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Abstract: Concerns about the environment, the cost of energy, and safety mean that low-energy cold-mix asphalt materials are very interesting as a potential replacement for present-day hot mix asphalt. The main disadvantage of cold bituminous emulsion mixtures is their poor early life strength, meaning they require a long time to achieve mature strength. This research work aims to study the protentional utilization of waste and by-product materials as a filler in cold emulsion mixtures with mechanical properties comparable to those of traditional hot mix asphalt. Accordingly, cold mix asphalt was prepared to utilize paper sludge ash (PSA) and cement kiln dust (CKD) as a substitution for conventional mineral filler with percentages ranging from 0–6% and 0–4%, respectively. Test results have shown that the incorporation of such waste materials reflected a significant improvement in the mixture's stiffness and strength evolution. The cementitious reactivity of PSA produces bonding inside the mixtures, while CKD is used as an additive to activate the hydration process of PSA. Therefore, based on the results, it will be easier to build cold mixtures by shortening the amount of time needed to reach full curing conditions.

Keywords: cold mix asphalt; bitumen emulsion; paper sludge ash (PSA); cement kiln dust (CKD); stiffness modulus; wheel track

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Citation: Dulaimi, A.; Al-Busaltan, S.; Kadhim, M.A.; Al-Khafaji, R.; Sadique, M.; Al Nageim, H.; Ibrahem, R.K.; Awrejcewicz, J.; Pawłowski, W.; Mahdi, J.M. A Sustainable Cold Mix Asphalt Mixture Comprising Paper Sludge Ash and Cement Kiln Dust. *Sustainability* **2022**, *14*, 10253. https://doi.org/10.3390/su141610253

Academic Editors: Ramadhansyah Putra Jaya, Khairil Azman Masri and Zaid Hazim Al-Saffar

Received: 19 July 2022
Accepted: 13 August 2022
Published: 18 August 2022

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1. Introduction

Due to the significant air-void content of cold mix asphalt, inadequate early life strength, and extensive curing time span durations required to achieve the desired level of performance, cold mix asphalt has been regarded inferior to hot mix asphalt [1–4]. Such bituminous mixtures can be prepared, mixed, and laid down at ambient temperature without the use of any heating methods. Many countries, such as the United States and France, utilized cold bituminous emulsion mixture (CBEM) for paving since the 1970s and seem to have extensive information about the performance of these mixtures. On the other side, this technology has just lately emerged in the UK because of weather changes, i.e., CBEM requires a long time to achieve the required strength in cold and wet climates [5]. Permanent cold lay surfacing materials (PCSMs) received their first accreditation in 1996, and Phillips [6] reported on the performance experiments on one of these materials.

The relatively high application temperature for heating bitumen and aggregate results in significant fuel consumption, which has a negative impact on the environment during the production of hot mix asphalt (HMA) [7–9]. Furthermore, the mixing process generates harmful gases that are extremely harmful to the human body and the environment [10–12]. Thus, there seems to be an increasing trend worldwide toward the usage of cold bitumen emulsion mixes for a variety of reasons [13], including the following: (i) the elimination of heating for both the binder and aggregate during mixture manufacture, (ii) contribution to environmental protection, and (iii) energy conservation. Nonetheless, such mixtures reported poorer mechanical characteristics, including low early strength, a lengthy curing period, and a significant air void content as compared to the HMA [14–18].

The aforementioned deficiencies have been addressed by substituting ordinary Portland cement (OPC) for the filler of CBEMs [2,19,20]. According to Li et al. [21], the cement-asphalt emulsion mixes have a greater toughness, a longer fatigue life, and a lower temperature susceptibility. It was proved that adding cement to cement asphalt emulsion mixes changes the pH, which causes the emulsion to break down quickly [22]. However, OPC is not a green product and is considered to be an expensive product. As a consequence, a variety of by-product materials produced industrially were chosen based on their preliminary characteristics, availability in the UK and globally, and waste status in terms of the pozzolanic activity, to assess the potential suitability to replace OPC collectively or individually. It is crucial to look for resources that may offer a sustainable element by lowering resource consumption, relieving landfill pressure, and keeping structural integrity. Recently, waste recycling brings more interest, which has a huge advancement in the construction area [23].

