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Polymer based grout with specially treated hazardous waste

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Abstract. The present research verified the possibility of using specially treated hazardous waste (solidification product) derived from a solidification agent (fly ash from fluidized bed combustion (FA)) and hazardous waste (end-product) as a filler in polymeric grout. Hazardous waste was pre-treated by solidification technology, while within the performed research, a total of two solidification paths were verified. One of them was a dry homogenisation (TOS1) and the other technology was wet granulation of the input materials (TOS2). The use of dry homogenization solidification technology, which is economically less demanding, seems to be more advantageous. Using such specially treated filler was verified by some selected tests and the results indicated grout with a relatively high compressive strength of about 50 MPa and a flexural strength of about 20 MPa. The developed polymeric grout also showed a very good adhesion to a cast basalt, which was tested by the pull-off test and subsequently observed in detail by optical microscope.

1. Introduction

To produce high-quality jointing compounds in demanding industrial applications, the most commonly used binder is polymer-based, which is distinguished by very good adhesion to most materials [1]. Some of the best binders are polymeric binders based on epoxy resin (EP), which exhibit excellent adhesion to most materials and, after curing, excellent physical and mechanical properties, minimal shrinkage and high chemical resistance [2,3]. Thus, this current research selected polymeric composition based on epoxy resin (ER) as a binder. Using the ER as a binder for a patching material was also discussed by Hodul et al. [4], wherein they dealt with a possibility of using fly ash contaminated by flue gas denitrification process as filler to polymer repair materials. Hazardous waste (HW) can be used as a filler in polymer compositions. However, it needs to be pre-treated appropriately. The most common type of treatment of HW is solidification and stabilisation (S/S) [5]. It is the mixing of HW with solidification agents that bind dangerous substances into a solid matrix to prevent the release of contaminated components into the environment. The most frequently used reagents are lime, cement or fly ash. This type of treatment has been also studied by Hodul et al. [6] in their experiment, in which the neutralization sludge solidification engaged. Lopéz et al. [7] who tried to stabilise the HW containing large amounts of mercury. The aim is to maximise the use of all waste in order to minimise their very expensive treating and landfill, which contributes to environmental protection. The aim of this work was to verify the possibility of using solidification product formed from selected secondary raw materials such as a solidifying agent and hazardous waste as a filler in



polymeric grout. This material is primarily intended for grouting elements in basalt sewer sewers and certain industrial plants where it will be exposed to a heavy chemical aggressive environment. In connection with the continuous production of hazardous waste (HW) in industry and in the effort to avoid landfilling these materials, we need to find a suitable use of such hazardous waste. HW was treated with the appropriate solidification technology in order to produce suitable fillers useful in polymeric grout. As part of the research, a total of two solidification technologies were verified. One of these was the dry homogenisation of all input materials (solidification technology TOS1) and a second one was wetted granulation (solidification technology TOS2). Solidification TOS2 was performed by granulation with wet water spray mixture made up of hazardous waste, fly ash from fluidized bed combustion (FA) and cement on a granulating plate. Once cured and dried, the pellets were then ground and a very fine filler in the form solidification product was created. Solidification by the way of dry homogenisation (TOS1) was carried out by intimate mixing of hazardous waste and FA in a proper homogeniser for 24 hours. The possibility of using such a specially treated filler was verified by the selected tests, which was the determination of the compressive and three-point flexural strength, the determination of adhesion to the basalt tile, determination of tensile strength and elongation at break, using an optical microscope that monitored distribution of the filler in an epoxy matrix, and also the connection interface with basalt grout tile. Due to the possibility of some aggressive components releasing hazardous waste from this filler, it cannot be used to grout elements in hygienically clean operations.

2. Identification of the materials

2.1. The filler material prepared by dry homogenisation (TOS1)

The first solidification technology was dry homogenisation of the individual components using 5% of hazardous waste resulting from the incineration of municipal solid waste (end-product). Furthermore, fly ash from fluidized bed combustion (FA) was selected as the solidification agent. The mixture was allowed to homogenise for 48 hours and then applied as the filler to the epoxy grout.

