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Experimental application of waste glass powder filler in recycled dense-graded asphalt mixtures

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This paper presents laboratory study results, including both bituminous mastics and mixtures tests, to assess the use of glass powder (GP) as possible surrogate to limestone filler in recycled dense-graded asphalt mixtures. To achieve a comprehensive approach, the analysis has been divided into three parts including filler, mastics and mixture characterisation. The GP has been completely studied with chemical and physical tests to compare the fundamental characteristics to the commonly used limestone filler. In the second part, rheological tests using the Dynamic Shear Rheometer has been implemented to evaluate fillers interaction with not modified and modified bitumen. Finally, the mechanical properties of the asphalt mixtures containing GP filler were investigated in terms of indirect tensile strength, indirect tensile stiffness modulus and creep characteristics. According to the obtained results, GP filler provided comparable values to limestone filler within both mastic and mixture study. It is noteworthy that from the permanent deformation analysis, both MSCR and RLAT tests confirmed the improvement of rutting resistance with using GP filler.

Keywords: road pavements; glass powder; recycling; asphalt mixture; rheology

Introduction

In order to minimise the environmental impacts of roads construction and maintenance, it is necessary to quantify the energy and resources savings. In particular, based on the European Green Public Procurement (GPP), criteria for road design are now crucial to provide a minimum requirement regarding to the amount of recycled content in road elements. In this context, the validity of sustainable construction focused on using recycled materials (Mazzotta et al., 2017; Vignali et al., 2016) becomes fundamental in terms of life cycle assessment (LCA) of road infrastructures. In the last decade, besides the environmental aspects, the high economic pressure of virgin material cost and the limited resources have obliged the decision-makers to use recycled materials more than before. Reclaimed asphalt pavement (RAP), recycled asphalt shingles (RAS), waste plastics and grind (and/or powdered) glass are some of the investigated materials in the pavement industry. While the objectives of RAP and RAS incorporation in asphalt mixtures are reusing both aggregate and binder, the waste powders such as waste brick (Arabani, Tahami, & Taghipoor, 2016), waste bleaching clay (Mazzotta, Sangiorgi, Vignali, Lantieri, & Dondi, 2015; Sangiorgi et al., 2016a), rice husk ash (Al-Hdabi, 2016) and glass powder (GP) were investigated to substitute the traditional limestone filler. In this regard, many studies have

shown the influence of filler type on the rheo-mechanical behaviour of bituminous mixtures (Anderson et al., 2001; Kim, Allen, & Little, 2007).

Among the studied materials, waste glass has been investigated as a portion of virgin aggregates in many previous researches. These studies have shown that the low absorption properties of glass particles play a key role for further performance deficiencies (Ghasemi & Marandi, 2013). The early stripping, high ravelling potential and reduced skid resistance are some of the main problems due to low porosity of glass particles, which led to use glass as an alternative filler or fine aggregates (Kandhal, 1996; Wu, Yang, & Xue, 2007). The nature of mineral aggregates is one of the main factors that influences both cohesion and adhesion properties (Lamperti, Grenfell, Sangiorgi, Lantieri, & Airey, 2016).

Considering the GP as filler, several researchers found that the use of recycled GP does not worsen mechanical and performance properties of asphalt mixtures. For instance, it is indicated that replacement of limestone filler by GP resulted in lower flow rate and higher stability (Flynn, 1993; Jony, Al-Rubaie, & Jahad, 2011). However, in contrast, another research showed that the replacement of limestone filler by GP increases the flow rate and decreases the stability (Saffar, 2013). Further studies proved that GP has an effective role in moisture susceptibility and resilient modulus of conventional asphalt mixtures (Wu et al., 2007). In literature, there is agreement for volumetric analysis of the mixtures containing GP. Many previous researches showed that the substitution of mineral filler by GP will result in lower density and higher air voids. The logical explanation for this goes to lower adsorption and chemical exchange between silica and asphalt, which makes more free binder in the mixture (Tang, Wu, Liao, & Huang, 2015). In this research, the GP filler, probably due the low mastic percentage in the studied mixtures, does not influence a lot the stability. The increase of mastic matrix in the volume of the mixture, typical of an SMA, has a proportional effect on the stability results of the GP filler mixture if you compare it with the control mixture using limestone filler (Udaya & Venkateswara, 2016).

