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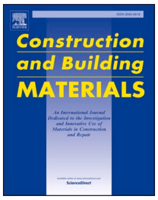

Laboratory evaluation of environmentally friendly alternative mineral powders for micro-surfacing treatments manufacture

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ABSTRACT

The effectiveness of using alternative environmentally friendly and cost-effective mineral powders to manufacture micro-surfacing treatments is examined. Four mineral powders (calcium carbonate, dolomite, PAVAL, and mineral dust from the recycling of used toner) as well as Portland cement (control) were used. The use of two of them, the PAVAL and the toner, is the main novelty of the present research. Their granulometry by means of an air jet sieve, their density, and their chemical composition by means of X-ray fluorescence (XRF) were obtained. To generalise the findings, two different types of aggregate (siliceous and porphyritic) have been used to manufacture the micro-surfacing treatment. The mixing time, setting and curing characteristics, and workability were analyzed. Additionally, the resistance to the loss of particles by the wet track abrasion was also studied. Likewise, the consistency assessment was done. Finally, the cohesion also was attained. The micro-surfacing treatments manufactured with all the alternative mineral powders comply with the technical requirements. Moreover, they improve the loss of particles, particularly the toner, with a loss of particles between 87% and 280% lower than the control micro-surfacing. Also, they are less hardening-controlling additive demanding. Particularly the PAVAL needs 0.2% of additive while the control micro-surfacing needs 0.6%. These findings are supported by statistical analysis of variance (ANOVA).

1. Introduction

Micro-surfacing treatments (MICROFs) are bituminous mixtures with the proper consistency to be prepared and applied on-site at room temperature utilising aggregates, water, mineral powder, and additives (when necessary) [1]. The 1930s saw the creation of MICROFs in Europe [2], but it wasn't until the 1960s–primarily in the United States, France, and Spain–that it really began to take off. However, it is now used extensively in numerous nations across five continents. These treatments are used as maintenance and preservations pavement treatments [3], specifically to improve the texture, increase the skid resistance, and to waterproof highways and airport pavements [4].

The recognised technical solvency and environmental efficiency of MICROFs are their main features. The significant technological advances made in this field since its beginnings, ensure their technical solvency. In this regard, highlights the use of bitumen emulsions with modified rheology, breaking time-controlling additives, that are increasingly adaptable, and, thus, usable in the most difficult conditions. Also, the use of fibres that permit the use of higher binder concentrations while still ensuring greater durability due to a better cracking resistance [5]. Environmental efficiency is based on two fundamental pillars:

- The current lack of a surface rehabilitation technique for highway and airport pavements that requires less consumption of raw materials. Indeed, pavement surfaces can be rehabilitated with extraordinary results with these asphalt mixtures and quantities always less than 15 kg/m^2 [1].
- It is a cold application, which translates into lower energy consumption and makes it more friendly to the environment, and the workers.

The features of a MICROF will largely depend on the selection of its constituents and characteristics. It is useful to distinguish between majority and minority components in this regard. Because they make up

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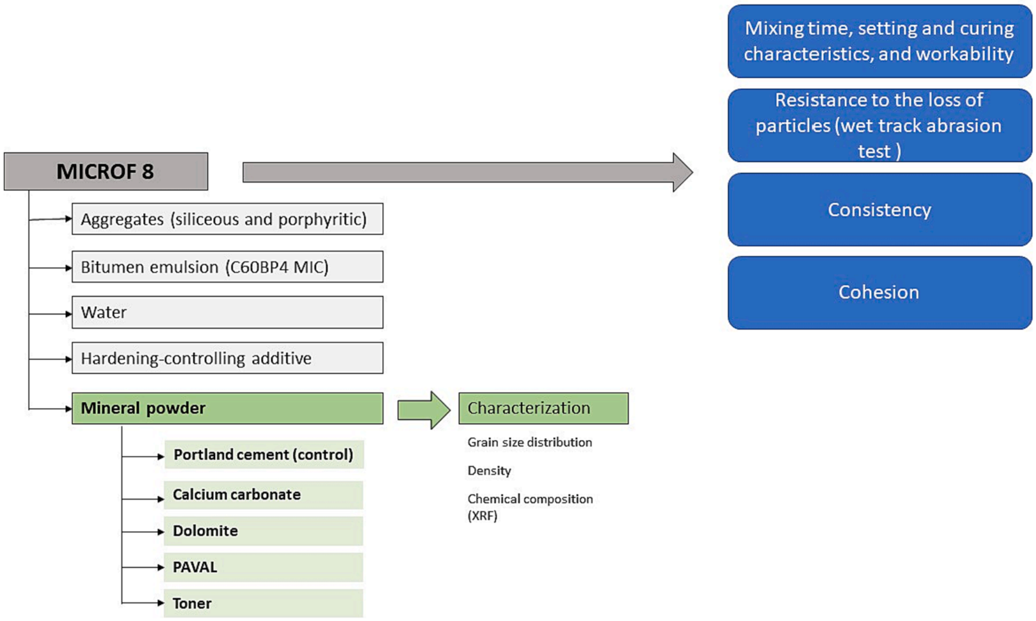


Fig. 1. Flow chart of the research.

Table 1
Main properties of the aggregates.

Property	Standard	Unit	Aggregate		PG-3 limits
			SI	PO	
Sand equivalent (SE)	EN 933-8 [20]	%	89	69	>60
Flakiness index (FI)	EN 933-3 [21]		17	15	<20
Crushed particles	EN 933-5 [22]		100	100	100
Rounded particles	EN 933-5 [22]		0	0	0
LA coefficient	EN 1097-2 [23]		11	11	<15
Polished Stone Value (PSV)	EN 1097-8 [24]	–	54	57	>50

Table 2
Main properties of the bitumen emulsion.

Property	Standard	Unit	Value
Original binder			
Polarity	EN 1430 [26]	–	Positive
Breaking value	EN 13075-1 [27]	g	150 ± 5
Binder content	EN 1428 [28]	%	60,5 ± 0,5
Oil distillate	EN 1431 [29]		1,0 ± 0,5
Efflux time (2 mm, 40 °C)	EN 12846-1 [30]	s	35 ± 5
Residue on sieving (sieve 0,5 mm)	EN 1429 [31]	%	0,05 ± 0,05
Settling tendency (7 d)	EN 12847 [32]		1,0 ± 0,5
Residual binder (EN 1431 [29])			
Penetration (25 °C, 5 s, 100 g)	EN 1426 [33]	0,1 mm	80 ± 5
Softening point (ring and ball)	EN 1427 [34]	°C	60 ± 5
Cohesion	EN 13588 [35]	J/cm^2	0,8 ± 0,2
Elastic recovery	EN 13398 [36]	%	60 ± 5

Table 3
Chemical and physical properties of the hardening-controlling additive.

Property	Value
State	Liquid
Colour	Brown
Viscosity at 25 °C	(0,5 ± 0,1) St
Density at 25 °C	(1,05± 0,1) g/cm^{-3}

more than 98% of the mass of the MICROF, the aggregates, bituminous emulsion, and water are its primary constituents. The choice of the primary components will depend on their accessibility to the road where the MICROF is conducted. The minor components of the MICROF are the hardening-controlling additives, and the mineral powders.