2.2. The filler material prepared by the wet granulation (TOS2)

The second solidification technology was represented by wet granulation of input. 5% amount of hazardous waste resulting from the incineration of municipal waste (end-product) was used again and as the solidification agent were used FA and cement. All materials were irrigated on the granulation plate with water to form a pellet-like small bowls. These pellets were subsequently dried and ground to a very fine fraction and the obtained filler was used in the grout.

2.3. Binder component – epoxy resin

A two-component, solvent-free and chemically resistant epoxy resin was used as a binder. The mixing ratio of the two components A: B was 1.8: 1.

Component A (epoxy resin) contained the following compounds: epoxy resin (alkoxymethyl) oxirane (C12 -C14 alkyl), formaldehyde, reaction products with 1-chloro-2,3-epoxypropane and phenol.

Component B (hardener side) comprised: 4,4-methylenebis (cyclohexylamine) -phenyl-methanol, formaldehyde polymer with benzenamine, hydrogenated.

The epoxy resin was chosen due to its excellent physical and mechanical properties, high chemical resistance and minimal shrinkage [8].

3. Examinations carried out

3.1. Determination of compressive strength and three-point bending strength

These tests were performed according to standard EN 13892-2 Methods of test for screed materials - Part 2: Determination of flexural and compressive strength. 20%, 30% and 40% filling of the epoxy grout with the filler from dry homogenisation (TOS1) and the filler from wet granulation (TOS2) was tested. The determination of flexural strength was carried out on $20 \times 20 \times 100$ mm test beams (Figure 1). Initially, the flexural strength test was performed, and the compressive strength test was performed on the beam fractions (Figure 2).



Figure 1. Determination of flexural strength.



Figure 2. Determination of compressive strength.

3.2. Determination of cohesion with cast basalt

This test was performed according to standard DIN EN 1542 Products and systems for the protection and repair of concrete structures - Test methods - Determination of consistency by tear test. On the surface of the grout were glued metal targets with a 50 mm diameter (Figure 3) which, after curing of the epoxy adhesive, were trimmed to the octagonal shape up to the base basalt paving. Subsequently, using the pull-off tester DYNA Z16 Proceq (Figure 4), the pull-off force and place of breakage was determined (Figure 5), and the cohesion with cast basalt was found.



Figure 3. Metal targets glued for the determination of cohesion to cast basalt.



Figure 4. Performing the pull-off bond strength test.



Figure 5. Place of failure after pull-off test in the contact zone grout/cast basalt surface.

3.3. Monitoring of the contact zone between the grout and cast basalt paving

Detail of grout connection to the cast basalt tiles surface was monitored using a digital microscope WHX-950F. This microscope is a digital CMOS image sensor with virtual pixels $1600 \text{ (H)} \times 1200 \text{ (V)}$ frame rate of 50 F / allowing magnification to 200x. The contact zone was observed at a magnification of $64\times$ and $6\times$.

3.4. Determination of tensile properties

This test was performed according to standards EN ISO 527-1 Determination of tensile properties - Part 1: General principles and EN ISO 527-2 Determination of tensile properties - Part 2: Test conditions for moulding and extrusion plastics. The test was performed according to the standards mentioned in special test specimens in the shape of dog bone (Figure 6) on a test press (Figure 7). Loading was carried out at rate of 5 mm/min and the initial gauge length of extensometer was 25 mm. Tensile properties were calculated from the measured values, as was the highest tensile strength (tensile strength at the break) and respective elongation at the break.



Figure 6. Specimens in the shape of dog bone for determination of tensile properties.



Figure 7. Loading test specimen of the polymer grout by a tensile force in the test device.

4. Results and discussion

4.1. Determination of compressive strength and three-point flexural strength

The results of the compressive strength (Figure 8) and three-point flexural strength (Figure 9) indicate that with an increasing percentage of filling, the resulting values decreased, and therefore for further testing, the 30% filling which guarantees relatively good properties and a relatively high percentage of the solidification product. Flexural strength of mass with 30% of the filling was about 25 MPa and compressive strength was about 50 MPa. When compared to reference materials, it is seen that almost the same characteristics of jointing grout material had 20% filler TOS2. At higher percentage of filling the strength was slightly smaller than the reference value, but the differences were not significantly large. As it can be seen, the type of solidification of HW had a significant effect on the final strength grout. The biggest difference is apparent in samples with 20% of filling, when with sample containing filler TOS2 was an increased compressive strength, probably due to better homogeneity of the particles of filler into the polymeric matrix. It can be also affected by the specific surface area and shape of the filler particles. This fact can be observed in both the compressive strength and flexural strength.