Starting from the review of the available literature of GP filler application, a lack of rheological characterisation of bitumen and mastics has been verified. The aim of the current study is to assess the applicability of GP as an alternative to commonly used limestone filler. The presented experimental work mainly included three parts, including GP prequalification assessments, rheological analysis on mastics made with GP and limestone, and asphalt mixture mechanical evaluation with static and dynamic tests.

Materials

Figure 1 shows the studied materials in view of a multi-scale approach for the eight different combination of fillers and bitumens. In particular, the research have been started by filler characterisation, followed by mastic rheological studies and concluded with an analysis of mixture properties. Therefore, after defining the mix design, fillers and mastics that compose the asphalt mixture have been studied. The products obtained with the use of GP were compared with the corresponding ones containing limestone filler. In order to provide a comprehensive knowledge on the applicability of using the same design, neat and modified bitumen were used. Table 1 shows the composition of the studied mastics and mixtures using both neat and modified bitumen:

Aggregates

The aggregates used in the current experimental work were 80% of virgin crushed limestone and 20% of RAP. The selected gradation of aggregates is shown in Figure 2. The choice of grain size was based on the one of the typical Italian specifications (ANAS) gradation limits of HMA mixtures for surface layers. The volumetric mass of the aggregates was calculated according to the EN 1097-6 standard.

Figure 1. Materials and experimental plan.

Table 1. Filler and binder content. Asphalt mixture samples and mastics.

Scale	GP filler (%)	Limestone filler (%)	Bitumen (% on weight of aggregate)	Mixture ID
Asphalt mixture	7	—	5.5	GP-NB GP-PmB
Mastic	66	—	44	GP-NB GP-PmB
Asphalt mixture	—	7	5.5	LS-NB LS-PmB
Mastic	—	66	44	LS-NB LS-PmB

Figure 2. Applied aggregate gradation (ANAS, Italian specifications).

Limestone filler

The used limestone filler properties complied with the following requirements:

- Calcium carbonate content $\geq 75\%$ by mass (typically $\text{CaCO}_3 \approx 95\%$);
- Clay content (adsorption of methylene blue) $\leq 1.20 \text{ g}/100 \text{ g}$;
- Organic content (TOC) $\leq 0.20\%$ by mass (for the type LL $\leq 0.50\%$ by mass, $\text{FeO}_2 \approx 0.2\%$ to $\text{SiO}_2 \approx 0.4\%$).

Figure 3. GP before and after drying process.

Table 2. GP chemical components.

Chemical component	Content (%)
SiO ₂	29.17
CaO	16.51
Al ₂ O ₃	18.85
Fe ₂ O ₃	6.97
MgO	2.60
Na ₂ O	10.72
K ₂ O	0.65
SO ₃	0.87
CaCO ₃	—
other	13.66

GP filler

Recycled GP is an innovative, eco-friendly and multifunctional filler coming from the processing residual of goldsmith industries. Before grinding, the waste is subjected to iron and plasticisation removal processes. Later it is sent to the crusher, which imparts a first crushing to the material, reducing the size to not more than 2 mm. Subsequently, the filler is sent directly to a rotary mill, which reduces the size to less than 75 μ m. Through a system of sieves or with a classifier, the turbine material is checked and calibrated. At this point, the material undergoes the washing operation because of the high dustiness possessed by the material. Chemical properties of the studied GP are provided in Table 2. As shown, the GP filler has a low content of silica SiO₂ (about 30%), although it is the main constituent. Calcium oxide (CaO), alumina (Al₂O₃) and sodium oxide (Na₂O) are present inside the compound in a percentage ranging between 10% and 19%. The percentages of the other oxides are less than 1%. Considering the chemical analysis, it can be stated that the source of recycled glass is various, which implies that this material has not been subjected to a special refining processes, making this a cheaper and environmentally friendly product. The wet GP is similar to a mud with gray-brown clay colour. Once dried turns out to be a fine powder of texture and colour similar to ash (Figure 3).

Table 3. GP filler vs. limestone filler characteristics.

