



Article

Thermally Treated Waste Silt as Geopolymer Grouting Material and Filler for Semiflexible Pavements

Abbas Solouki ^{1,2,*} , Piergiorgio Tataranni ¹ and Cesare Sangiorgi ¹

¹ Department of Civil, Chemical, Environmental and Materials Engineering, University of Bologna, Via Terracini 28, 40131 Bologna, Italy; piergiorgio.tataranni2@unibo.it (P.T.); cesare.sangiorgi4@unibo.it (C.S.)
² S.A.P.A.B.A. srl (Società Azionaria Prodotti Asfaltico Bituminosi Affini), 40037 Pontecchio Marconi, Italy
* Correspondence: abbas.solouki2@unibo.it

Abstract: Considering the future shortage of natural aggregates, various researchers have promoted the recycling of by-products into various asphalt pavement types. This paper promoted a double-recycling technique, where thermally treated waste silt was used as a filler for the bituminous skeleton and grouting material of a geopolymer-based semiflexible pavement. Semiflexible pavements (SFP) inherit the flexibility of common asphalt pavements and simultaneously benefit from the rigidity of cement concrete pavements. For this purpose, waste silt obtained from a local asphalt plant was thermally treated at 750 °C and was used as the filler to produce the porous skeleton. Two different materials, including conventional cement-based and a geopolymer-based cement, were used as the grouting material. The geopolymer grout was produced by mixing metakaolin (MK), potassium-based liquid hardener and calcined silt as filler. The porous and grouted samples were characterized in terms of indirect tensile strength (ITS), the indirect tensile strength modulus (ITSM) and moisture sensitivity. The use of thermally treated waste silt as filler in porous asphalt demonstrated promising results and was comparable to the control samples produced with limestone as the filler. However, the control samples grouted with cement-based material outperformed the geopolymer grout in all aspects. Moreover, the addition of calcined silt improved the low-temperature fatigue performance of porous and grouted asphalt pavements.

Keywords: porous asphalt; filler; geopolymer grout; waste silt; filler calcination; grouted macadam



Citation: Solouki, A.; Tataranni, P.; Sangiorgi, C. Thermally Treated Waste Silt as Geopolymer Grouting Material and Filler for Semiflexible Pavements. *Infrastructures* **2022**, *7*, 99. <https://doi.org/10.3390/infrastructures7080099>

Academic Editor: Dan Bompa

Received: 14 June 2022

Accepted: 22 July 2022

Published: 23 July 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

The non-stop growth in infrastructure construction and its developments could drastically affect the worldwide supply of natural aggregates. Aggregate production plants annually produce about 3000 million tons of nonrenewable natural aggregates [1]. With that, different types of aggregate waste materials and by-products are produced, which could potentially be of a high value. However, due to the use of traditional and linear recycling strategies, waste by-products have been dumped and landfilled, giving rise to potential environmental and safety issues. The incorporation of waste materials into asphalt pavements could decrease the demand for original and natural aggregates. This would be in line with the new economic cyclic flow model [2]. In a circular economy (CE) model, the secondary raw materials are reused, rehabilitated and maintained during their cradle-to-cradle life cycle, adding extra value to a business [3,4]. The circular economy has been conceptualized from the beginning of industrialization. However, linear flow models have conquered the market [2]. For example, a comprehensive review indicated 58 boundaries and 78 possibilities related to the waste-disposal reduction (WDR) of the Australian construction and demolition sector [4]. The results indicated the landfilling elements as the third variable demonstrating the most possibilities. This clearly shows that reusing the waste material could be more beneficial than landfilling. In this regard, various researchers have promoted the recycling of various by-products and waste material into asphalt pavements to provide a circular economy. For instance, recycled plastics were

substituted with coarse and fine aggregates to produce asphalt pavements [5]. In a separate attempt, original and recycled steel slag was used to produce hot mix asphalt [6]. The field performance of on-site pavement testing sections built with recycled base layers was evaluated [7]. Up to 100% of granite was substituted with recycled concrete aggregates (RCA) to produce sustainable porous pavements [8]. However, some approaches were more direct and included the re-use of quarry dust and/or its by-products into different types of pavements. For example, marble stone fragments of up to 10 mm in size were used as a replacement for conventional aggregates in asphalt pavement. According to the Marshall values, it was indicated that marble waste could be used as aggregate for producing flexible pavements [9]. The suitability of using different tailing mines such as marl, limestone and iron ore waste in asphalt pavement was studied [10]. The results showed that cement-stabilized limestone waste showed the most promising results and could be used in Algerian road foundations.

The incorporation of waste/recycled aggregates and materials is not limited to a specific type of pavement. Semiflexible pavements (SFP) inherit the flexibility of common asphalt pavement and simultaneously benefit from the rigidity of cement concrete pavements. The SFP has a porous skeleton with high air-void content of 25 to 30%. The voids are filled with special cementitious grouting material. Improved rutting resistance, durability, skid resistance and driving comfort are merely some of the benefits of semiflexible pavements [11]. However, like any other cementitious materials, the SFPs have a higher carbon dioxide footprint when compared to traditional bituminous pavements. In an attempt to reduce the cement proportions of a grout, polyethylene terephthalate (PET) was partially added to the mixture [12]. The formulated grout also contained silica fume and fly ash. It was suggested that a grout containing 10% PET, 10% fly ash and 5% silica fume would aid the recycling of PET and reduce the usage of cement in grouts. In a separate study, the self-healing properties of an engineered cement-based composite were used to increase the resistance of SFP against cracking [13]. Rubber tire has also been added to the SFP grouts to maximize their service lifespan and durability, providing a more sustainable pavement [14].

Among various sustainable solutions, the use of geopolymer-based binders as grouting material in SFP could reduce the carbon dioxide footprint of the final product. A geopolymer network is formed when highly reactive precursors such as metakaolin (MK) are mixed with alkaline-based liquid hardeners such as potassium/sodium silicates. The precursors must be rich in aluminum and silicates to produce a strong enough 3D polymer network [15]. Geopolymer-based binders are superior to cementitious materials and have a lower carbon dioxide footprint than concrete [16,17].