The laying and compaction temperatures are what differentiate Hot Mix Asphalt, Warm Mix Asphalt, Half-Warm Mix Asphalt, and Cold Mix Asphalt. The following temperatures are used to manufacture and lay these mixtures: 150–190 °C, 100–140 °C, 60–100 °C, and 0–40 °C, respectively [13,24,25]. However, there is some debate over the maximum limits of CMA. The term "CMA" may have been used to describe the production of bituminous mixes at temperatures up to 60 °C, according to some studies [26,27]. It is noteworthy that the energy required to produce CMA is roughly 95% lower than that required to produce hot and warm mix asphalt [13]. In recent years, several pieces of research have focused on such types of asphalt mixtures (CBEM) to overcome their weakness and obtain desirable strength comparable to the HMA in a short time of curing. Numerous researchers had reported that wastes that offer cementitious activity can be utilized in CBEM since they require a significant amount of water to lubricate the aggregates. They stated that this trapped water can be minimized by the hydraulic activity fillers and contribute to improving mixture efficiency. However, some waste fillers lack some chemical components that are necessary to complete the hydration process and require an activation agent to enhance filler activity [4,28,29].

Due to their filler and pozzolanic effects, fly ash and ground granulated blast furnace slag, two regularly used active mineral admixtures, have demonstrated considerable potential to improve the strength of cold mix asphalt [4,30]. Shalan et al. [31] conducted a study on a variety of storage-grade macadams produced from mixing various aggregate resources with bitumen emulsion and ground granulated blast furnace slag (GGBS). The results indicated that when GGBS is introduced in high humidity circumstances, stiffness and strength can emerge. Lu, Wang, Leng, and Zhong [4] revealed in their findings that the indirect tensile strength of cold mix asphalt comprising 2 percent OPC, 1 percent fly ash, and 1 percent GGBS is roughly 20% higher than that of the mixture containing only 2 percent OPC.

Al Nageim, Al-Busaltan, Atherton, and Sharples [20] conducted research to develop new CBEMs by incorporating fly ash and industrial by-products to observe the level of enhancement compared to those of conventional cold mix with OPC and two-grade HMA. They carried out experiments to demonstrate the improvement in the mechanical characteristics of CBEMs using OPC and to evaluate the possibility of utilizing paper sludge ash instead of OPC. The results indicated that with such modifications, the mechanical characteristics were comparable to HMA.

Utilizing waste materials reduces energy consumption, protects non-renewable natural resources, and reduces the quantity of material that causes environmental pollution. The recycling of waste papers process generates waste ash that may be used in paving roads due to its cementitious characteristics [32–35]. Waste paper sludge ash has recently received attention in construction applications due to its beneficial cementitious characteristics [36,37]. PSA can be strongly used as a cementitious filler instead of OPC if an activation agent is utilized with it to produce cold mixtures with superior or at least compatible properties to HMA [1,37].

Waste PSA consists of active elements like free lime (CaO) and inert mineral types. Sludge from paper mills is a by-product of the pulp and paper industries. In Europe, around 11 M tonnes of PS waste are generated each year, whereas the United Kingdom produces 1 M tonnes of such waste. The amount and kind of the produced waste PSA are determined by the raw materials (virgin or recycled paper), the manufacturing method, and the output aim. Recently, the burning of PSA for energy recovery has grown increasingly appealing as a result of environmental regulations and rising pricing on landfills, even though PSA is classified as trash in the United Kingdom.

On the other hand, ordinary Portland cement is considered to be one of the most important product lines in the construction field. A substantial amount of cement kiln dust (CKD) is generated as by-product waste during cement production. It is also referred to as by-pass dust composed of micron-sized particles collected from electrostatic precipitators during the clinker manufacturing process at high-temperature conditions. It is used as a filler material in asphalt paving mixes as an anti-stripping agent, for soil improvement, as well as in cement mortar [38]. The alkalinity of CKD and water combinations is often around 12, indicating a significant level of alkalinity. Thus, the high concentration of alkali and sulfate provides an effective activation of pozzolanic materials. The clinker's chemical components are determined by the raw materials used to prepare, as well as the kind and source of carbon-based fuel used to raise the materials' temperature in the rotary kiln. One of the most frequently used valuable applications of CKD areas is soil stabilization agent as a substitute for lime, waste treatment for waste stabilization and densification, supplementary cementitious material, asphalt pavement as a mineral filler in asphalt concrete mixes, and other applications [38,39].