The mineral powder, is the aggregate whose majority passes through the 0.063 mm sieve [1]. Particularly, the active mineral powders, while making up less than 2% of the mass of these mixtures, have a remarkable and essential impact on their characteristics, functionality, and quality. Particularly, this compound greatly influences the MICROF performance. In this regard, the mineral powder speeds up the breaking process and activate the achievement of final cohesion [1,6]. Also, the mineral powder is largely used to stiffen the mastic [7], control the rutting [8], reduce segregation [7], and increase resistance to fracture and crack propagation [9]. Also, it can be used to improve the gradation of the micro-surfacing treatments [7]. And when lime or cement are used as mineral powder, they also improve the antistripping properties of the mixture [9]. Specifically, the performance of the MICROF is highly influenced by the mineral powder specific gravity, particle size, porosity, shape, texture, mineralogy, and chemical composition [10].

Any changes to the mineral powder type or quantity may have a significant effect on the properties of the MICROFs [10]. Some authors state that the most often used mineral powders are asbestos, talc, silica, hydrated lime, limestone dust, Portland cement, slate dust, and pulverised fuel ash [11]. Among them, the most usual mineral powder is the Portland cement [9].

Nevertheless, there have been studies undertaken in this field examining the use of alternative mineral powders. Gujar et al. [11] successfully used machine learning techniques to predict and validate the use of fly ash, high calcium ash, and copper slag. Fly ash was also employed by Nikolaides and Oikonomou [12] in place of cement for micro-surfacing treatments. According to the findings, fly ash is more active than cement, hence less additive is necessary than in the case of cement. Keymanesh et al. [13] employed steel slag from electric arc furnaces (EAF) in percentages of 0%, 25%, 50%, 75%, and 100% as an alternative mineral powder for micro-surfacing treatments. All the combinations that were tested and contained EAF steel slag met the requirements, with the mixture giving the best performance when it contained 50% of alternative mineral powder. Ziari et al. [10] compared the use of coal waste powder (CWP) and natural aggregate mineral

Fig. 2. Detail of the mineral powders used in this research.

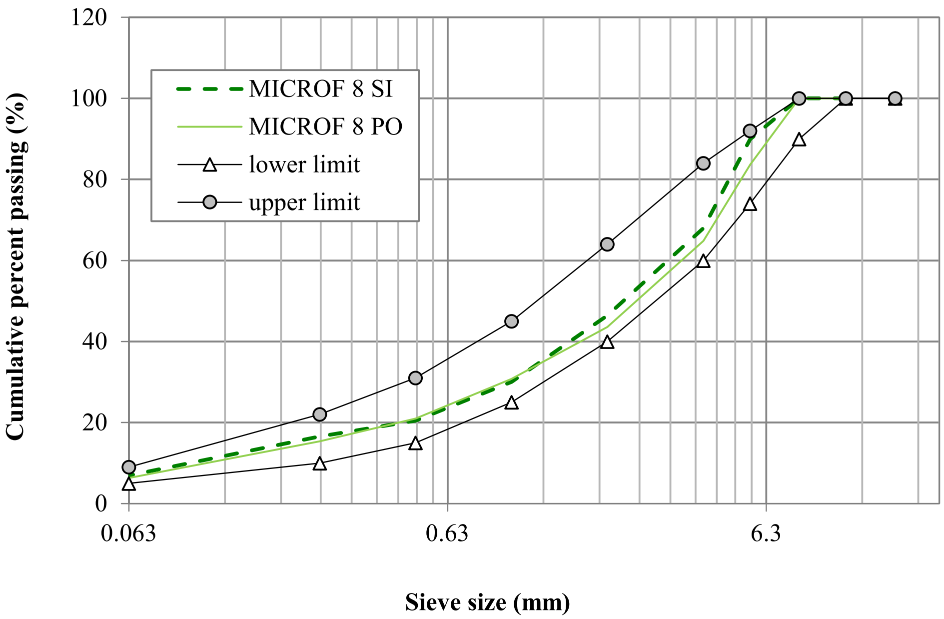


Fig. 3. Grain size distribution of the MICROF 8.

powders in MICROFs. They found increased bitumen adhesion, improved abrasion strength, and reduced bleeding potential when using 5% of CWP as mineral powder. Xu et al. [14] used toner to replace 50% of the mineral powder in coloured micro-surfacing treatments. Nevertheless, these authors did not analyze the effect of the use of toner on the micro-surfacing treatments performance.

According to some authors, more research into the use of such alternative materials could improve the environmental friendliness of micro-surfacing technology [15]. It is also necessary to promote the circular economy to avoid landfilling materials that still have a high value in the construction sector.

In this regard, in the current research, the performance of the

Fig. 4. MICROF 8 manufacture at laboratory scale to determine the breaking time.

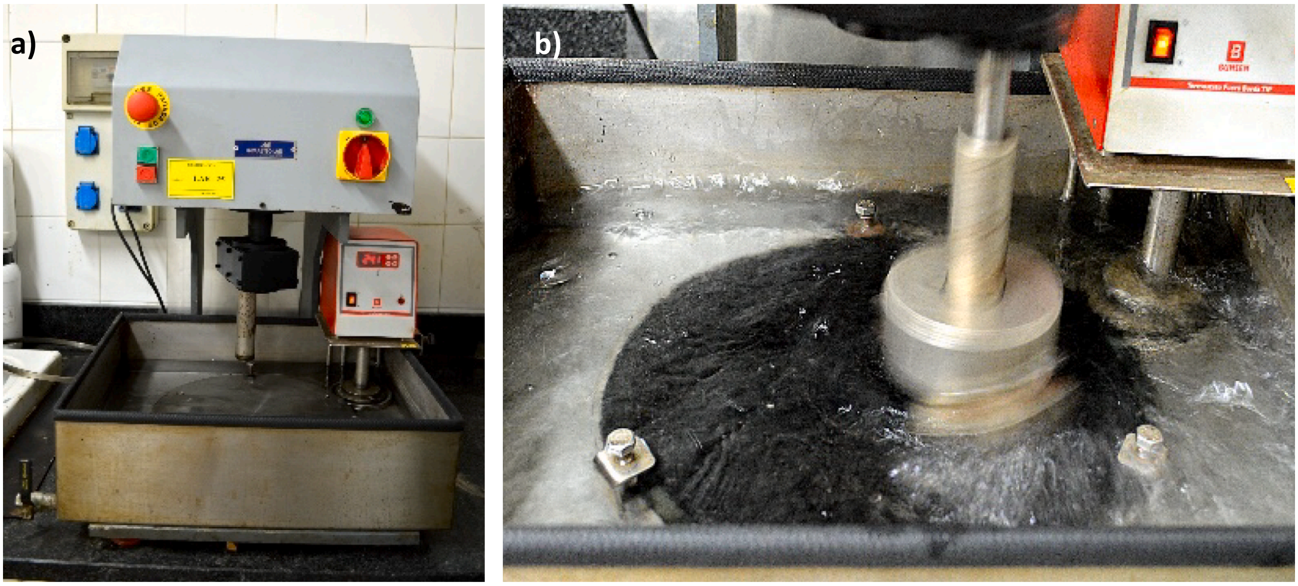


Fig. 5. Wet abrasion test: a) Equipment and b) detail of the test.

MICROFs made with the traditionally used active mineral powder, Portland cement, as well as alternative environmentally friendly and economically advantageous mineral powders, such as calcium carbonate, dolomite, PAVAL, and dust from unused toners, has been analysed and compared.

2. Aims and scope

The effectiveness of using different mineral powders to manufacture micro-surfacing treatments is examined in the current investigation. For this, four mineral powders of various chemical and mineralogical natures–calcium carbonate, dolomite, PAVAL, and mineral dust from the recycling of used toner cartridges–as well as Portland cement (control) are utilised to compare the mechanical properties of the MICROF.