Reclaimed asphalt pavement (RAP) and geopolymer-based grouts were used to produce semiflexible asphalt pavements that would not require heating or mechanical compaction energy [18]. Different geopolymer-based grouts combinations consisting of fly ash, ground granulated blast furnace slag, silica fume and metakaolin were produced and were used to grout porous asphalts made with RAP with varying air void content. The results suggested that grouts with high flowability and strength combined with low RAP content demonstrated the best results. A mixture of slag, fly ash, sodium silicate and sodium hydroxide was milled for 2 h and mixed with water to produce mechanochemical-activated geopolymer grout [19]. Different fly-ash-to-slag ratios and molarities of sodium hydroxide solutions were produced and the data were compared with conventional geopolymer cement grouts. The data revealed that the mechanical properties such as the compressive strength and ultrasonic pulse velocity of the mechanochemically grout samples were higher than those of the control mixture. A waste mud from Panasqueira (Portugal) Tungsten mine was used to produce different geopolymer-based grouts [20]. In addition, various cement-based grouting materials were produced. The mechanical properties of the final semiflexible pavements indicated better performance for cement-grouted asphalt pavements. The authors stated that the curing conditions of the geopolymer grouts need to be further studied. Nonetheless, geopolymer-grouted materials containing waste tungsten mud demonstrated valuable potential for being used as a grouting material. Several ex-

perimental applications of geopolymer for SFPs verified that the mix design phase of the grouting material is fundamental for the performance of the final material. The application of statistical modeling such as response surface methodology (RSM) could be beneficial [21]. The study successfully optimized the grouting formula based on various response outputs including fluidity, dry-shrinkage ratio and compressive strength after 7 days of curing and compressive strength after 28 days.

Reuse of quarry waste by-products is not limited to the replacement of coarse and fine aggregates of a pavement mixture. Replacing conventional filler with quarry dust and waste mineral fillers has also been considered as an alternative approach toward a sustainable pavement. In an asphalt pavement, the filler is referred to as the fine particles that mainly pass the 0.063 mm sieve (EN 13043) and generally make up about 5 to 10 percent of the total asphalt pavement. The dispersion of filler in asphalt binder produces the asphalt mastic, which is responsible for bonding the aggregates together, providing a strong load-bearing structure [22,23]. Fillers could also decrease the optimum binder content, increase pavement performance at lower temperatures and influence the thermal sensitivity and the performance of the mixtures [24–27]. The possibility of using different fly ash as filler in asphalt pavements was studied [28]. The authors claimed that up to 75% of class F or C fly ash could be used instead of the conventional limestone filler. Waste marble and granite were used as filler in asphalt pavements. The results showed superior performance for samples produced with marble waste as filler in terms of Marshall stability and resistance against moisture damage [29]. Different types of waste mineral fillers, including hydrated lime, steel slag, marble and granite, were used as asphalt fillers [30]. The results demonstrated that mixtures containing marble waste as filler had the highest Marshall stability, whereas samples produced with hydrated lime had the highest moisture resistance. Due to the vast amounts of clay in the Amazon region, calcined clay was used as synthetic aggregate for asphalt production. The clay underwent thermal treatment up to 1150 °C and was used as coarse aggregate. The authors indicated excellent physical and mechanical performance for the final product [31].

Società Azionaria Prodotti Asfaltico Bituminosi Affini (S.A.P.A.B.A. s.r.l.) is an aggregate and asphalt plant located in Bologna, Italy. During its production process, waste silt is produced. Our previous publications have demonstrated the feasibility of recycling the waste silt in cement-bound and geopolymer cement materials [32,33]. The current study aims at recycling the waste silt in a semiflexible pavement, providing an alternative solution toward a more sustainable pavement. For this purpose, the waste silt was thermally treated and then used as a filler in the open-graded asphalt skeleton of the SFP. Moreover, the calcined silt was also used as filler to produce the geopolymer-based grouting material. The feasibility of using geopolymer-based grouts in SFPs has been investigated by various researchers. However, the data on geopolymer grouts containing waste mineral fillers are very rare. Moreover, no literature was found on the double use of waste silt as filler for the asphalt concrete skeleton and the geopolymer grouting material for SFPs, making the current study a novel and innovative approach toward sustainable pavements.

2. Materials

2.1. Thermally Treated Waste Silt

The waste silt used for the current study is a by-product of the limestone aggregate production process. The raw silt was extracted from sedimentation lakes and stored in small stockpiles, allowing the excess water to evaporate. Unwanted materials such as crushed stones, wood, plant roots, etc. were separated from the raw material. The silt was then oven-dried at 120 °C for 48 h, crushed using a Los Angeles machine and sieved. The raw silt was thermally treated at 750 °C for 2 h and was then used as filler for the porous skeleton and geopolymer-based grout. A detailed description of the silt calcination and XRD results is fully discussed in our previous study [33].

2.2. Grouting Material

Two different types of grouts were used. A commercial cement-based grout was used as the control sample. The experimental grout was produced from geopolymer-based cement. For this purpose, an alkali solution consisting of potassium silicate (MR = 3.0) and sodium hydroxide (98% purity, 8 M) and water was produced. The alkali solution was prepared 24 h before mixing with precursors and waste calcined silt. The liquid part was mixed with an industrial metakaolin and stirred for 5 min. This allowed the metakaolin to blend with the liquid and produce a homogeneous mixture. The last stage continued by adding the thermally treated silt to the mixture as the filler. The mixing phase was continued for an additional 5 min.

2.3. Aggregates, Filler, Bitumen and Cellulose Fiber

The aggregates, including sand (0–4 mm) and gravel (4–8 mm), were provided from a local aggregate-production plant. The sand had bulk density (g/cm³) (EN 1097-6), sand equivalent (EN 933-8) and harmful fines (gMB/Kg) (EN 933-9) of 2.658, 90 and 0.5, respectively. The bulk density of gravel was 2.667 g/cm³. A traditional limestone filler was used as filler for the reference asphalt concrete, with a bulk density of 2.690 g/cm³.

Conventional basalt cellulose fibers were used to produce porous asphalt samples.

A PmB 25/55 modified bitumen was used as the binder for the asphalt concretes. The rheological properties of the bitumen are given in Table 1.

Table 1. Rheological properties of the bitumen.

Test	Unit	Value	Standard
Penetration @ 25 °C	Dmm	25–55	EN 1426
Softening Point	°C	70	EN 1427
Dynamic Viscosity @ 160 °C	Pa.s	0.4–0.7	EN 12596
Flash point	°C	250	EN ISO 2595

The same type of aggregates, basalt fiber and modified bitumen, were used for both gap-graded asphalt mixtures. The only change for the experimental mixture was the total substitution of limestone filler with the calcined silt.

3. Experimental Program

The experimental plan was divided into two sections. The first part investigated and verified the use of calcined silt as filler in porous asphalt, whereas the second section compared the mechanical properties of the different grouted asphalt concretes. In summary, two different types of mixtures were produced. The first batch used calcined silt as the filler and was grouted with a geopolymer binder. The second batch was used as the control and was produced with the company’s limestone filler and cement-based grout.

