]. This paper follows up their research and its partial aim is to explore the possibilities of using fly ash after denitrification of flue gas process and waste container glass.

2. Materials

Following the work of Singla and Chawl [4], the possibility of using fly ash in the epoxy matrix was investigated. Compared to the work of these researchers, the reprofiling material was, from an economic and ecological point of view, filled with the maximum possible quantity of secondary raw materials as it concerns the workability and preservation of important properties.

2.1. Composition of the polymer concrete patching material (PM)

The tested polymeric repairing substances contained a bisphenol A-based epoxy resin (P-A) as a binder. The polymer binder is composed of component A, which is an epoxy monomer (epoxy resin), and hardening agent B based on aliphatic and aromatic amines. Two substances, PM-P (Premium) and PM-E (Economy), were tested for long-term durability and microstructure. The composition of these substances is shown in table 1.

Table 1. The composition of tested polymer concrete patching materials.

Formulation	Binder	Filler
PM-P	40 % P-A	60% filter fly ash devalued by the denitrification process
PM-E	25 % P-A	75% crushed transparent glass recyclate (0-1.5 mm)

2.2. Properties of input raw materials

A two-component, low viscosity solvent-free epoxy-based substance with a density of 1.1 kg/l was used as a binder. Its workability ranged from 15 minutes to 20 minutes. The modulus of elasticity is 3000 N/mm², elongation at break is 4% to 7%, and compressive strength ranges from 80 MPa to 90 MPa. This resin is primarily used for repairing mechanically demanding parts, and to repair locations exposed to thermal and chemical stress.

The fly ash, which was influenced by the denitrification of the flue gas was used as a filler in one material (PM-P). The fly ash used was generated by the combustion of pulverised coal in a thermal power plant at high-temperature, in the north of Bohemia. Due to the new technology of flue gas denitrification, ammonium ions are present in fly ash created in the power plant. In the second substance, glass recyclate was used as a filler. These are grounded glass fragments of transparent container glass, collected during waste separation.

Table 2. Chemical composition of the fly ash (filler to PM-P material).

Parameter	SiO ₂	CaO	Total organic carbon (TOC)	Ammonium ions	Sulphates
Amount	53.40%	4.13%	5.20%	3.79 mg/kg dry	0.25%

Table 3. Chemical composition of the glass recyclate (filler to PM-E material).

Parameter	SiO ₂	Na ₂ O	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	K ₂ O	BaO
Amount	74.80%	11.91%	7.69%	4.27%	0.55%	0.17%	0.19%	0.09%

3. Methods

For cured epoxy repairing polymer concrete patching mortar, the effect of cyclic temperature change (cyclic freezing and defrosting) was monitored over a total of 300 hours. The microstructure was also monitored by means of an optical microscope and computer tomography (CT).

3.1. Determination of adhesion following freezing cycles

Samples coated with a thin layer of tested substances were first saturated with water and subsequently subjected to cyclic temperature changes in the range of -15°C to 20°C . One cycle lasted six hours. During this time, four hours were employed for cooling to -15°C and two hours for heating to 20°C . The substances were exposed to 50 freezing cycles.

3.2. Determination of the microstructure

Monitoring of the microstructure of the tested polymeric repairing substances was performed by computed tomography (CT) and a high resolution optical microscope.

3.2.1. Optical microscope. The developed polymer concrete patching mortars (PM-P, PM-E) were monitored by an optical microscope for effects of weathering. Samples of individual substances were taken and subsequently observed with an optical microscope.

3.2.2. CT Tomography. Using special computed tomography (CT) designed for building materials, it was possible not only to observe the microstructure of the test samples, but also to consider the internal structure and distribution of pores and filler. In our case, whether the influence of weathering on the reprofiled part of the concrete curb degraded the polymeric repairing substance was monitored. As a tomographic sample, a cut-off section of the reprofiled corner of the concrete curb was used (see figure 1 and figure 2).



Figure 1. Sample of the PM-E patching mortar for CT Tomography.

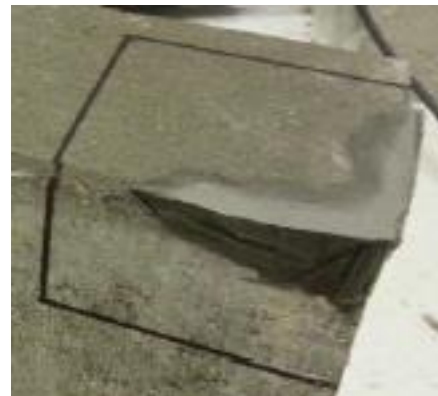


Figure 2. Sample of the PM-P patching mortar for CT Tomography.

The observation primarily focused on structural failures, particularly crack formation, the separation of the repairing substance from the underlay, the irregular dispersion of filler, and the inhomogeneity of the developed polymer patching mortar.