These four alternative mineral powders have been selected based on environmental criteria, considering their great abundance in the earth's crust (calcium carbonate and dolomite) and/or the fact that they can be obtained as waste from various industrial processes (PAVAL and toners).

To ensure that there are several environmentally friendly and economically viable substitutes for the traditional mineral powder, the purpose of this research is to confirm the potential employability of these four alternative mineral powders. Additionally, all of this is being examined to see whether it might help micro-surfacing perform better.

For this, as illustrated in Fig. 1, firstly, a characterization of the five mineral powders used (Portland cement, calcium carbonate, dolomite, PAVAL and toners) has been carried out. Thus, its granulometry was obtained by means of an air jet sieve, its density, and its chemical composition by means of X-ray fluorescence (XRF) techniques.

After that, the MICROF's performance was examined. A MICROF 8 has been employed throughout the entire study because it is one of the

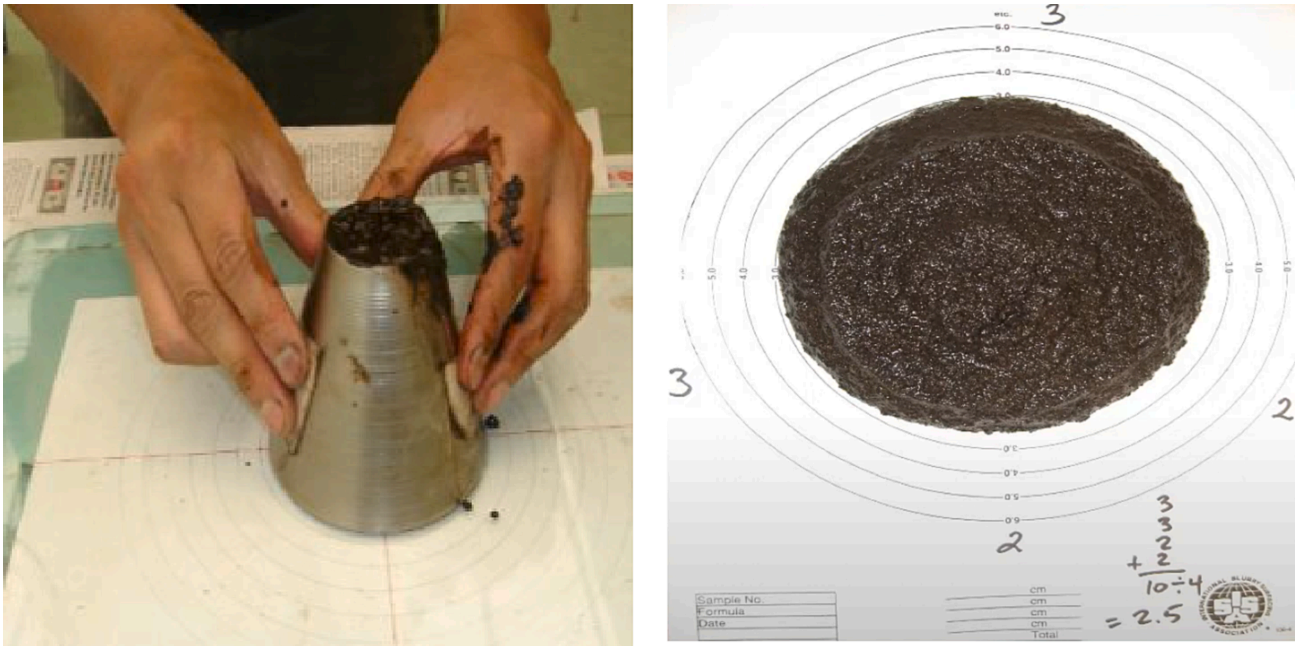


Fig. 6. Consistency test: a) Flow scale with the truncated cone and b) detail of the flow measurement.

Fig. 7. Cohesion test: a) Cohesiometer and b) Torque measurement of a sample of MICROF 8.

Table 4
Cumulative percent passing (%).

Sieve (mm)	Cement	Calcium carbonate	Dolomite	PAVAL	Toner
2	100	100	100	100	100
0.125	97.5	94.5	95.6	92.8	94.1
0.063	85.6	82.5	84.3	80.3	93.2

most popular micro-surfacing procedures. To be able to generalise the findings of the research, two different types of aggregates–one of a siliceous nature and the other of a porphyritic character–have been used to manufacture the MICROF 8. A C60BP4 MIC bitumen emulsion was employed. Water and a hardening-controlling chemical had to be used in addition.

Regarding the mechanical properties, the MICROF́s mixing time, setting and curing characteristics, and workability were analyzed according to the NLT-316 standard [16]. Additionally, the resistance to the

Table 5
Chemical composition (%).

Compound	Quantity (%)				
	Cement	Calcium carbonate	Dolomite	PAVAL	Toner
CaO	53.4	47.1	40.2	1.9	0.727
SiO_2	19.2	0.5	0.1	4.5	5.263
Al_2O_3	6.0	0.3	0.1	68.1	0.344
SO_3	0.42	–	0.01	0.4	–
Fe_2O_3	4.0	0.1	0.04	2.2	40.901
MgO	2.3	9.7	16.0	4.9	1.877
K_2O	1.2	–	0.01	0.5	–
TiO_2	0.8	–	0.01	0.7	0.434
P_2O_5	0.3	–	0.01	–	0.244
Na_2O	–	–	–	1.0	1.461
ZnO	–	–	–	–	0.065
Loss on ignition (LOI)	6.7	41.3	43.3	13.8	46

loss of particles by the wet track abrasion test in accordance with the EN 12274-5 [17] standard was also analyzed. Likewise, the consistency assessment was done in compliance with the EN 12274-3 [18] standard. Finally, the cohesion was attained in accordance with the EN 12274-4 [19] standard to certify the opening time to traffic.

3. Materials and methods

3.1. Quarry aggregates

In this research, to generalise the research's findings, two aggregates were chosen, siliceous (SI) and porphyritic (PO), which are the two main types of aggregates used in Spain for micro-surfacing treatments. Table 1 contains the main properties of both aggregates. The aforementioned data show that the aggregates are in compliance with the General Technical Specifications for Road and Bridge Works (PG-3) [1].

3.2. Bitumen emulsion

According to the EN 13808 [25] standard, a slow-setting cationic bitumen emulsion type C60BP4 MIC was employed to manufacture the MICROF. It was chosen because it is the most common type of bitumen emulsion used in this kind of treatment in Spain for medium to high traffic and for all the weather conditions [1]. Table 2 provides a summary of its primary attributes.

3.3. Water

Water from the Tierra de Arévalo industrial estate's irrigation system in the Ávila province (Spain) is utilised to manufacture the micro-surfacing treatment. This decision is supported by the fact that the manufacturing of these treatments frequently uses this kind of supply source.

3.4. Additive

ADDIBIT from Química de los Pavimentos has been used as hardening-controlling additive. Table 3 lists its main properties.

3.5. Mineral powders

As shown in Fig. 2, five mineral powders have been used in the present research:

- Portland cement (control): it's a commercial cement type CEM II B-L 42.5 N.
- Calcium carbonate: it is a commercial mineral powder with a calcium carbonate content higher than 98%.
- Dolomite: it is a commercial mineral powder mainly composed of calcium carbonate (53.90%) and magnesium carbonate (45.10%).
- Paval: it is a waste from the aluminium industry, made up of inorganic oxide powder, mainly alumina (68–73% of Al_2O_3), gray in color, with a granulometry of less than 150 microns, and with a density of 3.2 g/cm^3.
- Toner: it is a commercial powder obtained from unused toner.