3.1. Gap-Graded Asphalt Mix Design

A specific mix design for gap-graded asphalt was adopted based on the aggregates available. Some preliminary tests verified the optimum binder content for the experimental mixture with waste calcined silt, which was found unchanged if compared to the reference asphalt concrete with limestone filler.

The details of the mix design are shown in Table 2. A total of 0.35% of cellulose fiber was used. The aggregate gradation curve is shown in Figure 1.

Two different mixtures were prepared based on the details mentioned in Table 2. The first set was produced with a common limestone filler (labeled as PA-C), whereas the second set was produced with calcined silt as the filler (labeled as PA-SC).

Table 2. Porous asphalt mixture proportions.

Test	Amount (%)
Optimum binder content	4.2
Aggregates	90.67
Filler	4.78

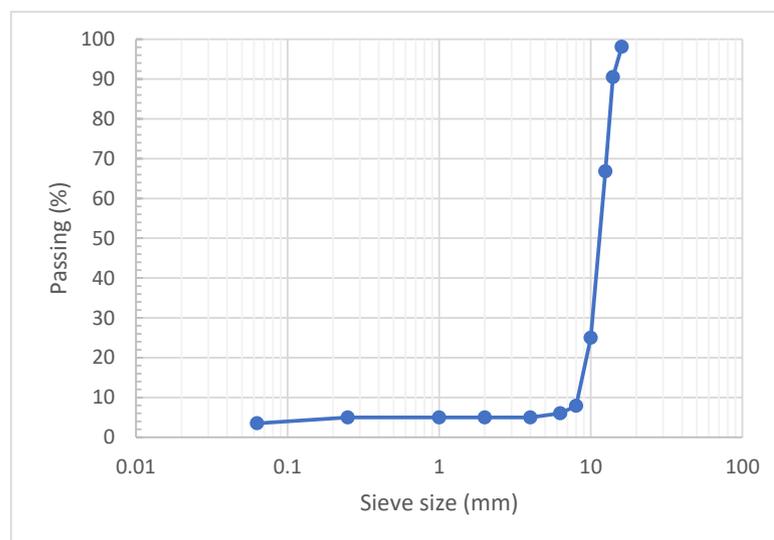


Figure 1. Porous asphalt gradation curve.

The porous asphalt samples were produced using a gyratory compactor (EN 12697-31). The internal angle of the compactor was set to 1.250 °C and a 600 kPa load was applied. Samples with a diameter of 100 mm were produced at 50 and 130 cycles applying a speed of 30 RPM.

The air-void content was calculated based on the EN 12697-8 standard. The air-void content was suggested to be between 25 to 30%, providing adequate interconnected pores, making the specimens suitable for grouting.

The mechanical properties of the mixtures were investigated with static and dynamic tests. For this purpose, a total of six samples (for each set) were produced using a gyratory compactor (50 cycles). The indirect tensile strength (ITS) (EN 12697-23) test was then conducted on the samples. Constant loading of 50 mm/min was applied until sample failure. The determined maximum load was used to calculate the ITS. Three samples were tested under dry at 25 °C conditions and the remaining three were tested after being submerged in water (40 °C) for 72 h. The susceptibility of the mixtures to moisture was evaluated by comparing the ITS of wet and dry samples based on EN 12697-12 (indirect tensile strength Ratio, ITS_R).

The indirect tensile strength modulus (ITSM) was obtained by applying controlled strain-rate loads to the specimens in the indirect tensile configuration. The test followed the EN 12697-26 Annex C standard and was conducted at three different temperatures of 10, 20 and 30 °C to evaluate the thermal sensitivity of the asphalt concretes. The samples were conditioned for at least four hours before testing.

3.2. Grout Formulation and Structure

Two different types of grouting mixtures were used for the current study. A common cement-based grout was used as the control sample. The grout was produced by mixing 70% of specific cement with 30% of water. The liquid mixture was mixed for five minutes to produce a homogenized material.

The geopolymer-based grout was produced by mixing the precursor, alkaline hardener and the calcined waste silt according to the specified ratios presented in Table 3. For this

purpose, metakaolin was mixed with potassium silicate and sodium hydroxide solution for 5 min. The calcined silt was then added as the filler and the mixing was continued for an additional 5 min. A liquid-to-solid ratio of 1.5 was adopted to ensure adequate fluidity of the geopolymer mixture, considering that the grouting material should penetrate the porous skeleton of the asphalt concrete, filling the voids.

Table 3. Geopolymer-based grout mix design.

Material	Amount (% Total Weight)
Metakaolin	24
Calcined silt	16
KOH (solution)	48
NaOH (8M)	12

The grouting mixtures were characterized in terms of unconfined compressive strength (UCS). The data are presented in Table 4. The cement and geopolymer grouts were produced according to Table 3. The mixtures were then placed inside cubic molds (4 × 4 × 4 cm) and were cured for 28 days at room temperature. The samples were unmolded and were tested for UCS using a hydraulic press. The highest strength was obtained for the control samples. It is worth mentioning that the most widespread Italian technical specifications for grouted macadams suggest 35 MPa as the lower limit for the UCS after 28 days of curing.

Table 4. Average UCS results for geopolymer-based and cement-based grouting mixtures.

Grout	UCS (MPa)
Geopolymer-based grout	13.9 ± 0.4
Control grout	38.8 ± 1.5

3.3. Grouted Asphalt Concretes

Once the mechanical properties of the grouting mixtures were defined, the asphalt samples were kept inside Marshall molds, where the bottom section was sealed to prevent any possible leaking of the grouting material. The prepared grouts were stirred thoroughly before use to ensure a homogenized mixture. The material was then poured on top of the porous asphalt specimens. This phase continued until the surface of the samples was covered with the grouting material (Figure 2). The samples were then cured at room temperatures for 24 h, demolded and cured for another 28 days. GM-C refers to the control grouted samples, whereas GM-SC designates samples containing silt-based geopolymer grouts.



Figure 2. Grouted macadam sample-producing phase.

The air-void content of the asphalt specimens was calculated before and after grouting. The data are summarized in Table 5. Both porous asphalt mixtures showed to have the required adequate porosity. The grouting of the samples dropped the air-void content to 7.11 and 8.9% for the GM-C and GM-SC samples, respectively.

Table 5. Air-void content for porous and grouted macadam asphalt samples.