4. Results and Discussion

4.1. Determination of the adhesion after freezing cycles

The adherence results of the tested repairing mortars after 50 freezing cycles (T50) are shown in table 4. The samples examined showed a breakage in the concrete underlay, which means that the adhesion of the repairing material to the underlay exceeded the cohesion of the underlying concrete. Highest cohesion was recorded for the PM-P substance, namely 2.2 MPa. It can therefore be concluded that developed substances are able to withstand temperature cycles and low temperatures, while

maintaining high adhesion to the concrete underlay. The use of secondary raw materials as fillers did not affect the cohesion of substances following cyclic temperature changes.

Table 4. Results of the adhesion after 50 freezing cycles (T50).

Type of patching mortar	Stress at breakage before T50 [MPa]	Stress at breakage after T50 [MPa]	Place of breakage
PM-P	2.20	1.28	Sub-concrete
PM-E	1.67	1.48	Sub-concrete
PM-REF	1.98	1.02	Sub-concrete

4.2. Determination of the microstructure

4.2.1. *Optical microscope.* From figure 3 and figure 4, it is evident how the epoxy resin, due to its low viscosity, flows into the porous structure of the underlay concrete and thus creates a strong connection at the contact zone. This is very important to ensure good adhesion of the reprofiled mortar to the underlay. The PM-P sample in figure 4 is not sufficiently transparent for viewing in transmitted light, but the interface is visible very well, even in incident reflected light. The figure shows the PM substance in the left lower part, and it is obvious that it is very well grounded in the underlying concrete. In the microscopic observation of the PM-E sample surface (figure 3), it is possible, with transmitted light, to observe in detail the interface of the existing concrete underlay (at the top of the figure), and the polymer concrete patching mortar, due to its high transparency. Furthermore, it is possible to observe the perfect anchoring of the reprofiling substance into the concrete underlay.

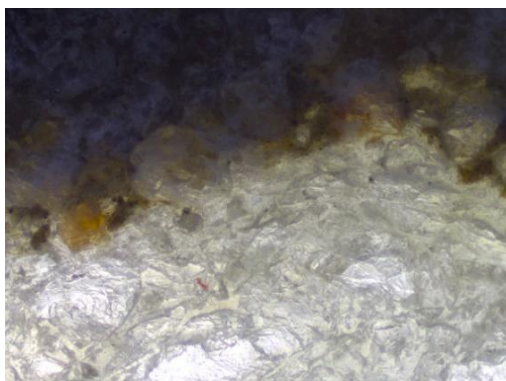


Figure 3. Connection detail of the PM-E reprofiling substance to the concrete base (at the top of the figure) (100x).

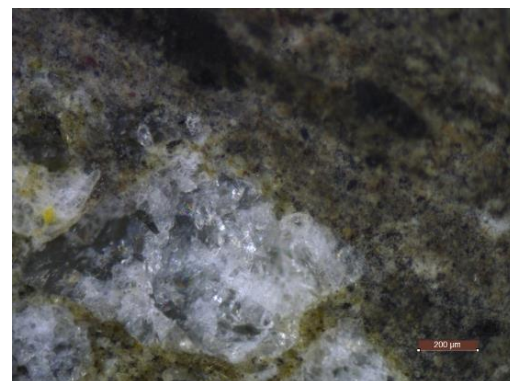


Figure 4. Connection detail of the PM-P reprofiling substance (in the lower left part) to the concrete substrate (100x).

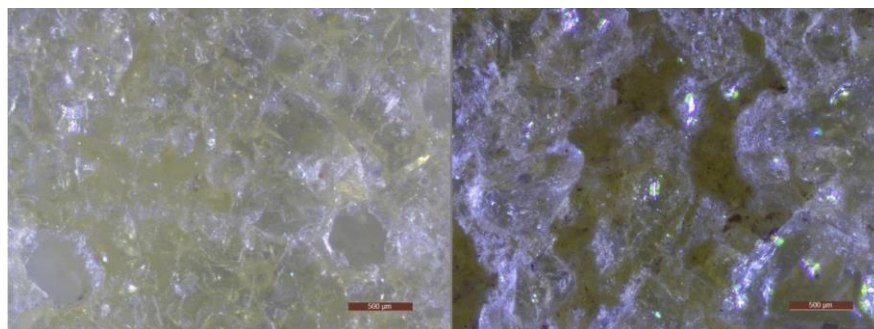


Figure 5. Surface detail of PM-E sample – left sample intact by sunlight (UV rays), UV affected surface can be seen in the right image (50x).

Figure 5 clearly shows the perfect coating of glass shards in an epoxy matrix, which is slightly yellowish. The micrograph in the figure 5 right shows the degradation effect of UV radiation, which affected the applied substance as a result of the exposure of the reprofiled curb to weather effects. Due to the effects of solar radiation, the epoxy matrix decreased slightly as a result of photooxidation (macromolecule breakdown by UV radiation).

Figure 6 on the left shows the microstructure of the PM-P substance surface, where it is possible to observe the uniform distribution of fly ash grains (cenospheres) in the epoxy matrix. In figure 6 on the right, it is possible to observe the apparent change in the surface of the substance due to photooxidation; this is a sample that has been subjected to weathering for 300 hours.