3.6. Grain size distribution of the micro-surfacing treatment

The MICROF 8 granulometric spindle was selected because it is one of the most widely used for both SI and PO aggregates. Fig. 3 shows the selected granulometry for the MICROF 8 manufactured with SI aggregates (MICROF 8SI) and the MICROF 8 made with PO aggregates (MICROF 8 PO), as well as how it relates to the upper and lower limits of the PG-3 [1].

3.7. Mineral powder characterization

The suitability of the five mineral powders (cement, calcium carbonate, dolomite, PAVAL and toner) to be used in the micro-surfacing manufacture, has been carried out by studying three of their properties.

Firstly, its grain size distribution by sieving in a jet of air following the EN 933-10 [37] standard has been determined. In this test, a sample of 50.0±1.0 g of each of the mineral powders dried in an oven at 110 ±5 °C until constant mass, is sieved through the 2 mm, 0.125 mm, and 0.063 mm sieves, one by one, in increasing order. To do this, a dry air suction pressure of 3.0 ± 0.5 kPa is applied under each sieve, which must be properly covered with a plexiglas lid during the entire test. After three minutes of sieving, the sample mass retained by each sieve is noted.

Secondly, its fineness and activity have been analyzed by determining its loose bulk density in kerosene, following the procedure provided for in standard 1097-3 [38]. To this purpose, for each mineral powder, 3 samples of 10 g are prepared, dried in an oven until constant mass at 110±5 °C. Each sample is immersed in a graduated cylinder with 25 ml of kerosene, shaken several times, and allowed to settle for 6 h. After that time, the apparent volume of the mineral powder is determined by taking the reading on the test tube. Using the apparent volume and mass, the loose bulk density is determined.

In third and last place, its chemical composition was obtained by X-ray fluorescence (XRF) techniques using the Bruker S8 Tiger X-ray fluorescence equipment.

3.8. Mixing time, setting, and curing characteristics, and workability

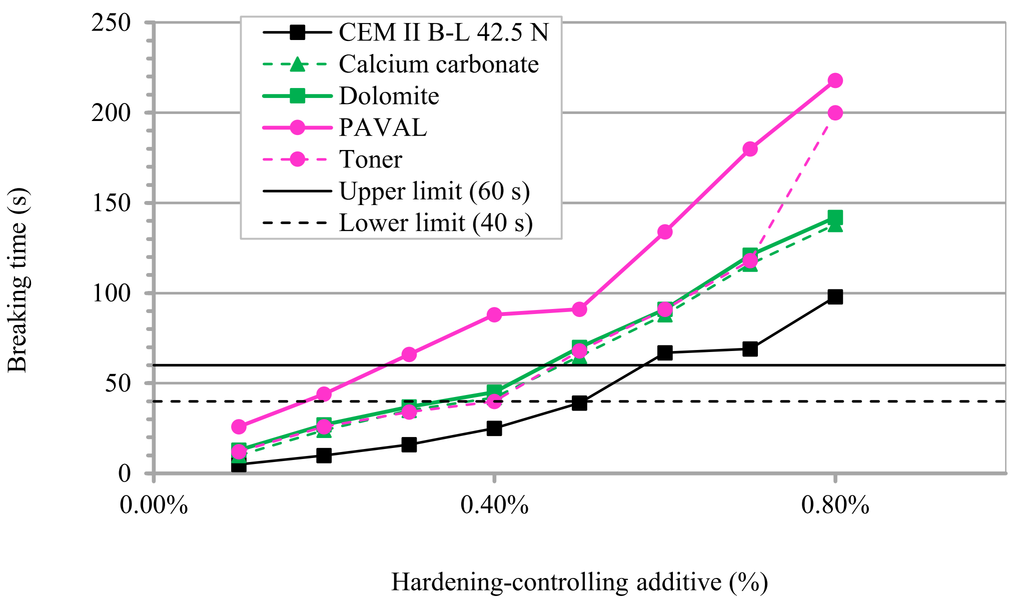
The NLT-316 standard [16] was followed in the analysis of the mixing time, setting and curing characteristics, and workability. For this, a MICROF 8 sample must be manually manufactured on a laboratory scale, using various concentrations of a hardening-controlling additive along with the same amounts of mineral powder, bituminous emulsion, and water. Particularly, the following quantities were employed:

- 0.50% of mineral powder, which is typically used in this kind of treatments.
- 11% bituminous emulsion, because is the minimum specified by the current standards (PG-3) [1].
- 10% water, as it was discovered after a series of tests that this is the one that enables an easier coating.

The amount of hardening-controlling additive was varied until breaking times of between 40 and 60 s were achieved, because, from experience, it has been verified that they are the ones that have an optimal transfer on an industrial.

Due to two types of aggregates (PO and SI) and five types of mineral

a) SI aggregates



b) PO aggregates

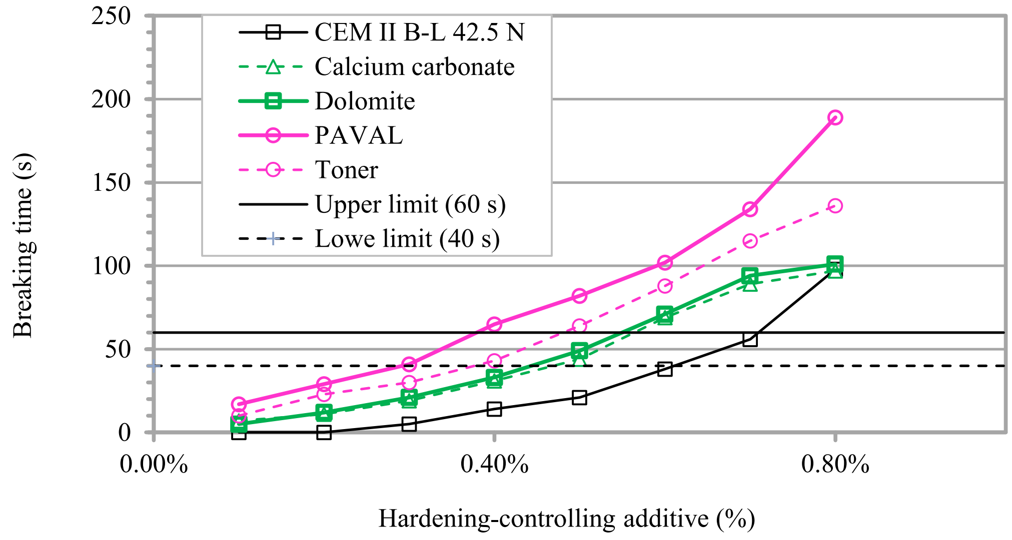


Fig. 8. Breaking time for: a) SI aggregates and b) PO aggregates.

Table 6
Two-way ANOVA analysis for the SI aggregates (breaking time).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Additive	89043.575	7	12720.51071	53.5515896	1.53427E-14	2.359259854
Mineral Powder	17295.35	4	4323.8375	18.20275746	1.80665E-07	2.714075804
Error	6651.05	28	237.5375			
Total	112989.975	39				

powders (cement, calcium carbonate, dolomite, PAVAL and toner) have been used, it has been necessary to manufacture a total of 10 different MICROF 8 samples. And each one of them, with different amounts of additive (from 0.10% to 0.8% with increases of 0.10%).