Specimen	Air Voids (%)
PA-C	26.22 ± 1.86
PA-SC	25.18 ± 1.09
GM-C	7.11 ± 1.09
GM-SC	8.94 ± 2.1

4. Results and Discussion

4.1. Porous Asphalt Mixture Characterization

4.1.1. Indirect Tensile Strength Results

The effect of different asphalt fillers on the mechanical properties of porous asphalt samples was investigated through indirect tensile strength (ITS) (EN 12697-23). Three specimens were cured at 25 °C for 4 h before testing. The ITS results are summarized in Figure 3.

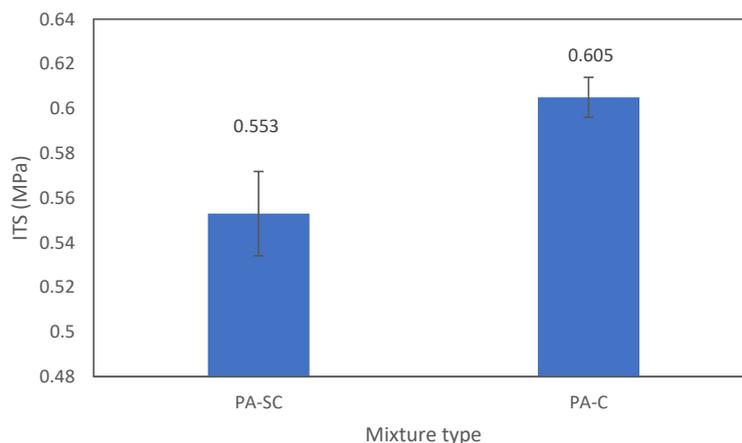


Figure 3. Average ITS for porous samples.

The control porous asphalt samples produced with common limestone filler showed an ITS value of 0.605 MPa, whereas the samples produced with thermally treated silt followed up closely showing an 11.9% loss of strength compared to the control samples. The statistical analysis showed an insignificant difference in terms of ITS between the PA-C and PA-SC samples ($p = 0.568$).

4.1.2. Indirect Tensile Strength Ratio Results

The indirect tensile strength ratio (ITSR) was used to compare the resistance of the asphalt mixtures produced against moisture damage. For each specimen type, three replicas were tested. All samples were treated in a water bath at 40 °C for 72 h before testing. The average ITSR values for the porous asphalt specimens are shown in Figure 4. The control sample produced with limestone filler had the highest resistance against moisture damage demonstrating an ITSR value of 94%. The inclusion of calcined silt led to a slight reduction in the moisture damage resistance presenting an ITSR value of 92%. The statistical analysis showed no significant difference between the two groups ($p = 0.218$). As for the previous ITS analysis, the ITSR values were above the suggested lower limit for porous asphalt ($ITSR > 75%$).

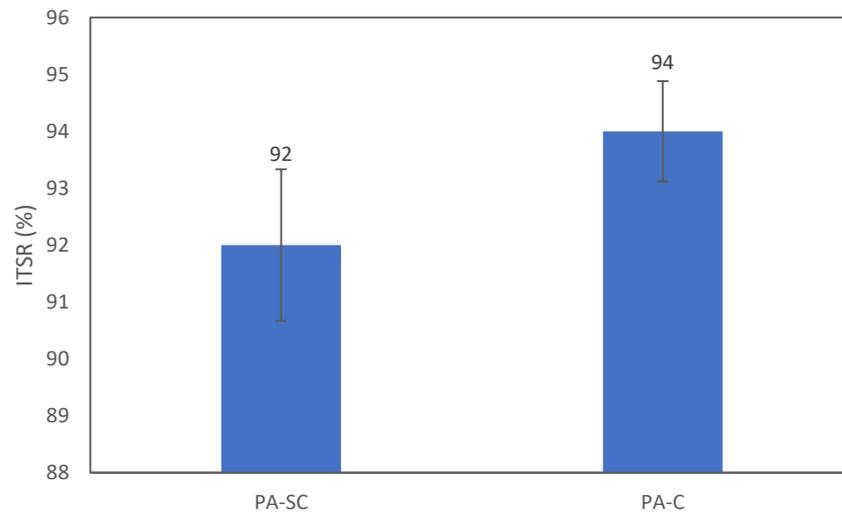


Figure 4. Average ITSR results for porous samples.

4.1.3. Indirect Tensile Stiffness Modulus Results

The summary of the average ITSM values is presented in Figure 5 for the porous specimens. The thermal sensitivity of the mixtures was studied by testing three samples (for each filler type) at 10, 20 and 30 °C. When calcined silt was used as filler in porous asphalt, lower ITSM values were obtained at every testing temperature. One-way ANOVA was conducted to determine the significance of the results for each testing temperature. The results showed that at high and low testing temperatures, a significant difference was observed between the two groups. However, samples tested at 20 °C proved to be similar in terms of ITSM values. Thus, by considering the reference temperature as 20 °C, no significant difference was observed between the mixtures.

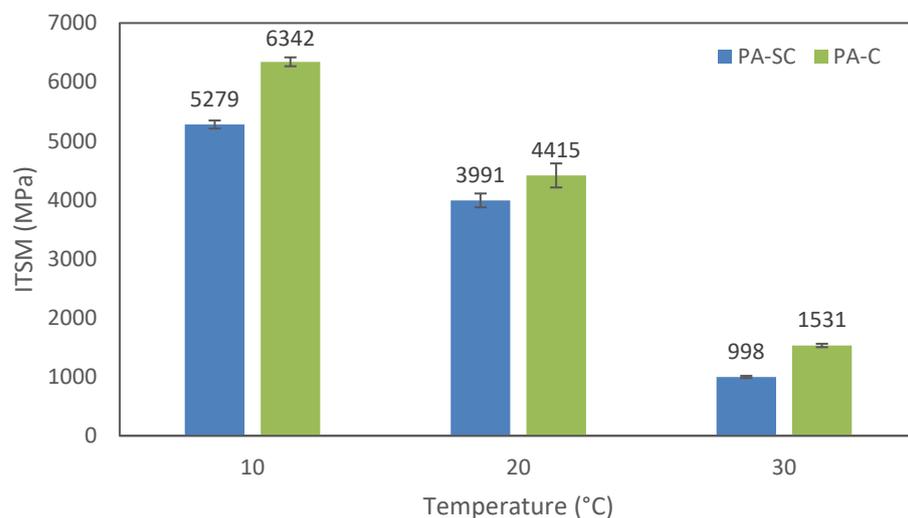


Figure 5. Average ITSM values for porous asphalt specimens.