Many researchers had focused their research on utilizing CKD in cold mix asphalt mixtures. Al-Merzah et al. [40] investigated the possibility of incorporating CKD as a partial replacement filler instead of the conventional limestone mineral filler and ordinary Portland cement to develop the performance of a sustainably cold mixture for binder course. They reported that incorporating 25% of CKD with 75% OPC can achieve a mixture strength improvement in terms of resistance to plastic deformation, rutting, and cracking resistance. Abdel-Wahed, Dulaimi, Shanbara, and Al Nageim [2] reported that the combination of OPC and CKD as a filler in CBEM mixtures can improve mixture performance significantly in terms of stiffness modulus, fatigue life, and resistance to rutting deformation.

This study is predicted to provide a wide range of advantages, which may be divided into two categories: current and future outcomes. The current benefit can be accomplished using waste and by-product materials in road construction (PSA and CKD fillers). This has gained widespread acceptance and is becoming a more important aspect nowadays, while the future benefits result in a reduction of adverse environmental impacts of the waste materials. In addition, the economic factor is represented by cost reduction through using waste materials.

There are currently only a few pieces of research that address the production of cold bituminous emulsion mixture using paper sludge ash and cement kiln dust as alternatives to traditional mineral filler. This research aims to produce a cold bituminous emulsion mixture that overcomes the shortcomings of emulsion-based mixes, notably the stiffness modulus, rutting, and fatigue. To accomplish this, a sustainable approach was used, beginning with the substitution of the two waste and by-product materials for the mineral filler. This was followed by the evaluation of the mechanical properties and a comparison to the reference CBEM and the two conventional hot mix asphalt types. Such CBEM is an environmentally friendly method of producing bituminous mixtures.

2. Materials Properties and Testing Method

2.1. Materials

The materials used in this research work are briefly presented as follows:

(a) Aggregate

The crushed granite type was utilized for both coarse and fine aggregates, which are typically used to prepare asphalt concrete. Their physical characteristics are shown in Table 1. AC 20 aggregate gradation was adopted in this study, which is a commonly used type for binder layer purposes, as seen in Figure 1. It is a dense bitumen macadam mixture that is the most often used binder course and base in road pavements in the United Kingdom. It is constantly graded and derives its strength from the interlocking of coated aggregates, which is the primary mechanism by which the material transmits weight [41]. AC is a continuously graded combination with a high degree of aggregate interlock. As a result, this material has excellent load spreading characteristics and strong resistance to permanent deformation. Moreover, the used aggregates in this study followed the BS EN 13108-1 [42].

Table 1. Aggregate physical properties.

Material	Property	Value
Limestone filler	PD, Mg/m^3	2.57
Fine aggregate	BPD, Mg/m^3	2.54
	APD, Mg/m^3	2.65
	WA, %	1.7
Coarse aggregate	BPD, Mg/m^3	2.62
	APD, Mg/m^3	2.67
	WA, %	0.8

PD = Particle density, BPD = Bulk particle density, APD = Apparent particle density, WA = Water absorption.

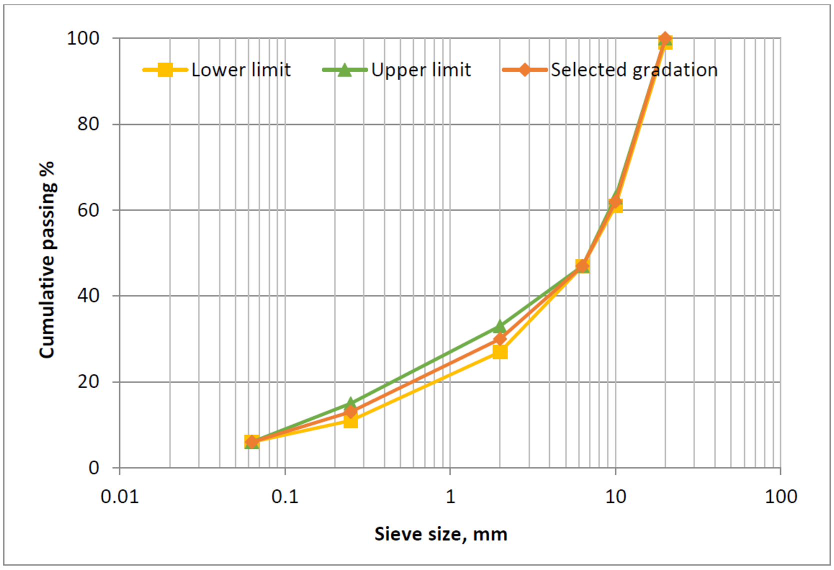

Figure 1. Aggregate gradation of dense binder course type (AC 20 mm).