To manufacture each of the micro-surfacing sample, first, the aggregate was mixed with the mineral powder. Next, the hardening-controlling additive and water were added, and finally, with all of this mixed, the bitumen emulsion was added. From then on, and without

Table 7
Two-way ANOVA analysis for the SI aggregates (breaking time).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Additive	60516.18	7	8645.17	61.31	2.66E-15	2.36
Mineral powder	12973.65	4	3243.41	23.00	1.66E-08	2.71
Error	3947.95	28	141.00			
Total	77437.78	39				

Table 8
Additive optimum quantities (%).

Mineral powder	MICROF 8 (SI)	MICROF 8 (PO)	Selected additive content
PAVAL	**0.20**	0.30	**0.2**
Toner	**0.40**	0.40	**0.4**
Dolomite	**0.40**	0.50	**0.4**
Calcium carbonate	**0.40**	0.50	**0.4**
CEM II B-L 42.5 N	0.50	**0.60**	**0.6**

ceasing to stir manually with a spatula (Fig. 4), the time elapsed until the emulsion broke was recorded, which was determined visually by a decrease in fluidity and by the change colour of it, from brown to black.

3.9. Resistance to the loss of particles

Wet abrasion testing in accordance with EN 12274-5 [17] is required to assess the resistance to particle loss. The water and mineral powder contents established in the preceding section (0.5% mineral dust and 10% water) as well as the additive content identified using the test described in the preceding section were adopted for this purpose. For each aggregate (PO and SI) and for each mineral powder (cement, calcium carbonate, dolomite, PAVAL and toner), MICROF 8 samples were manufactured, varying the bitumen emulsion contents (9.5%, 11.5% and 13.5%). These samples were subjected to a standard abrasion (Fig. 5), after which the loss of particles is quantified. Using this test, the optimum bitumen emulsion content is selected as the minimum emulsion content that ensures the maximum particle loss can be identified. For medium and high traffic, PG-3 [1] defines a maximum value of 350 g/m^2 for particle loss by abrasion. However, the MICROF 8 will function better because it will be more durable and resistant to the effects of traffic if this loss is reduced.

3.10. Consistency

The consistency test with the cone is performed in accordance with EN 12274-3 [18] standard to examine the impact of water. For this test, a manufactured sample of MICROF 8 will be poured inside a truncated cone of 75±1 mm high, with a 40±1 mm upper internal diameter and a 90±1 mm of lower internal diameter. The cone is positioned on the centre circle of an eight-circle flow scale (Fig. 6a). The circles are spaced 10 mm, with the inner circle having a diameter of 90 mm. A smooth vertical motion lifts and flushes the cone. After 10±2 s, the flow of the MICROF 8 is measured using the scale at four points 90° distant from one another (Fig. 6b). The average of the 4 values is termed as the consistency.

PG-3 [1] establishes the maximum flow value for these treatments at 20 mm.

The five examined mineral powders (cement, calcium carbonate, dolomite, PAVAL and toner) were used to manufacture the MICROF 8 with PO and SI aggregates. In each case, the emulsion and additive contents were those found in the earlier testing, and the amount of mineral dust was 0.5%. Samples of MICROF 8 with different water contents (8%, 10%, and 12%) were produced.

3.11. Cohesion

To evaluate the traffic opening times, the cohesion test is used, according to the EN 12274-4 [19] standard. To this purpose, with a cohesiometer (Fig. 7a), measurements of the torque (Fig. 7b) are made on the same sample of MICROF 8. In the present investigation, MICROF 8 has been manufactured with SI-type aggregates and with PO-type aggregates. And for each type of aggregate, the five mineral powders studied have been used (cement, calcium carbonate, dolomite, PAVAL and toner). In all cases, the amount of mineral powder was 0.5%, and the emulsion, water, and hardening-controlling additive contents were those determined in the previous tests. Once the sample is manufactured, it is poured into circular molds of small height (±1%) and torque measurements are made at time intervals between 5 and 120 min after molding. In the present investigation, measurements have been made at 0, 15, 20, 30, 60, 90 and 120 min at room temperature (18–28 °C).

The PG-3 [1] establishes the minimum value for these treatments at 2 N m at 30 min, in the most demanding case for heavy traffic.

4. Results and discussion

4.1. Mineral powder characterization

The grain size distributions of the mineral powders are listed in Table 4. The five mineral powders are comparable in terms of granulometry, as can be shown, hence the variations in their performance cannot be attributed to these variances.

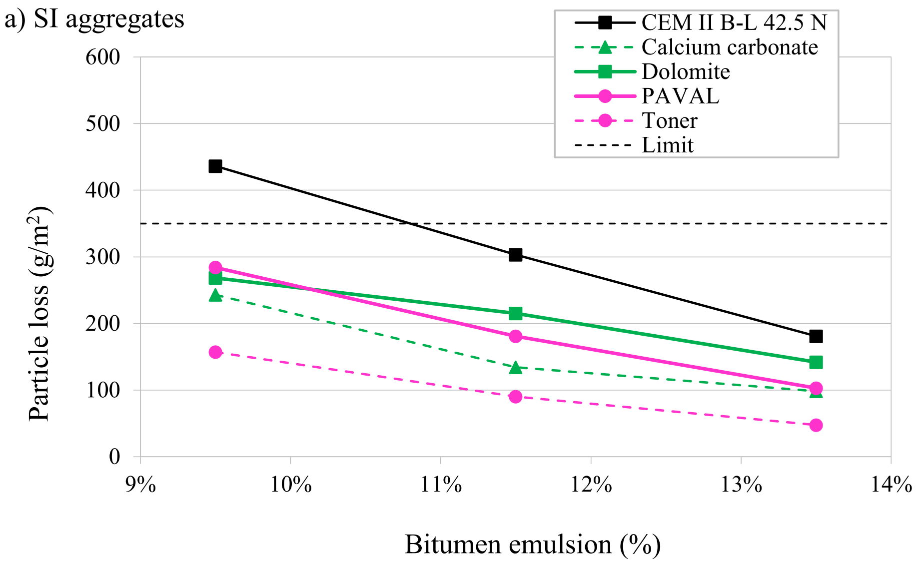
However, there are noticeable variances in the loose bulk density, with the PAVAL (0.745 g/cm^3) being the least dense and the toner residue (1.147 g/cm^3) being the densest. Dolomite (0.928 g/cm^3), carbonate (0.925 g/cm^3), and cement (1.025 g/cm^3) are three minerals with comparable densities. This illustrates how each of them engages in a distinct type of activity. In this regard, higher densities are typically correlated with lower activity. Although the PG-3 [1] specifies that these densities should be in the range of 0.5–0.8 g/cm^2, cement is frequently used despite being outside of this range. Therefore, using alternative mineral powders is possible without adhering to this criterion.

Table 5 shows that three groups can be established in terms of the chemical composition. Cement, calcium carbonate, and dolomite, which mostly consists of calcium and magnesium oxides, would be used to create the first of them. The PAVAL, which is primarily made of aluminium oxide, would be included in the second group. The toner mineral dust, which is primarily made of iron oxide, is found in the third. Since iron oxide is the heaviest of the components and aluminium oxide is the lightest, these groupings match the density values in Table 4.

4.2. Mixing time, setting, and curing characteristics, and workability

Fig. 8a shows the breaking times depending on the type of mineral powder and the percentage of additive used for the MICROF 8 manufactured with SI aggregate. Fig. 8b show the same results but for the MICROF 8 manufactured with PO aggregate.