4.2. Grouted Macadam Asphalt Mixture Characterization

4.2.1. Indirect Tensile Strength Results

The ITS data of the grouted samples are shown in Figure 6. Three grouted samples for each grout type were tested. The highest ITS value of 2.16 MPa was obtained for the control samples, whereas an ITS of 1.55 was measured for the geopolymer cement-grouted macadam specimens. The statistical analysis indicated a significant difference between the different types of grouted mixtures in terms of the ITS ($p = 0.0184$).

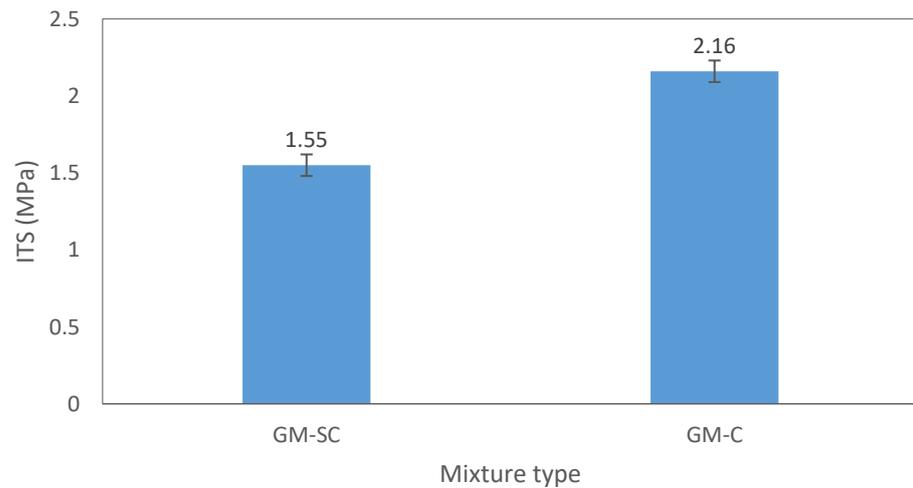


Figure 6. Average ITS of grouted mixtures.

4.2.2. Indirect Tensile Strength Ratio Results

The ITS_R of grouted samples is shown in Figure 7. The control sample presented an ITS_R of 93%, while the ITS_R of the geopolymer-grouted samples dropped by 13%. However, no significant difference was observed in terms of ITS_R between the two groups ($p = 0.061$).

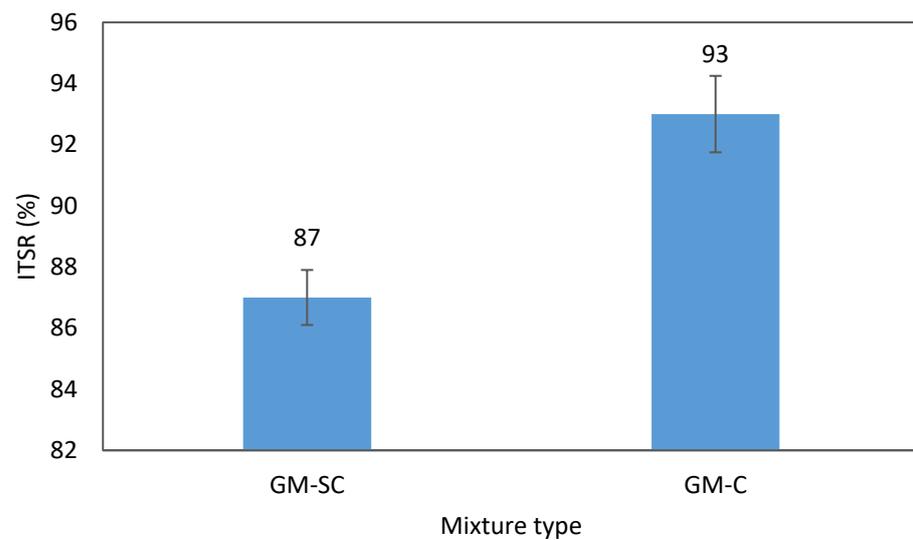


Figure 7. Average ITS_R of grouted macadam samples.

4.2.3. Indirect Tensile Stiffness Modulus Results

Figure 8 depicts the ITSM values obtained at three different testing temperatures of 10, 20 and 30 °C. The grouted samples showed an ITSM value of 8991 MPa at 10 °C, which decreased by 4.42% when compared to the control sample. The loss of stiffness was observed at each testing temperature. At 20 and 30 °C a decrease of 42.97 and 39.40% was reported, respectively.

The geopolymer cement had a low UCS value of 13.97 MPa, which was approximately 64% lower than the control grout cement.

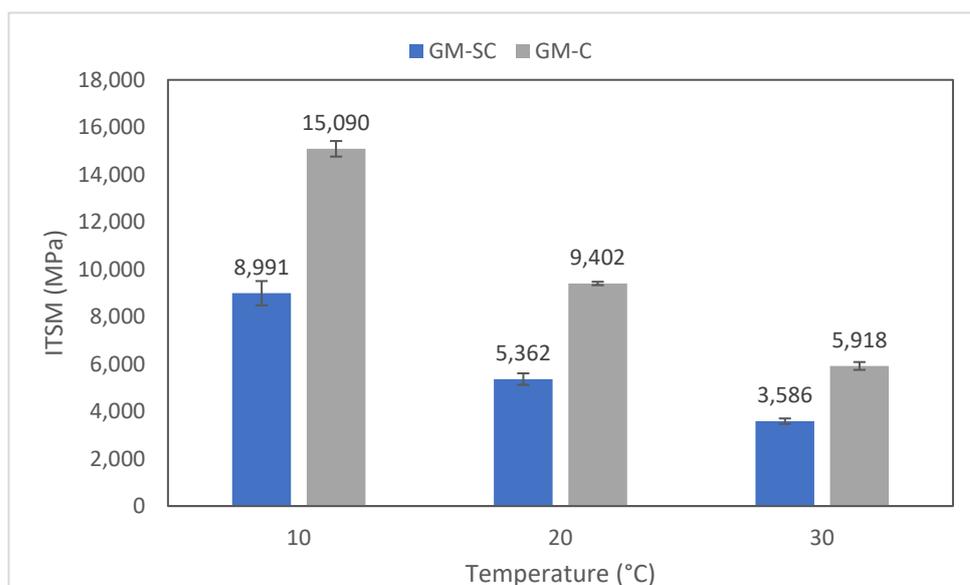


Figure 8. Average ITSM values for grouted macadam samples.

5. Discussion

Double recycling of thermally treated waste silt as a filler into the porous skeleton and geopolymer grout of a semiflexible pavement is presented.