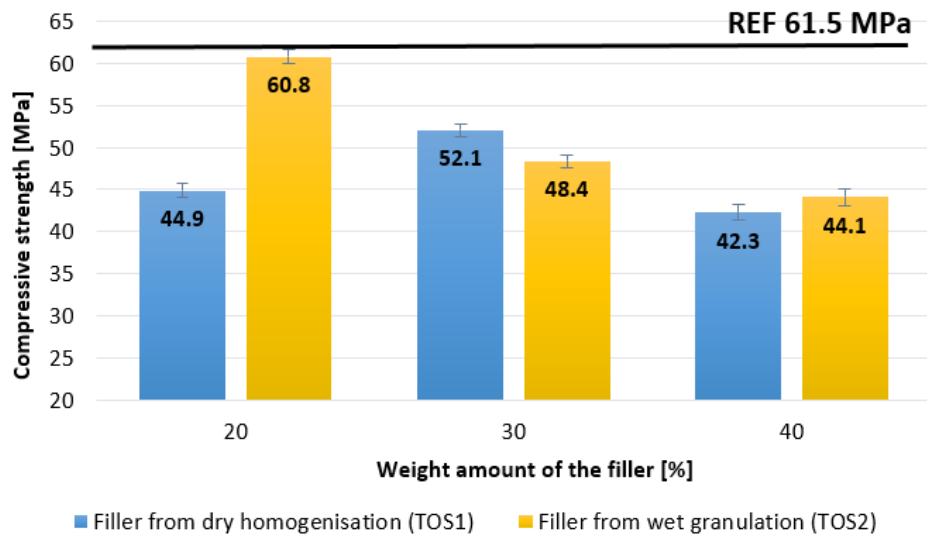


Figure 8. Results of the developed epoxy grout compressive strength.

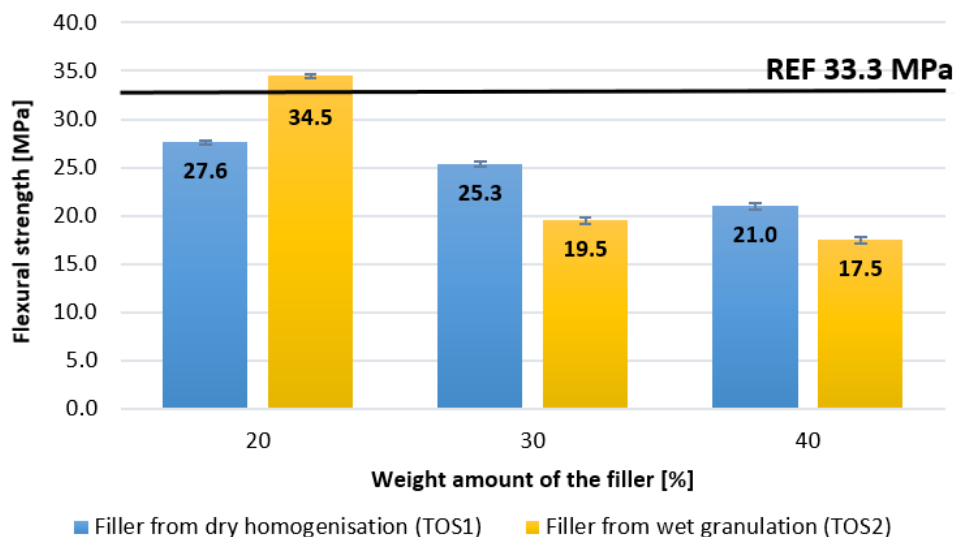


Figure 9. Results of the developed epoxy grout three-point flexural strength.