In both cases, as expected, the breaking time increases as the percentage of additive increases for all the mineral powders. However, there are notable differences in breaking times depending on the type of mineral powder used. In this regard, cement is the mineral powder that requires the greatest consumption of additive for a given breaking time and PAVAL the least. Calcium carbonate, dolomite and toner are in an intermediate situation. This may be due to the chemical composition of the mineral powders because cement has the highest calcium oxide content and PAVAL has the least. The toner, which according to this premise should perform like the PAVAL does not, probably due to its


a) SI aggregates

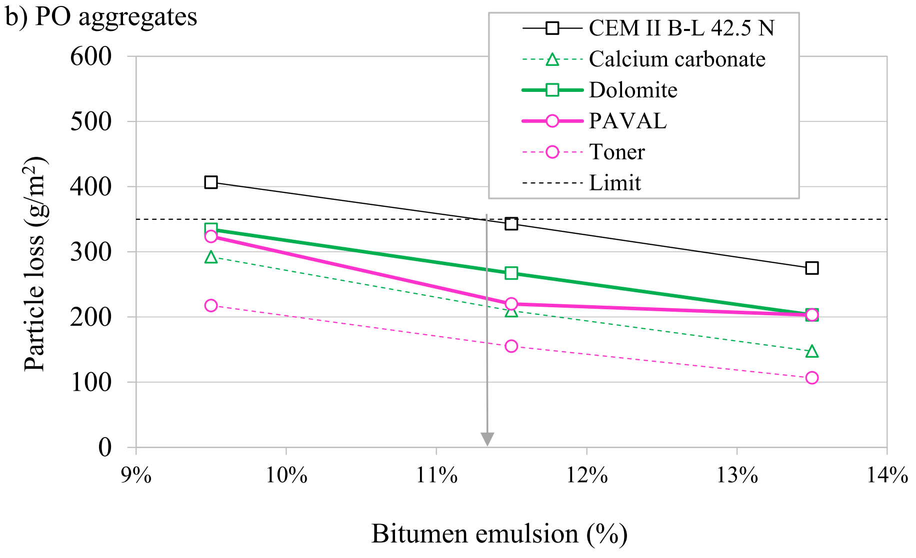

b) PO aggregates

Fig. 9. Abrasion results for: a) SI aggregates and b) PO aggregates.

high iron content, which stabilizes the emulsions.

This performance is evident with the two types of aggregates, although with the particularity of a lower dispersion in the case of SI aggregates. This may be because PO aggregates generally consume more additive and therefore the influence of iron is minimized.

These trends are supported by statistical analysis. Tables 6 and 7 show the two-way analysis of variance (ANOVA) for the micro-surfacing breaking time. As can be seen, the p-value is less than 0.05 for both factors (type of mineral powder and percentage of additive) and both aggregate types (SI and PO). That is, the results are statistically significant.

Table 8 shows the optimum percentages of additive, obtained from Figs. 8a and 8b. To carry out the following tests, to avoid problems during the pouring into the moulds, the additive contents determined for the SI aggregates were used. In the case of cement, because of the high porphyry-cement activity, the value of the aggregate PO was adopted.

It is noteworthy to note that in all instances, strong cohesion was obtained during the testing to determine the breaking time (by visual evaluation). The coating conditions were deemed to be good because neither fines segregation nor aggregate detachment were seen.

4.3. Resistance to the loss of particles

For the MICROF 8 with SI aggregate, Fig. 9a shows the results of the loss of particles due to abrasion based on the type of mineral powder and the percentage of bitumen emulsion, whereas Fig. 9b shows the same results for the MICROF 8 with PO aggregates.

As can be seen, as the bitumen emulsion percentage rises, the abrasion loss decreases. Because the aggregates are bound together by the bitumen in the emulsion, this outcome is expected. Therefore, MICROF 8 becomes more resistant to the effects of water and traffic as the bitumen percentage rises.

The acquired abrasion values also show the aggregate's nature's significant influence. Abrasion levels are often lower in PO aggregates. This was expected because the PO aggregates have improved mechanical characteristics, such as greater aggregate-binder affinity, lower wear values, and higher polished stone values.

Regarding the type of mineral powder, for both types of aggregate, toner (Fig. 10a) is the one that presents the least amount of abrasion, and cement (Fig. 10b) the most. In this regard, when PO aggregates are used, the particle loss of the MICROF 8 made with cement is between 87% and

Fig. 10. Abrasion samples for MICROF 8 made with SI aggregates and 11.5% of bitumen emulsion: a) toner as mineral powder and b) cement as mineral powder.

Table 9
Two-way ANOVA analysis for the SI aggregates (loss of particles).

Source of variation	Sum of squares	Degrees of freedom	Media of squares	F	p-value	Critical F
Bitumen emulsion	123928.311	2	61964.156	6702.853	1.71E-40	3.316
Mineral powder	161231.689	4	40307.922	4360.231	5.315E-41	2.689
Interaction	3608.57778	8	451.072	48.79387	4.21E-15	2.266
Within	277.333333	30	9.244			
Total	289045.911	44				

Table 10
Two-way ANOVA analysis for the PO aggregates (loss of particles).

Source of variation	Sum of squares	Degrees of freedom	Media of squares	F	p-value	Critical F
Bitumen emulsion	201352.71	2	100676.36	14246.65	2.13E-45	3.32
Mineral powder	210354.76	4	52588.69	7441.80	1.77E-44	2.69
Interaction	22366.18	8	2795.77	395.63	3.09E-28	2.27
Within	212	30	7.07			
Total	434285.64	44				

158% higher than when toner as mineral powder is used. When SI aggregates are used, the particle loss of the MICROF 8 made with cement is between 178% and 280% greater than the loss of particles in the MICROF 8 made with toner. The behaviour of the other three mineral powders, PAVAL, calcium carbonate, and dolomite, is in the middle.

The statistical analysis confirms these findings. In this regard, Tables 9 and 10 include a two-way analysis of variance (ANOVA) for resistance to loss of particles. As can be seen, the p-value for all factors (type of mineral powder and percentage of bitumen emulsion) and both types of aggregates (SI and PO) is less than 0.05. That is, the findings are statistically significant.

Therefore, for a given percentage of bitumen emulsion, the abrasion values would fluctuate noticeably depending on the mineral powder employed, leading inevitably to more resilient treatments that can withstand the impact of traffic. Toner mineral powder can be used in this way to lessen abrasion loss in systems with limited values or in treatments that are more demanding.

As said before, according to the specifications [1], the minimum bitumen emulsion content for MICROF 8 is 11%. The MICROF 8 manufactured with all the mineral powders comply with the abrasion limit of 350 g/cm^2 [1] when using this minimum bitumen emulsion content, except when manufacturing MICROF 8 the cement in the case of the MICROF 8 with PO aggregates. In this case, to comply with the abrasion limit, 11.3% of bitumen emulsion content should be used. Nevertheless, the next tests, for homogeneity, have been performed using as optimum bitumen emulsion content the minimum of 11%.

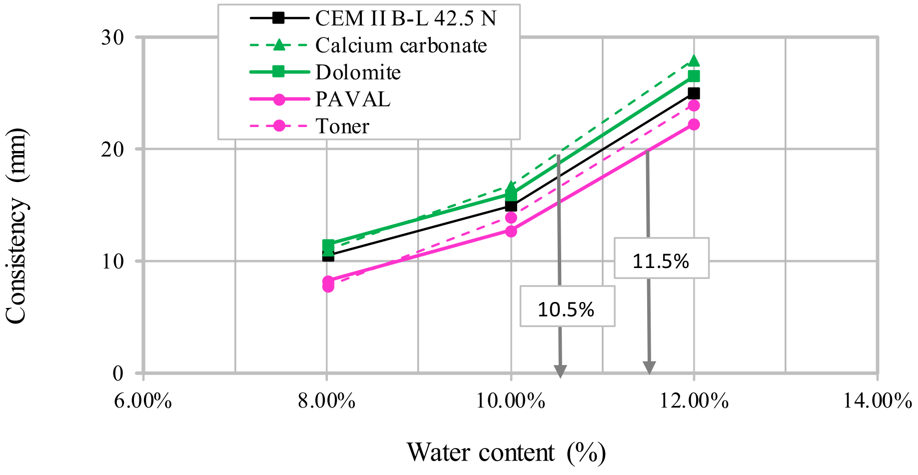
4.4. *Consistency*

Fig. 11a shows the consistency depending on the type of mineral powder used and the percentage of water used for manufacturing the MICROF 8 with SI aggregates, while Fig. 11b show the same results for the MICROF 8 manufactured with PO aggregates.