(b) Bitumen emulsion and bitumen

All CBEM mixtures were prepared using a cationic slow-setting bitumen emulsion (C 60 B 5). Nikolaids [43] proved that cationic emulsions are beneficial because of their capacity to cover the provided mixed aggregates sufficiently and maintain proper particle adherence. The characteristics of the selected bitumen emulsion are listed in Table 2. Additionally, two grades of bitumen types (131 pen and 49 pen) were employed to prepare HMA; their characteristics are listed in Table 3. The two binders for HMA are chosen as additional references, representing the traditional binder band for HMA and the performance comparison.

Table 2. Properties of (C 60 B 5) bitumen emulsion.

Description	(C 60 B 5) Bitumen Emulsion
Appearance	Black to dark brown liquid
Type	Cationic
Bitumen content	60%
Base bitumen	100/150 pen
Base bitumen, 1/10 mm	147 pen
Softening point, °C	40.5
Relative density at 15 °C, g/mL	1.05
Boiling point, °C	100

Table 3. Properties of 131 pen and 49 pen bitumen binders.

Bituminous Binder 49		Bituminous Binder 131	
Property	Value	Property	Value
Appearance	Black	Appearance	Black
Density at 25 °C	1.02	Density at 25 °C	1.05
Softening point, °C	51.5	Softening point, °C	43.5
Penetration at 25 °C	49	Penetration at 25 °C	131

(c) Fillers

This study used three types of filler: conventional limestone mineral filler (CLMF), paper sludge ash (PSA), and cement kiln dust (CKD), which was used as an activator in different percentages ranging from 0.5% to 4% of the aggregate's total weight.

It is worth mentioning that the PSA samples were collected from Aylesford Newsprint Mill Limited Ltd. (ANL), which is Europe's largest recycled newsprint mill using recycled recovered magazines and newspapers. PSA results from the incineration of PSA and other input materials throughout the recycling of newspapers and similar products. The waste fly ash produced from this activity has been collected for the analysis of its properties. The CKD for this study was supplied by CEMEX from the Rugby cement plant, which comes from the production of cement clinker. They produce approximately 25 Kt per year of bypass dust at the Rugby Cement plant.

2.2. Physical and Chemical Properties of Fillers

PSA is a type of industrial waste generated during the burning of useless paper in a power plant. CKD is produced during the cement manufacturing process. The X-ray powder diffraction (XRPD) spectra of dry samples are shown in Figures 2 and 3. The mineralogical configuration of PSA in XRD indicates that the particle diffraction peaks are crystalline since it has distinct peaks with little background noise. The detected principal crystal peaks were lime (CaO), calcite ($CaCO_3$), mayenite ($Ca_{12}Al_{14}O_{33}$), merwinite ($Ca_3Mg[SiO_4]_2$), and gehlenite ($Ca_2Al_2SiO_7$).

The powder XRD of CKD discovered the mineralogy mainly composed of lime (CaO) as a major constituent, and a small amount of quartz together with calcite ($CaCO_3$), anhydrite (CaSO4), Sylvite (KCl), and Portlandite ($Ca(OH)_2$). The concentration of K_2O and the linked Sylvite was prominent and was expected to play a vital role, showing chemical properties largely varying from conventional coal burning fly ash and PSA. Sylvite was predicted to behave similarly to arcanite, creating an ambient environment for breaking the glass phase of PSA particles, additionally acting as a motivator during the early phases of hydration [37]. The existence of sulfates from potassium sulphates was predicted to promote the dissolution of PSA by decreasing the amount of Ca^{+2} and Al^{+3} in the mixture, forming ettringite and hence enhancing the system solidity [44].