Filler ID	Volumetric mass (mg/m ³)	Rigden voids (%)	Blue of methylene (g _{blu} /kg _{filler})	Water solubility (%)	Delta Ring & Ball (°C)
GP filler	2.47	28.67	5.0	2.0	10.20
Limestone filler	2.55	35.40	1.2	0.2	7.50

In order to use GP as the filler in asphalt mixtures, it has been completely dried and later been characterised with the following tests described below (Figure 4):

- Particle size analysis was conducted with the photo sedimentation process (Micrometrics SediGraph 5100) for the fraction between 0.5 and 100 μm , and through wet sieving for the portion greater than 100 μm .
- The Rigden void test (EN 1097-4) was performed to determine the void volume in the dry-compacted fillers. It is based on the assumption that the maximum bulk density of fines can be obtained by compacting the dry fines in a mould (Figure 4).
- The specific bulk density test (EN 1097-7) was carried out to determine the specific bulk gravity of the filler, using standardised pycnometers.
- The blue of methylene test (EN 933-9) was performed to determine the amount of methylene blue adsorbed by GP. The result is reported as a methylene blue value in units of (mg) of methylene blue adsorbed per (g) of fine filler. The methylene blue value is a function of the amount and of characteristics of clay minerals present in the specimen. High methylene blue values indicate increased potential for reduced fine aggregate or mineral filler performance in aggregates mixture due to the presence of clay (Figure 4).
- The water sensitivity test (EN 1744-1) was performed to evaluate filler solubility resistance in water (Figure 4).
- The Delta Ring and Ball test (EN 13179-1) was implemented to assess the stiffening effect of mineral fillers used in asphalt mixtures.
- GP and limestone filler particle size analysis results are reported in Figure 5; the GP is clearly finer than the limestone filler.

In Table 3, GP characteristics are compared with traditional limestone filler ones. Rigden voids test provides information about the potential amount of bitumen that the filler can adsorb and, as a consequence, is an indicator of the stiffening power that the filler exercises on the bituminous mastic (Sangiorgi et al., 2017). According to the EN 13043 standard, the values of the Rigden Voids are restricted to the interval 28–55%. Taking this standard as reference, the GP filler respects the defined limits. About the methylene blue, it can be stated than the presence of the fine part is higher in GP filler than limestone; however, both filler respects the standard limits. The EN 13043 standard suggests that the limit value of solubility in water is 10%. The water solubility for the GP filler is close to the value obtained for the limestone one and Delta Ring & Ball values close to 10°C guarantees a good workability of the mixtures.

Binders

For this research, one SBS-modified bitumen and a commonly used 50/70 pen-graded neat bitumen were used producing in both asphalt mixtures specimens and mastics samples. It is worth mentioning that the SBS polymer-modified bitumen (3% in weight of bitumen) has been

Figure 4. GP filler characterisation tests; (I) Water sensitivity test EN 1744-1, (II) Blu of Methylene test EN 933-9, and (III) Rigden Void EN 1097-4.

Figure 5. GP gradation vs. limestone particle grading.

Table 4. Physical properties of the binders.

Property	Unit	Bitumen type		Standard
		Pen grade 50/70	PmB (SBS)	
Penetration @ 25°C	dmm	50	55	EN 1426
Softening Point	°C	50	70	EN 1427
Force Ductility test @10°C	J/cm ³	—	3	EN 13589
Dynamic Viscosity @160°C	Pa s	0.5	0.7	EN 12596
Elastic Recovery @25°C	%	—	80	EN 13398

described as type P by Dondi et al. (2016). The characteristics of the bitumens were evaluated and shown in Table 4.

Experimental work

Mastic rheological investigation

Amplitude Sweep test

Amplitude Sweep tests, according to EN 14470, were preliminarily carried out to investigate the viscoelasticity region at 10°C by applying a constant frequency of 10 rad/s (1.59 Hz).

Frequency Sweep test

In order to represent the complex modulus (G^*) and phase angle (δ) of asphalt mastics, frequency sweep tests according to EN 14470 (Figure 6) were conducted. Master curves were calculated from the tests, finding the relationship between load frequencies and complex modulus G^* and phase angle δ . The reference temperature for the master curves was 20°C. The Williams–Landel–Ferry model was used to obtain the temperature shift factors. The frequency range was between 0.01 and 10 Hz from 0°C to 60°C.