As expected, in all cases, the consistency increases with increasing water content. Particularly, for water contents greater than 10% (10.4% to 11.5% depending on the type of aggregate and mineral powder), the maximum limit established in PG-3 (20 mm) is exceeded. Then, this water content has been selected for the next tests.

The influence of the type of mineral powder on the consistency is rather scarce, obtaining very similar values in all cases. In this regard,

a) SI aggregates



b) PO aggregates

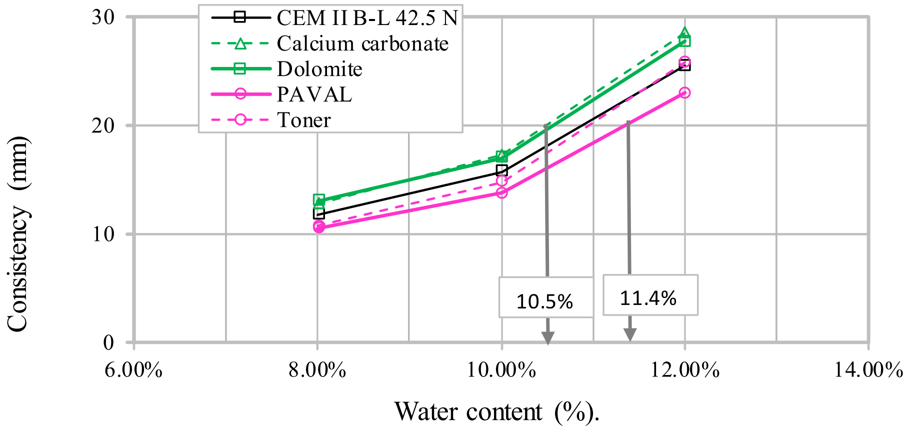


Fig. 11. Consistency for MICROF 8 manufactured with: a) SI aggregates and b) PO aggregates.

Table 11
Two-way ANOVA analysis for the SI aggregates (consistency).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Water content	611.16	2	305.58	680.64	1.17E-09	4.46
Mineral powder	37.61	4	9.40	20.94	2.69E-04	3.84
Error	3.59	8	0.45			
Total	652.36	14				

Table 12
Two-way ANOVA analysis for the PO aggregates (consistency).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Water content	550.30	2	275.15	647.16	1.42E-09	4.46
Mineral powder	29.07	4	7.27	17.09	5.52E-04	3.84
Error	3.40	8	0.43			
Total	582.77	14				

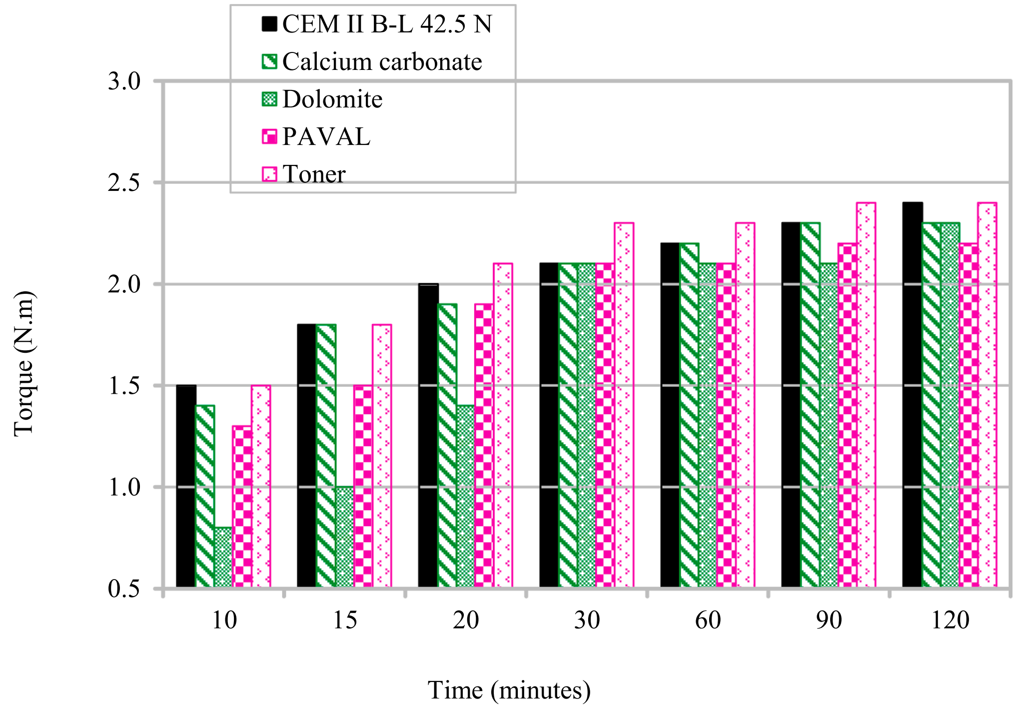
Table 13
Materials quantities (%).

Material	Type of mineral powder	Quantities (%)
Hardening-controlling additive	PAVAL	0.20
	Toner	0.40
	Dolomite	0.40
	Calcium carbonate	0.40
	CEM II B-L 42.5 N	0.60
Water		10
Bitumen emulsion		11
Mineral powder		0.50

the calcium carbonate is the less water consuming mineral powder and the PAVAL is the most water consuming, with a difference of only 1% for the SI aggregates and of 0.9% for the PO aggregates. This shows that the choice of one or the other mineral powder will not have much significance for the purposes of consistency. This may be because the consistency basically depends on the content of fines and total liquids in the mixture, which is not modified by the minor components.

These findings are supported by the statistical analysis. Tables 11 and 12 incorporate a two-way analysis of variance (ANOVA) for consistency in this regard. As can be seen, the p-value is less than 0.05 for both types of aggregates (SI and PO) and all of the parameters (type of mineral powder and water content). Consequently, the findings are statistically significant.