4.2. Determination of cohesion with cast basalt

In determining the cohesion of polymer grout with cast basalt tiles, it was found that the cohesion of the two developed grout jointing materials is approximately 3.5 MPa (see Fig. 10), which is very satisfactory value for the purpose of intended use. Reference material containing approximately 30% wt. of silica flour showed almost the same cohesion with cast basalt (3.55 MPa) as developed grout containing solidification product. The violation has occurred in all the samples on the surface of the pavement of basalt - see Figure 5. Epoxy resins generally have a high cohesion with almost all materials. It was demonstrated here that the cast basalt with a smooth surface is not an exception. It was successfully shown that the developed grout can be safely used for grouting cast basalt elements.

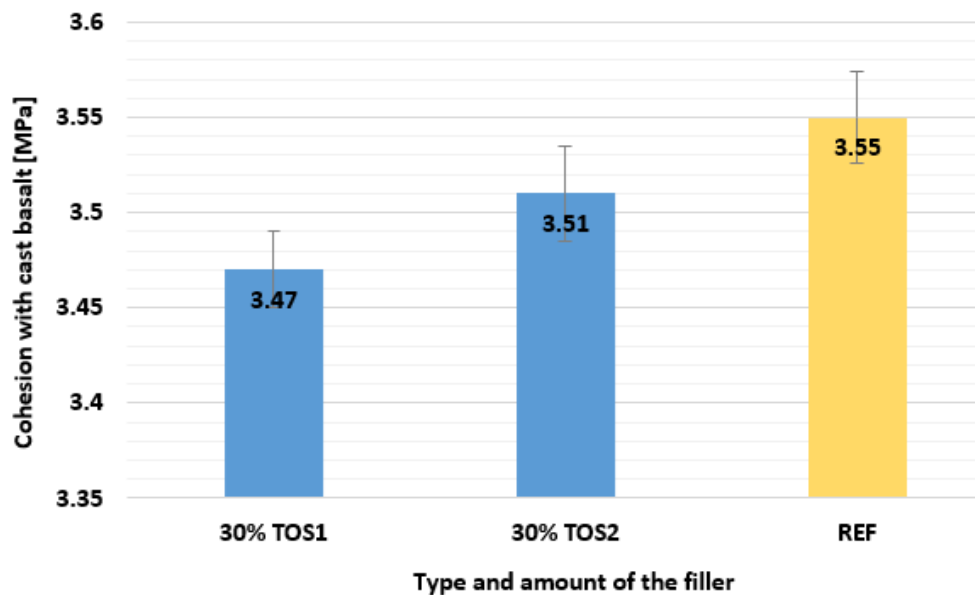


Figure 10. Results of the cohesion with cast basalt tiles

4.3. Monitoring of the contact zone between the grout and cast basalt

To find the detail of grout and cast basalt connection was monitored using a digital microscope contact zone between these materials. In 6x magnification (Figure 11), air voids in the grout were noticed likely due to mixing. However, in the contact zone, cast basalt tiles had no imperfections or free space, indicating a high cohesion between the materials. For more detailed observation of the connection between the developed epoxy grout cast basalt tile were chosen to 64x magnification (Figure 12) where it was even better to see the perfect connection of the grout to the tiles, which perfectly mirror the mass of fine unevenness on the surface of the tile. Among other things, at such magnification, the structure of both the grout and the cast basalt paving surface is already well observable. It can be seen in the structure of the grout that the filler is perfectly distributed throughout the epoxy matrix and does not form clumps.