From the microscopic analysis, overall, the results indicate that the effect of atmospheric influences does not produce micro-cracks or the separation of substances from the concrete underlay.

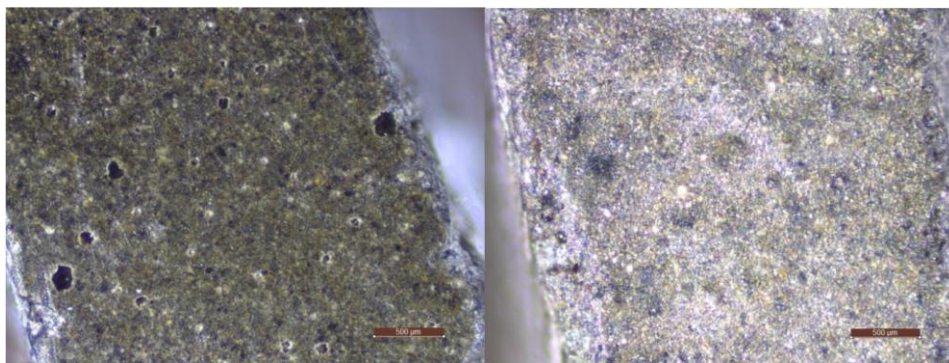


Figure 6. Surface detail of PM-P sample – left sample intact by sunlight (UV rays), UV affected surface by photooxidation can be seen in the right image (50x).

4.2.2. CT Tomography. From a computer tomography (CT) observation (see figure 7 and figure 8), it can be concluded that the patching mortar is compact in both variants on overall cross-section, shows no cracks, tears or higher porosity or separation of the repairing substance from the underlay, compared to the comparative REF samples. Due to the appropriate viscosity, the substance is firmly anchored to the underlay. Additionally, the PM-P substance had a denser and more compact structure compared to the PM-E substance, which can be said to have higher strength and better long-term durability.

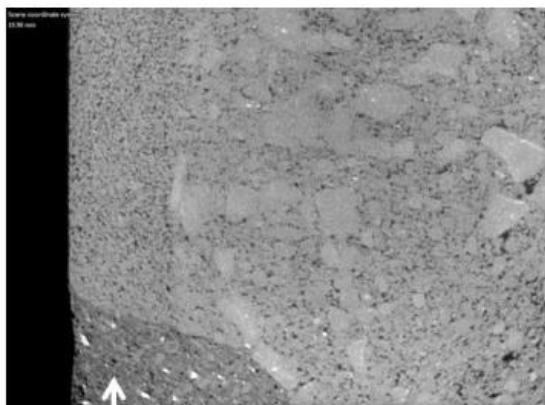


Figure 7. Internal structure of the PM-E substance (indicated by arrow) and detail of connection to the concrete curb.

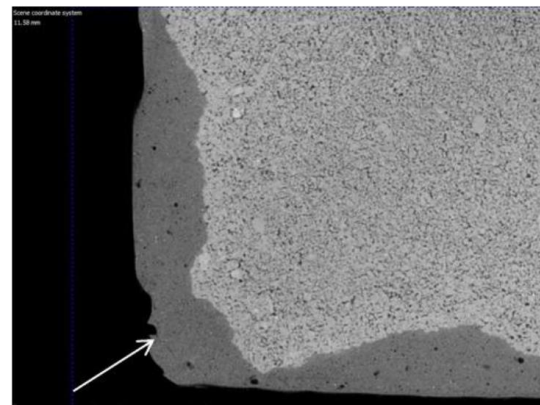


Figure 8. Internal structure of the PM-P substance (indicated by arrow) and detail of connection to the concrete curb.

5. Conclusion

In this paper, the long-term exposure influence of two developed polymer concrete patching mortar samples to atmospheric influences, and the monitoring of their microstructure using progressive analytical methods was investigated. Approximately one year later, the surface was slightly degraded by UV radiation (photooxidation). Based on the visual assessment, slight cracks and wrinkles were also observed. In the case of the substance with waste container glass as a filler, the surface damage was greater than for the substance filled with high temperature fly ash contaminated with ammonium ions. This greater surface disturbance was likely due to the transparency of the glass filler, which allowed for easier penetration of the sun's rays, and more intense UV exposure to the epoxy matrix. It is known that strong UV radiation can weaken chemical bonds in epoxy resin and thus accelerate aging and degradation of the material due to photooxidation. However, this effect did not impact physical and mechanical properties. The substance was subsequently subjected to a microstructure analysis by CT tomography and using an optical microscope. There was no evidence of any substance breakage or loss of cohesion with the sub-concrete in either case. A significant benefit of these patching mortars is the use of a large amount of secondary raw material in the form of fillers. Newly developed polymer concrete patching mortars indicate, in addition to existing commercially produced substances, better physical and mechanical parameters.

Acknowledgments

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