a) SI aggregates



b) PO aggregates

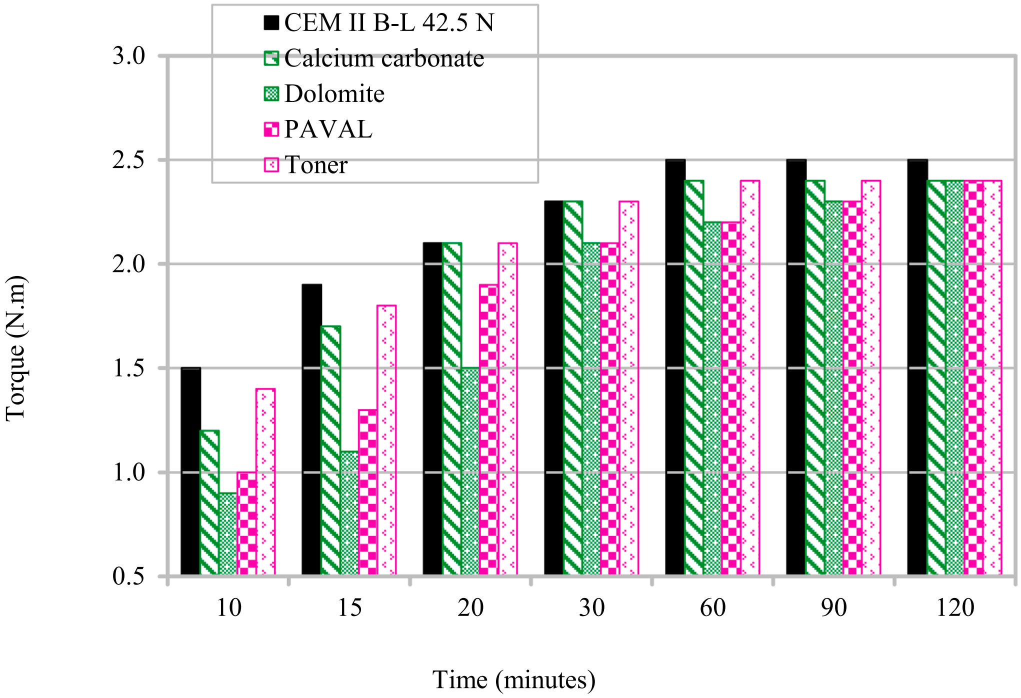


Fig. 12. Cohesion results for MICROF 8 manufactured with: a) SI aggregates and b) PO aggregates.

4.5. *Cohesion*

As previously mentioned, the contents of hardening-controlling additive, water, bitumen emulsion, and mineral powder, were determined with the previous tests. Table 13 summarizes the amounts finally used to carry out the cohesion test.

Fig. 12a shows the torque results as a function of the time it takes to reach them for MICROF 8 made with SI aggregates and the different

Table 14
Two-way ANOVA analysis for the SI aggregates (cohesion).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Time	4.45	6	0.74	36.54	6.40E-11	2.51
Mineral powder	0.77	4	0.19	9.53	9.26E-05	2.78
Error	0.49	24	0.02			
Total	5.71	34				

Table 15
Two-way ANOVA analysis for the PO aggregates (cohesion).

Source of variation	Sum of squares	Degrees of freedom	Mean of squares	F	p-value	Critical F
Time	6.61	6	1.10	69.34	5.68E-14	2.51
Mineral powder	0.78	4	0.19	12.26	1.43E-05	2.78
Error	0.38	24	0.02			
Total	7.77	34				

mineral powders analysed. Fig. 12b shows the same results, but for the MICROF 8 made with PO aggregates.

In both cases, as expected, the torque increases with the application time, due to a greater hardening of the mixture.

In a first analysis of the data, it can be observed that the choice of any of the mineral powders studied would meet the requirement of obtaining a minimum value of 2 N.m at 30 min. This fact shows that it is a MICROF of fast curing and opening to traffic. Therefore, in a first approximation it could be said that the influence of the nature of the mineral dust on the cohesion of the different mixtures is scarce.

A more thorough analysis, however, reveals significant variations in the cohesion values. Cement and toner have values that are noticeably greater than those of the other three mineral powders in this regard, especially in brief periods of time, up to 30 min, with values becoming comparable for all mineral powders after 90 min. These differences can be crucial in extreme circumstances in which high cohesion is needed in short times, night work, adverse weather conditions or any other circumstance that requires it.

According to the two-way ANOVA analysis (Tables 14 and 15), for all the factors (time and type of mineral powder) and for both aggregates (SI and PO), the p-value is less than 0.05. That is, the results are statistically significant.

5. Cost-effectiveness analysis

A cost-effectiveness analysis was performed using the average unitary costs in the first quarter of 2023. The cost per ton of the MICROF8 made with the five tested mineral powders is shown in Table 16. As can be seen, the more expensive micro-surfacing is made with the control mineral powder (CEM II B-L 42.5 N), while the less expensive is made with PAVAL, with 6.49% savings over the control mineral powder. When compared to the control mineral powder, the savings from the other tested mineral powders range from 3.51 to 3.65%.

6. Conclusions

The use of alternative, eco-friendly, and cost-effective mineral powders (calcium carbonate, dolomite, PAVAL, and leftover toners) to produce micro-surfacing treatments type MICROF 8 is the subject of this paper's research. The outcomes were contrasted with those obtained using Portland cement type CEM II B-L 42.5 N as mineral powder, which is routinely utilised for these treatments. The following are the primary conclusions:

- All the tested alternative materials comply with the technical requirements to be used as mineral powders for the manufacture of MICROF 8, regardless of whether a siliceous or porphyritic aggregate is used.
- All the alternative mineral powders have demonstrated to need less quantity of hardening-controlling additive than the typically used mineral powder (the Portland cement). Thus, their use could produce a saving of a chemical compound, thus collaborating with a greater sustainability of the micro-surfacing treatment, and with a lower economic cost. Among all the tested materials, the PAVAL is the one that requires fewer additive quantities.
- The resistance to the loss of particles is improved when use any of the alternative mineral powders. That is, the MICROF 8 manufactured with alternative mineral powders has improved performance in terms of loss of particles. Therefore, the MICROF 8 will be more resistant to the effects of water and traffic when using alternative mineral powders than when using cement. Particularly, the unused toner in the one that produces a lower loss of particles.
- The cohesion results indicate that all the mineral powders comply with the specifications. Nevertheless, in extreme circumstances in which high cohesion is needed in short times, night work, adverse weather conditions or any other circumstance that requires it, the cement is more adequate. However, the toner has also good performance in these cases.

The findings of the current study are based on a limited number of

Table 16
Cost-effectiveness analysis.

		Percentage (%) in the MICROF8 made with:					Cost (€/ton) of the MICROF8 made with:				
Component	Cost (€/ton)	CEM II B-L 42.5 N	Calcium carbonate	Dolomite	PAVAL	Toner	CEM II B-L 42.5 N	Calcium carbonate	Dolomite	PAVAL	Toner
Aggregate (fraction 0/6)	9	65.52	65.63	65.63	65.74	65.63	5.90	5.91	5.91	5.92	5.91
Aggregate (fraction 4/8)	9	16.38	16.41	16.41	16.43	16.41	1.47	1.48	1.48	1.48	1.48
Bitumen emulsion	490	9.01	9.02	9.02	9.04	9.02	44.14	44.22	44.22	44.29	44.22
Water	0	8.19	8.20	8.20	8.22	8.20	0.00	0.00	0.00	0.00	0.00
Additive	1100	0.49	0.33	0.33	0.16	0.33	5.41	3.61	3.61	1.81	3.61
CEM II B-L 42.5 N	150	0.41					0.61				
Calcium carbonate	55		0.41					0.23			
Dolomite	55			0.41					0.23		
PAVAL	75				0.41					0.31	
Toner	75					0.41					0.31
Total (€)							57.53	55.43	55.43	53.80	55.52
Savings (%)							0.00	3.65	3.65	6.49	3.51

laboratory tests. Other specifications in other countries, such as those recommended by the International Slurry Surfacing Association (ISSA), include tests that have yet to be performed but could provide more conclusive results. Similarly, a test section would be interesting to conduct.

CRediT authorship contribution statement

S. Corraliza: Conceptualization, Investigation, Resources, Writing – original draft. **A.R. Pasandín:** Writing – original draft, Writing – review & editing, Supervision. **I. Pérez:** Supervision, Writing – original draft, Writing – review & editing. **C. Delgado:** Conceptualization, Investigation, Resources, Writing – original draft.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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