Multiple Stress Creep Recovery test

The Multiple Stress Creep and Recovery test (MSCR) was run according to the AASHTO TP 70-07 (2013) “Standard Method of Test for Multiple Stress Creep and Recovery (MSCR) of Asphalt Binders using a Dynamic Shear Rheometer”. According to this standard, the mastic sample is loaded at a constant creep stress for 1 s, followed by a zero stress recovery of 9 s. Ten cycles of creep and recovery are run at 0.1 kPa creep stress, followed by 10 at 3.2 kPa creep stress. The non-recoverable compliance (J_{nr}) and the percent recovery after 10 cycles at 0.1 and 3.2 kPa were studied. The J_{nr} value was calculated as the ratio between the average non-recoverable strain for 10 creep and recovery cycles, and the applied stress for those cycles. The testing temperature of 46°C was adopted.

Asphalt mixture characterisation

Volumetric properties

The 150 mm diameter specimens were compacted by a gyratory compactor under constant 600 kPa pressure and an external angle of 1.25°C for 100 gyrations at 155°C.

The volumetric characteristics have been determined according to the standard EN 12697-8. In addition to compaction, the curves were analysed to investigate the rate of workability of different mixtures.

Figure 6. Dynamic Shear Rheometer (Anton Paar MCR302) test set-up and test sample.

Indirect tensile strength

Indirect tensile stiffness modulus. The test was carried out according to EN 12697-26 at 5°C, 20°C and 35°C. The test has been conducted considering the target of 5 microstrain deformation with the standard recommended rise time of 124 ± 4 (ms).

Repeated Load Axial Test. For this research, the test has been conducted following the standard EN 12697-25 at 40°C under the stress level of 100 kPa and all the specimens were subjected to pre-test conditioning loading of 10 min, with the main purpose of ensuring the load platens were seated properly prior to the deformation measurement (Figure 7). In the following, the results are presented in terms of accumulated strain and creep stiffness

$$E_n = \frac{\sigma}{\varepsilon_n} \times 1000, \quad (1)$$

where E_n is the creep modulus after n load applications, σ is the applied stress and ε_n is the cumulative axial strain of the specimen after n load applications (MPa).

Results and discussion

Effects of GP filler on mastic rheological properties

Linear visco-elastic field (γ_{LVE}) of mastic samples is 0.75% for unmodified bitumen and 0.5% for SBS-modified bitumen independent of the filler type. The bitumen characteristics have a significant effect on the LVE range, such as the LVE limit deformation of neat bitumen is 50%

Figure 7. RLAT configuration.

Figure 8. Master curves comparison: (a) mastic with neat bitumen and (b) mastic with polymer-modified bitumen.

higher than the SBS-modified bitumen (Airey, 1997). From the comparison between master curves of LS-NB and GP-NB mastics (Figure 8(a)), it can be noted that the GP presence tends to increase the mastic stiffness in all frequency range. In particular, this behaviour is enhanced at high and low frequencies. The phase angle behaviour of mastics follows the base bitumen trend; both fillers do not change the visco-elastic response of the bitumen, showing a completely viscous response at high temperature and a predominant elastic response at low temperatures. As shown in Figure 8(b), the SBS presence, in both mastics, is confirmed by the complex modulus increase and phase angle reduction at high temperatures (Airey, 1997). However, in this case, the GP-PmB mastic, containing GP, has higher moduli values than the mastic containing limestone filler at low frequencies, showing the horizontal asymptote in correspondence of the low frequencies. The GP presence increases mastic permanent deformation endurance at high temperatures exalting the polymer effects in terms of shear stress resistance. Moreover, analysing the phase angle trend it can be noticed that the phase angle values of GP-PmB mastic are lower at high temperature compared to the LS-PmB ones. The GP increases the SBS effects in terms of elastic response of the bituminous part.

Effects of GP filler on the asphalt mixture

Figures 9 and 10 show the compaction curves for the studied mixtures. From the developed compaction curves, it is evident that the slope of the mixtures containing the two investigated

Figure 9. GP and LS mixtures' compaction curve comparison (neat bitumen).

Figure 10. GP and LS mixtures' compaction curve comparison (polymer-modified bitumen).

Table 5. Volumetric properties of asphalt mixtures.