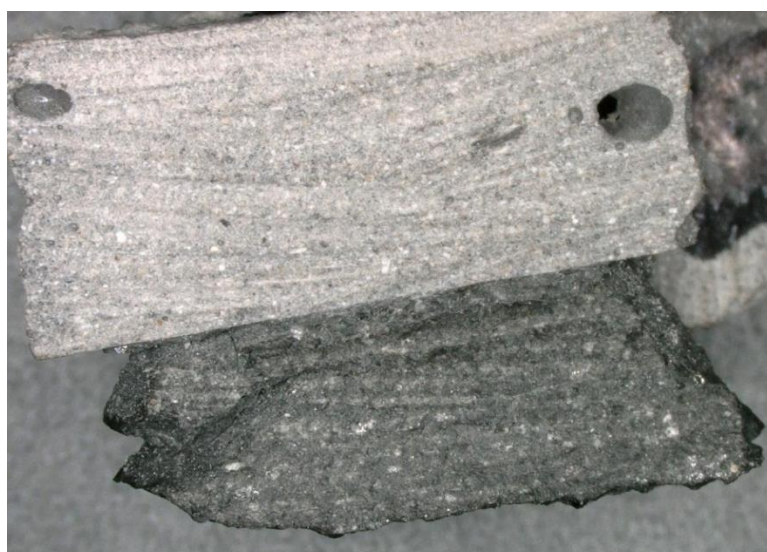


Figure 11. The connection detail of developed grout (30% TOS2) to cast basalt tile (magn. 6x).



Figure 12. The connection detail of developed grout (30% TOS2) to cast basalt tile (magn. 64×).

4.4. Determination of tensile properties

By examining the results of tensile stress and elongation at break (Figure 13), it is apparent that improved stress exhibited the grout containing the filler prepared by wet granulation, but this difference is minimal. Tensile stress of grout fluctuated values around 18 MPa, and elongation at break of 0.50%. These differences may be caused in the same way as with the compressive strength distribution in the mass of the filler, the shape of the filler particles, and by the amount and distribution of pores in the samples – break was observed always at the point of the largest pores that weaken the cross-section. From the results, it is seen that reference material exhibited a higher tensile strength with a corresponding elongation at break, but since the strength of the difference is minimal, so the filler material based on solidification product appears to be a suitable replacement for primary materials.

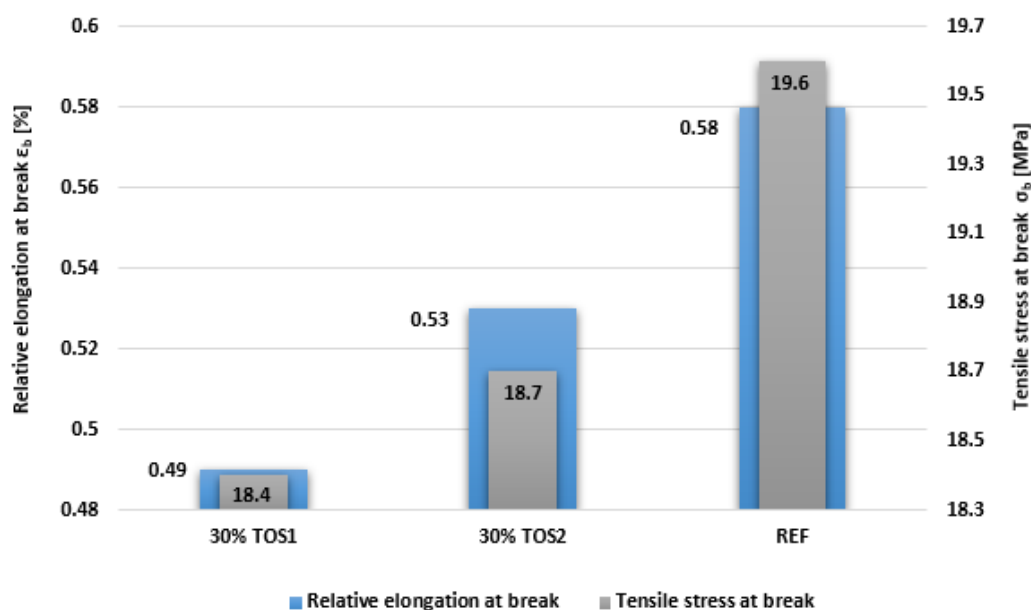


Figure 13. The results of tensile properties of tested epoxy grouts.

5. Conclusion

The research results, which dealt with the use of specially treated hazardous waste (HW), solidification product, as a filler in a polymeric grout, showed that this developed fine material seems very suitable as a filler. A total of two proposed solidification technologies (TOS1, TOS2), namely one wet granulation and one dry homogenisation. Due to the fact that the results of both prepared fillers were very similar, and the kind of solidification technology did not show a significant effect on the resulting physical and mechanical properties of the grout, it appears to be economically more advantageous to use dry homogenisation technology. Utilising solidification product containing HW and appropriate secondary materials in grout contribute to the reduction of landfill materials and reducing environmental burdens.

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