Based on our previous analysis [33], the raw silt was found to be rich in aluminosilicate with a crystalline structure, making it a potential option to be used as filler in geopolymer cement production. However, primary results indicated that a thermal treatment at 750 °C would be in favor of the geopolymer compressive strength [33]. Since the waste filler was going to be used as the filler for the geopolymer grout, the same thermal treatment of 2 h at 750 °C was performed. The semiquantitative X-ray powder diffraction (XRD) studies conducted on the untreated silt [33], determined sharp peaks of calcite (CaCO_3). However, further examination of the XRD peaks revealed that the calcite had completely degraded into calcium oxide (CaO) and carbon dioxide at 750 °C. This was observed by the complete loss of the calcite peak. Various studies have indicated the presence of both CaO and CaCO_3 in limestone filler [34–36].

Thermally treated waste silt and limestone fillers were used to produce porous asphalt, which would serve as the skeleton of the semiflexible pavements. An indirect tensile strength test was conducted on the specimens, which is an indication of pavement strength and resistance against fatigue cracking and rutting. According to the results, the addition of waste silt did not affect the cohesion properties of the porous asphalt producing strength comparable with the control sample [37]. Based on common Italian technical specifications, the minimum required ITS for porous mixture is generally 0.40 MPa. The similar behavior of the calcined silt could be due to its similarity to the limestone filler in terms of chemical composition [38]. Porous asphalt specimens produced with calcined silt showed high resistance against moisture damage. According to the literature, a strong filler–binder bond could form in fillers that contain water-insoluble minerals such as calcite, portlandite and dolomite. Therefore, the high resistance of the calcined silt could be attributed to the presence of its calcium-based minerals [39,40].

The porous samples were then filled with different grouting materials to produce semiflexible pavement specimens. The ITS of the geopolymer-grouted pavement specimens was lower than the control sample. This could be related to the low strength of the geopolymer mortar, which was about 2.8 times lower than the control mortar. The geopolymer grout was designed in such a way to ensure enough fluidity of the sample. Thus, the liquid-to-solid ratio of the mixture used as grout was much higher than the 0.45–0.55 ratios suggested for common geopolymer binders [15,16]. Higher liquid-to-solid ratios reduce the alkaline

concentration of the mixture and could decrease the compressive strength of the sample. The results are comparable with Panesquera mine, where the addition of geopolymer grout reduced the ITS values of the asphalt specimens [20]. Decreasing the geopolymer-grout fluidity could be in favor of gaining higher ITS strength. Regarding the ITS values, the control samples grouted with the concrete-based grout showed higher resistance against moisture damage. However, both grout types showed acceptable resistance, which were within common Italian limits (>75%). As discussed earlier, higher liquid-to-solid ratios decrease the rate of geopolymerization. Thus, there would be a possibility that some of the solid particles of the geopolymer mixture remain unreacted [16]. Consequently, the presence of water particle loss could result in lower moisture resistance.

Advanced dynamic tests were conducted on both porous samples that investigated the specimen's stiffness variation in terms of thermal sensitivity. The mechanical behavior (ITSM) of the porous asphalt containing limestone as filler showed to have a higher sensitivity to temperature compared to that of the PA-SC. Very high stiffness at low testing temperatures could lead to fatigue cracking of asphalt pavements. The application of calcined silt significantly reduced the stiffness of the sample at a low testing temperature (10 °C) by 16.76% when compared to the control sample. Thus, the addition of calcined silt as filler improved the fatigue performance of the porous mixture. The performance of various asphalt fillers in terms of fatigue has been fully reviewed, and the literature shows that some fillers such as hydrated lime could have a superior performance in that regard [38].

The ITSM results for the grouted samples followed a similar trend to the porous specimens. The thermal sensitivity of the geopolymer SFP was much lower than that of the GM-C. The control grout showed better performance in terms of rutting, whereas the geopolymer mixture outperformed the control sample in terms of fatigue cracking.

SAPABA has been aiming to reuse and recycle the waste silt that is stored in its sedimentation lakes. Distancing from a linear production workflow and adopting a circular economy model could be in favor of a more sustainable production scheme. Waste minimization is one of the main objectives of a circular economy. The focus is to find methods that could serve as a sustainable alternative, which could reduce landfilling and transportation costs and decrease the non-stop exploitation of natural aggregates and resources. Consequently, this would reduce related environmental and economic costs [3,4,41]. The complete replacement of limestone with calcined silt as the filler in porous asphalt skeleton was a step towards a circular economy. The mechanical properties of the pavement were in line with the common Italian specifications. Revalorizing the waste silt can also facilitate the minimization of waste production. Consequently, the volume of landfills allocated to the silt could decrease over time. Transportation costs could be deducted dramatically, since the waste silt is produced within the company's premises. Thus, the need for transportation costs, from warehouses to the asphalt plant for example, would be eliminated. The paper has used a static furnace to calcinate the silt at a laboratory scale. However, at a commercial scale, clay calcination is conducted using flash calcination, where the clay particles are exposed to high temperatures for a fraction of a second [42]. The data revealed that replacing 30% of cement clinker with calcined clay could reduce CO₂ emissions by 40% and requires 30% less fuel [43]. A full life-cycle analysis (LCA) could further determine the environmental and economic effects of using waste silt as filler in porous and semiflexible pavements.

6. Conclusions

This paper promoted a double-recycling technique, where a thermally treated waste silt was used as filler for the bituminous skeleton and the geopolymer-based grouting material of grouted asphalt concrete. The proposed recycling technique could benefit the road industry by reducing production costs (aggregate extraction and cement production) and increasing the sustainability of road pavements. The resulted semiflexible pavement

containing waste silt was compared with a conventional grouted mixture. The main conclusions are as follows:

- The air-void content of the porous samples was within the suggested range of 25–30%. Thus, the inclusion of thermally treated waste silt as filler does not alter the workability and compactability properties of the porous asphalt concrete.
- The ITS and ITSR values of both porous samples were statistically similar. A slight decrease in the indirect tensile strength was observed when calcined silt was added. However, both values were considerably above the lower cohesion limit suggested for porous asphalt. A similar trend was observed for the ITSR.
- The ITSM values of the control porous asphalt were higher than those of the geopolymer-grouted samples.
- The addition of calcined silt improved the fatigue resistance of the porous mixture at low temperatures compared to the control sample.
- The ITS and ITSR of the grouted mixtures indicated better performance for the control mixture when compared to the experimental one. A drop of approximately 28% was observed when geopolymer was used as the grouting material. The ITSR of the geopolymer was 6.5% lower than that of the control specimen. Due to the lower mechanical performances of the geopolymer-based grouting material, the experimental samples showed lower ITSM values.
- Geopolymer-grouted mixtures showed better low-temperature fatigue properties compared to the control sample.
- Geopolymer-grouted SFP showed lower temperature sensitivity compared to the control grout.