Specimen ID	VMA	VA %	VFA
GP-NB	15.4	4.0	73.9
LS-NB	15.7	4.4	72.3
GP-PmB	15.9	4.6	71.3
LS-PmB	16.6	5.40	67.3

Note: Gyration number: 100.

fillers is the same. It can be deduced that the GP filler has no negative effect on the mixture compaction rate (Table 5). Moreover, the mixture containing GP filler has less air voids than mixtures with conventional limestone one, reflecting the results obtained from the Rigden test.

In asphalt mixtures, the mineral skeleton (aggregates) affords the compression strength, and the bitumen and mastic cohesion deliver the tenacity (furthermore the bitumen/aggregates adhesion) (Moreno & Rubio, 2012). Given that the tensile characteristics of asphalt mixtures are

Figure 11. The average ITS values at 25°C.

Table 6. ITSM values in (MPa).

Spec. ID	Test temperature (°C)			Spec. ID	Test temperature (°C)		
	5	20	35		5	20	35
GP-NB	11,960	4638	1010	LS-NB	10,188	4114	1163
GP-NB	12,743	3413	816	LS-NB	11,312	3090	811
GP-NB	11,996	3340	966	LS-NB	11,543	3769	957
GP-NB	11,972	3454	768	LS-NB	11,735	3896	941
Ave. GP-NB	12,168	3711	890	Ave. LS-NB	11,194	3717	968
GP-PmB	10,469	2958	750	LS-PmB	12,914	4031	1046
GP-PmB	9711	2102	724	LS-PmB	12,602	4149	916
GP-PmB	15,220	4648	1479	LS-PmB	12,702	4055	1047
GP-PmB	13,578	3806	1011	LS-PmB	13,076	3941	906
Ave. GP-PmB	12,244.6	3378	991	Ave. LS-PmB	12,823	4044	979

indicators of cracking potentiality, it is considered as a fundamental parameter of rutting (Khosla & Harikrishnan, 2007), thermal and fatigue cracking resistance. In addition, the indirect tensile strength (ITS) value directly depends on the tenacity reached by aggregate/filler and bitumen bonding level (Dondi et al., 2014); hence, the ITS value is considered as a reliable comparative parameter. Figure 11 presents the average values of ITS tests. As expected, the ITS values for the mixtures with PmB are higher than the mixtures with neat bitumen. It can be seen that the test results of mixtures with GP filler are comparable with the ones containing LS filler. In detail, from the result analysis of the mixtures made with unmodified bitumen, it is noted that the presence of GP filler increases the mixture's tenacity. The low viscosity of the unmodified bitumen led to better GP bitumen absorption. On the other hand, for the mixtures made with PmB, the ITS values for control mixture are higher than the mixtures containing GP filler. As found in literature (Ghasemi & Marandi, 2013), the low bitumen absorption properties of glass increase with higher viscosity bitumen, which leads to lower adhesion properties.

At common traffic flow speeds and pavement temperatures, asphalt mixtures act almost elastically; therefore, indirect tensile stiffness modulus (ITSM) is a measure of its resistance to bending and relatively its loads spreading ability (Nunn & Smith, 1996). In literature, ITSM is also considered as an indicator for the structural properties of mixtures as it is related to the capacity of mixture to traffic loads bearing (Nassar, Khashaa Mohammed, Thom, & Parry, 2016). ITSM

Figure 12. ITSM values.

Figure 13. ITSM values.

values greatly depend on test temperature and the results are often considered for thermal sensitivity analysis of asphalt mixtures (Sangiorgi et al., 2016b). From the results showed in Table 6 and Figures 12 and 13, it can be clearly seen that at medium temperatures the difference can be considered negligible as noticed though frequency sweep test results. A discussable difference emerges at low temperatures where, especially in the presence of unmodified bitumen, the higher ITSM values for the mixture with GP filler and neat confirm mastics rheological results. The GP stiffens the bituminous mixtures and increases the bearing capacity.

Permanent deformation evaluation

Mastic scale permanent deformation susceptibility

In order to study the rutting resistance of the studied mastics and mixtures, permanent deformation properties were characterised by means of MSCR at 46°C and Repeated Load Axial Test (RLAT) at 40°C, evaluating the probable effect of the GP filler. The standardised temperature was chosen in order to compare these results with RLAT results on asphalt mixture. Figure 14 shows the MSCR curves at the end of the 3.2 kPa shear stress level cycles. Mastics containing GP exhibit stiffer behaviour, accumulating less deformation at the end of the test. The bitumen characteristics of the studied mastics exhibit significantly different response, influencing the glass

Figure 14. MSCR test result for GP and LS with virgin bituminous mastic at 46°C under 0.1 kPa and 3.2 kPa.