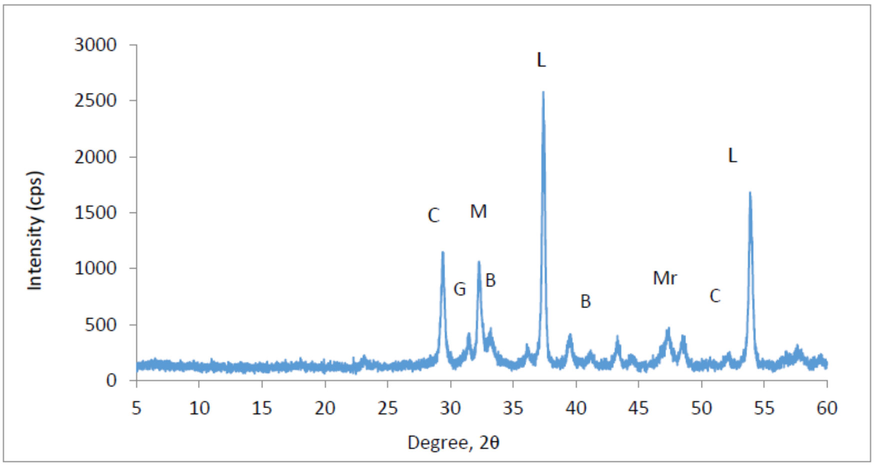

Figure 2. Powder XRD pattern of PSA (merwinite-Mr, lime-L, gehlenite-G, calcite-C, Belite-B, mayenite-M).

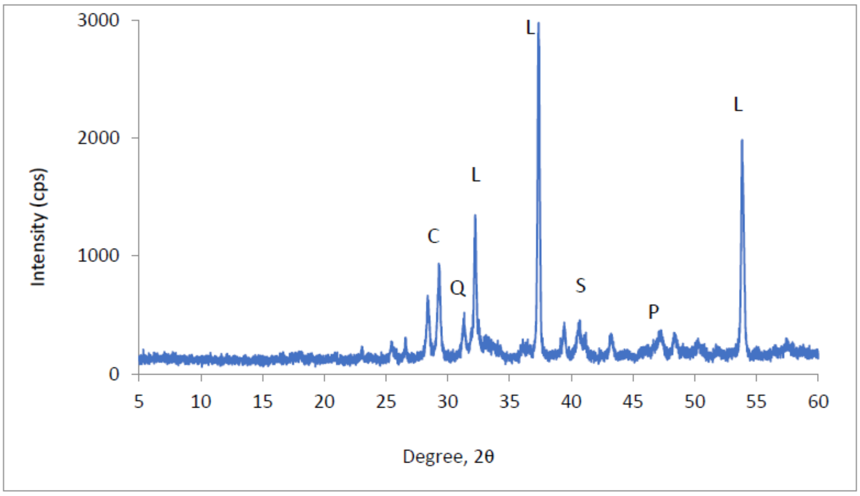

Figure 3. Powder XRD pattern of CKD. (Lime-L, calcite-C, quartz-Q, Sylvite-S, Portlandite-P).

Figure 4 illustrates the SEM micrographs of the two mentioned waste materials in addition to the CLMF. The morphology of the filler has a significant effect on the emulsion breaking, where the agglomerated morphology provides paths for water suction. The morphology of the SEM micrograph reveals the agglomerated and non-spherical-shaped particles for PSA. The CLMF particles appear as irregular shapes with sharp angles in contrast to the CKD particles, which have a range of angular shapes. Table 4 presents the chemical composition of used fillers determined by EDXRF analysis. The major oxides found in EDXRF are Ca, Si, K_2, and SO_3.

Table 4. EDXRF analysis of the selected filler materials, %.

Filer Type	CaO	SiO_2	Al_2O_3	MgO	Fe_2O_3	SO_3	K_2O	Na_2O
PSA	70.276	24.671	2.209	2.721	0	0.342	0.335	1.811
CKD	59.319	20.192	0.882	1.287	1.551	3.852	4.88	2.177
CLMF	76.36	16.703	0	0.981	0	0.096	0.348	2.258

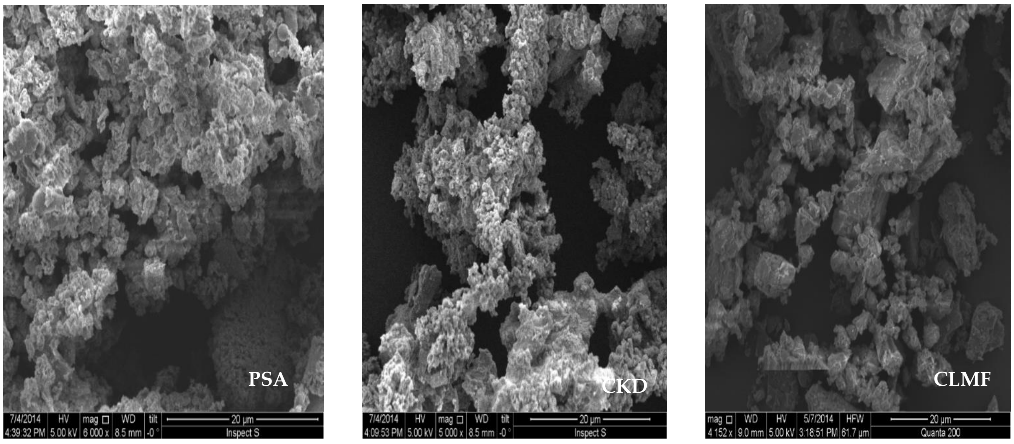

Figure 4. SEM view of filler particles.