Based on the preliminary results, the use of thermally treated waste silt as filler in porous asphalt demonstrated promising results. However, the use of waste-calcined silt to produce geopolymer-based grouting materials led to a significant drop in the mechanical properties of the final grouted asphalt concrete. Future studies will focus on the optimization of the grouting mixture aiming to replicate the performance of the cement-based one. LCA study could shed light on the environmental and economic aspects of using waste silt as filler in SFP.

Author Contributions: Conceptualization, A.S.; methodology, A.S.; validation, P.T.; formal analysis, A.S.; investigation, A.S.; data curation, A.S.; writing—original draft preparation, A.S.; writing—review and editing, P.T and C.S.; visualization, A.S.; supervision, P.T and C.S.; project administration, P.T and C.S.; funding acquisition, C.S. All authors have read and agreed to the published version of the manuscript.

Funding: This paper was written for the SAFERUP! Project, which received funding from the European Union’s Horizon 2020 research and innovation program under Marie Skłodowska-Curie grant agreement No. 765057.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Generation of Waste by Waste Category, Hazardousness and NACE Rev. 2 Activity (env_wasgen). 2020. Available online: http://appsso.eurostat.ec.europa.eu/nui/show.do?lang=en&dataset=env_wasgen (accessed on 13 June 2022).
2. Frosch, R.A.; Gallopoulos, N.E. Strategies for manufacturing. *Sci. Am.* **1989**, *261*, 144–153. [[CrossRef](#)]
3. Korhonen, J.; Honkasalo, A.; Seppälä, J. Circular Economy: The Concept and its Limitations. *Ecol. Econ.* **2018**, *143*, 37–46. [[CrossRef](#)]
4. Shooshtarian, S.; Maqsood, T.; Caldera, S.; Ryley, T. Transformation towards a circular economy in the Australian construction and demolition waste management system. *Sustain. Prod. Consum.* **2022**, *30*, 89–106. [[CrossRef](#)]

5. Xuan Lu, D.; Giustozzi, F. Recycled plastics as synthetic coarse and fine asphalt aggregate. *Int. J. Pavement Eng.* **2022**, 1–16. [[CrossRef](#)]
6. Goli, A. The study of the feasibility of using recycled steel slag aggregate in hot mix asphalt. *Case Stud. Constr. Mater.* **2022**, *16*, e00861. [[CrossRef](#)]
7. Titi, H.; Qamhia, I.I.A.; Ramirez, J.; Tabatabai, H. Long-Term Performance of Flexible Pavements Constructed on Recycled Base Layers. *Transp. Res. Rec.* **2022**, 03611981221092000. [[CrossRef](#)]
8. Nwakaire, C.M.; Yap, S.P.; Onn, C.C.; Yuen, C.W.; Moosavi, S.M.H. Utilisation of Recycled Concrete Aggregates for Sustainable Porous Asphalt Pavements. *Balt. J. Road Bridg. Eng.* **2022**, *17*, 117–142. [[CrossRef](#)]
9. Kushwaha, P.; Chauhan, A.S.; Swami, B.L. Utilization of waste materials from marble processing industry for sustainable pavement design. *Mater. Today Proc.* **2022**, *63*, 547–552. [[CrossRef](#)]
10. Djellali, A.; Laouar, M.S.; Saghafi, B.; Houam, A. Evaluation of Cement-Stabilized Mine Tailings as Pavement Foundation Materials. *Geotech. Geol. Eng.* **2019**, *37*, 2811–2822. [[CrossRef](#)]
11. Hassani, A.; Taghipoor, M.; Karimi, M.M. A state of the art of semi-flexible pavements: Introduction, design, and performance. *Constr. Build. Mater.* **2020**, *253*, 119196. [[CrossRef](#)]
12. Khan, M.I.; Sutanto, M.H.; Khan, K.; Iqbal, M.; Napiah, M.B.; Zoorob, S.E.; Klemeš, J.J.; Bokhari, A.; Rafiq, W. Effective use of recycled waste PET in cementitious grouts for developing sustainable semi-flexible pavement surfacing using artificial neural network (ANN). *J. Clean. Prod.* **2022**, *340*, 130840. [[CrossRef](#)]
13. Cai, X.; Huang, W.; Wu, K. Study of the Self-Healing Performance of Semi-Flexible Pavement Materials Grouted with Engineered Cementitious Composites Mortar based on a Non-Standard Test. *Mater.* **2019**, *12*, 3488. [[CrossRef](#)] [[PubMed](#)]
14. Davoodi, A.; Aboutalebi Esfahani, M.; Bayat, M.; Mohammadyan-Yasouj, S.E. Evaluation of performance parameters of cement mortar in semi-flexible pavement using rubber powder and nano silica additives. *Constr. Build. Mater.* **2021**, *302*, 124166. [[CrossRef](#)]
15. Liew, Y.M.; Heah, C.Y.; Mohd Mustafa, A.B.; Kamarudin, H. Structure and properties of clay-based geopolymer cements: A review. *Prog. Mater. Sci.* **2016**, *83*, 595–629. [[CrossRef](#)]
16. Davidovits, J. *Geopolymer Chemistry and Application*, 4th ed.; Institut Geopolymere: Saint-Quentin, France, 2015; ISBN 9782951482098623.
17. Davidovits, J. Environmentally Driven Geopolymer Cement Applications. 2002 Geopolymer Conference, Melbourne, Australia, 28–29 October 2002; pp. 1–9.
18. Thao, A.; Magee, B.; Woodward, D. A preliminary characterisation of innovative semi-flexible composite pavement comprising geopolymer grout and reclaimed asphalt planings. *Materials* **2020**, *13*, 3644. [[CrossRef](#)]
19. Hamid Abed, M.; Sabbar Abbas, I.; Hamed, M.; Canakci, H. Rheological, fresh, and mechanical properties of mechanochemically activated geopolymer grout: A comparative study with conventionally activated geopolymer grout. *Constr. Build. Mater.* **2022**, *322*, 126338. [[CrossRef](#)]
20. Afonso, M.L.; Dinis-Almeida, M.; Pereira-De-Oliveira, L.A.; Castro-Gomes, J.; Zoorob, S.E. Development of a semi-flexible heavy duty pavement surfacing incorporating recycled and waste aggregates—Preliminary study. *Constr. Build. Mater.* **2016**, *102*, 155–161. [[CrossRef](#)]
21. Zhang, Y.; Wang, Y.; Wu, Z.G.; Lu, Y.M.; Kang, A.H.; Xiao, P. Optimal design of geopolymer grouting material for semi-flexible pavement based on response surface methodology. *Constr. Build. Mater.* **2021**, *306*, 124779. [[CrossRef](#)]
22. Chen, M.; Lin, J.; Wu, S. Potential of recycled fine aggregates powder as filler in asphalt mixture. *Constr. Build. Mater.* **2011**, *25*, 3909–3914. [[CrossRef](#)]
23. Wang, H.; Al-Qadi, I.L.; Faheem, A.F.; Bahia, H.U.; Yang, S.-H.; Reinke, G.H. Effect of Mineral Filler Characteristics on Asphalt Mastic and Mixture Rutting Potential. *Transp. Res. Rec.* **2011**, *2208*, 33–39. [[CrossRef](#)]
24. Huang, B.; Shu, X.; Chen, X. Effects of mineral fillers on hot-mix asphalt laboratory-measured properties. *Int. J. Pavement Eng.* **2007**, *8*, 1–9. [[CrossRef](#)]
25. Diab, A.; Enieb, M. Investigating influence of mineral filler at asphalt mixture and mastic scales. *Int. J. Pavement Res. Technol.* **2018**, *11*, 213–224. [[CrossRef](#)]
26. Sakanlou, F.; Shirmohammadi, H.; Hamedi, G.H. Investigating the effect of filler types on thermodynamic parameters and their relationship with moisture sensitivity of asphalt mixes. *Mater. Struct. Constr.* **2018**, *51*, 39. [[CrossRef](#)]
27. Choudhary, J.; Kumar, B.; Gupta, A. Utilization of solid waste materials as alternative fillers in asphalt mixes: A review. *Constr. Build. Mater.* **2020**, *234*, 117271. [[CrossRef](#)]
28. Wozzuk, A.; Bandura, L.; Franus, W. Fly ash as low cost and environmentally friendly filler and its effect on the properties of mix asphalt. *J. Clean. Prod.* **2019**, *235*, 493–502. [[CrossRef](#)]
29. Tarbay, E.W.; Azam, A.M.; El-Badawy, S.M. Waste materials and by-products as mineral fillers in asphalt mixtures. *Innov. Infrastruct. Solut.* **2018**, *4*, 5. [[CrossRef](#)]
30. Awed, A.M.; Tarbay, E.W.; El-Badawy, S.M.; Azam, A.M. Performance characteristics of asphalt mixtures with industrial waste/by-product materials as mineral fillers under static and cyclic loading. *Road Mater. Pavement Des.* **2022**, *23*, 335–357. [[CrossRef](#)]
31. Nilton de Souza Campelo; Arlene Maria Lamêgo da Silva Campos; Aroldo Figueiredo Aragão Utilization of Synthetic Coarse Aggregate of Calcined Clay in Asphalt Mixtures in the Amazon Region. *J. Geol. Resour. Eng.* **2018**, *6*, 321–324. [[CrossRef](#)]