Figure 15. J_{nr} values from the MSCR test at 3.2 kPa.

powered effects. In the mastics made with the unmodified bitumen, GP filler increased the resistance to deformation during the creep phase but this mastic exhibited a slight recovery during the recovery phase. In contrast, the modified elastomeric bitumens recovered more deformation

Figure 16. J_{nr} and accumulated strain correlation.

Table 7. RLA test results at 40°C.

Specimen ID	Accumulated strain (%)	Creep stiffness MPa
GP-NB	0.886	11.42
GP-NB	0.808	13.35
GP-NB	0.735	13.72
Ave. GP-NB	0.810	12.83
LS-NB	0.739	13.51
LS-NB	1.178	8.57
LS-NB	0.808	12.35
Ave. LS-NB	0.908	11.48
GP-PmB	0.738	13.52
GP-PmB	0.612	16.31
GP-PmB	0.645	15.57
Ave. GP-PmB	0.665	15.13
LS-PmB	0.851	11.60
LS-PmB	0.699	14.23
LS-PmB	0.737	13.52
Ave. LS-PmB	0.762	13.12

during the unload phase. Polymer-modified bituminous mastic has significantly higher capacity of recoverable strain and, as confirmed by the FS sweep test analysis, the GP presence exalts this characteristic. At both shear stress levels, the GP-PmB exhibits lower values of accumulated strain than the mastic containing limestone filler.

The results of MSCR curves are confirmed studying the non-recoverable compliance values (Figure 15). J_{nr} values were calculated for the four mastics under 3.2 kPa shear stresses at 46°C. The GP mastics, obtained with both unmodified and modified bitumen, have lower values of non-recoverable compliance at 3.2 kPa, showing less sensitivity to permanent deformations.

Asphalt mixture permanent deformation susceptibility

In the current study, the direct uniaxial cyclic compression configuration, termed the permanent deformation properties was followed by applying RLAT. From the results presented in Table 7, the mixtures containing GP filler show less accumulated strain values and higher creep stiffness

moduli if compared to the mixtures containing the traditional limestone filler. From the results provided by the RLAT test, in accordance with the studies conducted by Khosla and Harikrishnan (2007), GP filler was found to increase surface layer resistance to permanent deformations. This analysis has been thoroughly investigated correlating the mixture response to RLAT load to that of the mastic during the MSCR test. Based on the correlation between ALF rutting parameter and J_{nr} extracted by D'Angelo, Kluttz, Dongré, Stephens, and Zanzotto (2007), a correspondence between the accumulated strain and J_{nr} has been extrapolated (Figure 16). As found in the cited studies, also in this case the coefficient of determination is greater than 0.8 confirming how the rheological results, obtained through the MSCR test, can be related to mixture performances. From the permanent deformation evaluation, it is clear that the stiffness increase given by the GP filler is reflected on the resistance to rutting phenomena.

Conclusions

The present research confirmed the applicability of reusing fine GP as a filler with both neat and modified binders. The main purpose of this research was to characterise the GP as a suitable material for the road pavements construction, replacing with traditional limestone filler. The multi-scale approach has been validated within the consistent results obtained from both the rheological and mixture analysis results. In detail, both ITSM and RLAT results reflected the FS and MSCR data relating mastic rutting performance to in service pavements. The use of GP as a filler in the asphalt mixtures follows the GPP criteria in terms of recovery and reuse of a waste material for the main road elements. As part of the infrastructures, LCA future studies will involve the evaluation of mixture durability in term of fatigue life and energy savings achieved through the use of GP.

The results obtained from the physical characterisation confirmed the suitability of GP to use as filler. In terms of interaction between unmodified and modified bitumen with GP, the rheological properties of mastics were studied. From the results obtained by performing FS and MSCR tests, the GP filler increases the mastic stiffness and at the same time exalts SBS polymer modification in terms of elastic shear stress response. From the mixture point of view, the ITSM and RLAT results indicated that the GP filler could provide to improve mixtures performance in terms of bearing capacity and permanent deformations resistance.

Disclosure statement

No potential conflict of interest was reported by the authors.

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