32. Solouki, A.; Tataranni, P.; Sangiorgi, C. Mixture Optimization of Concrete Paving Blocks Containing Waste Silt. *Sustainability* **2022**, *14*, 451. [[CrossRef](#)]
33. Solouki, A.; Fathollahi, A.; Viscomi, G.; Tataranni, P.; Valdrè, G.; Coupe, S.J.; Sangiorgi, C. Thermally treated waste silt as filler in geopolymer cement. *Materials* **2021**, *14*, 5102. [[CrossRef](#)]
34. Chen, M.; Lin, J.; Wu, S.; Liu, C. Utilization of recycled brick powder as alternative filler in asphalt mixture. *Constr. Build. Mater.* **2011**, *25*, 1532–1536. [[CrossRef](#)]
35. Cheng, Y.; Tao, J.; Jiao, Y.; Tan, G.; Guo, Q.; Wang, S.; Ni, P. Influence of the properties of filler on high and medium temperature performances of asphalt mastic. *Constr. Build. Mater.* **2016**, *118*, 268–275. [[CrossRef](#)]
36. Hanein, T.; Thienel, K.-C.; Zunino, F.; Marsh, A.T.M.; Maier, M.; Wang, B.; Canut, M.; Juenger, M.C.G.; Ben Haha, M.; Avet, F.; et al. Clay calcination technology: State-of-the-art review by the RILEM TC 282-CCL. *Mater. Struct.* **2021**, *55*, 3. [[CrossRef](#)]
37. Tataranni, P.; Sangiorgi, C. Synthetic Aggregates for the Production of Innovative Low Impact Porous Layers for Urban Pavements. *Infrastructures* **2019**, *4*, 48. [[CrossRef](#)]
38. Chen, Y.; Xu, S.; Tebaldi, G.; Romeo, E.; Chen, Y.; Xu, S.; Tebaldi, G.; Romeo, E. Role of mineral filler in asphalt mixture. *Road Mater. Pavement Des.* **2020**, *23*, 247–286. [[CrossRef](#)]
39. Modarres, A.; Alinia Bengar, P. Investigating the indirect tensile stiffness, toughness and fatigue life of hot mix asphalt containing copper slag powder. *Int. J. Pavement Eng.* **2019**, *20*, 977–985. [[CrossRef](#)]
40. Choudhary, J.; Kumar, B.; Gupta, A. Evaluation of engineering, economic and environmental suitability of waste filler incorporated asphalt mixes and pavements. *Road Mater. Pavement Des.* **2021**, *22*, S624–S640. [[CrossRef](#)]
41. Marsh, A.T.M.; Velenturf, A.P.M.; Bernal, S.A. Circular Economy strategies for concrete: Implementation and integration. *J. Clean. Prod.* **2022**, *362*, 132486. [[CrossRef](#)]
42. Inocente, J.M.; Elyseu, F.; Nieves, L.J.J.; Jiusti, J.; Cargnin, M.; Peterson, M. Applied Clay Science Production and characterization of high-reactivity metakaolins calcined in flash reactor. *Appl. Clay Sci.* **2021**, *213*, 106247. [[CrossRef](#)]
43. FLsmidth Revealing the Numbers behind Calcined Clay. Available online: <https://www.flsmidth.com/en-gb/discover/cement-2021/revealing-the-numbers-behind-calcined-clay> (accessed on 11 July 2022).