2.3. Sample Preparation and Conditioning

The primary stage of this study was to construct a cold mix asphalt by substituting PSA for the mineral filler and activating it with various amounts of CKD. The selected aggregate gradation was for the binder course to prepare the CBEM, because it is the most frequently used type of bitumen-bound material road base of trunk roads (including motorways) in the United Kingdom, due to its excellent load-spread properties and resistance to deformation. This was accomplished by substituting PSA with the conventional mineral filler in CBEM at five different replacement percentage ratios (0%, 1.5%, 3%, 4.5%, and 6% by aggregate weight). Moreover, CKD was incorporated as an activation agent with percentages of 0.5%, 1%, 2%, and 4% by the total weight of the aggregates.

Currently, there is no globally approved design mixture for CBEMs. Certain authorities and scholars have proposed many mixed design approaches for CBEMs [14,32,45]. The design approach was based on the Asphalt Institute's method for constructing CBEM (Marshall Method for Emulsified Asphalt Aggregate Cold Mixture Design (MS-14) [46]. Compaction was accomplished by hammering the diametrical specimens each face 50 times with a Marshall hammer.

The Indirect Tensile Stiffness Modulus test (ITSM) was used to determine the effect of substituting PSA with CLMF. Additionally, the findings were compared to those obtained using conventional HMA specimens. Serfass et al. [47] revealed that cold mixes demonstrated evolutionary characteristics, particularly throughout their early life, when cohesiveness is low at first and subsequently increases. Therefore, two phases of curing were required prior to evaluating the samples. The first stage was completed after one day at 20 °C with the specimens remaining in the mold, and the specimens were ejected the next day to prevent disintegration. Following that, the second stage was completed by placing the specimens in a ventilated oven for 24 h at a temperature of 40 °C. Following these phases, all samples were stored at 20 °C and examined at various ages, namely 2, 7, 14, and 28 days. This curing technique reflects 7–14 days in the field according to Jenkins [48]. On the other hand, two grades of AC 20 hot mix were prepared and kept at a constant temperature of 25 °C and evaluated at comparable ages of CBEM. It is worth mentioning that the CBEM specimens were prepared and compacted at ambient temperatures, whilst the preparation temperatures of the HMA specimens were maintained at (150–160 °C) and (160–170 °C), respectively.

2.4. Methods

2.4.1. Indirect Tensile Stiffness Modulus (ITSM)

The ITSM was adopted as a primary test to evaluate the mixtures' performance. The test was carried out in compliance with British Standard EN 12697-26 [49]. Cooper research technology HYD 25 testing equipment was used for such a purpose. The followed conditions of ITSM testing are listed in Table 5.

Table 5. ITSM Test Conditions.

Item	Range
Specimen temperature conditioning	4 h before testing
Rise time	124 ± 4 ms
Specimen thickness mm	63 ± 3
Transient peak horizontal deformation	5 µm
Specimen diameter mm	100 ± 3
Poisson's ratio	0.35
Loading time	3–300 s
No. of conditioning plus	5
No. of test plus	5
Test temperature °C	20 ± 0.5
Compaction Marshall	50 × 2

2.4.2. Resistance to Permanent Deformation

The permanent deformation of asphalt pavement is a major disease type, and asphalt's resistance to permanent deformation is one of the key elements influencing the performance of asphalt pavement [50], and as a result, resistance to permanent deformation has become one of the most significant essential attributes to evaluate. As a result of accumulated strain caused by increased tire pressure, axle loads, and loading time, permanent deformation occurs in the pavement layer, putting greater stress on the asphalt surfacing layers closest to the tire-pavement contact area. The materials' characteristics, temperature, load level, and loading period all influence the extent of pavement deformation.

The permanent deformation for cold mixes was evaluated according to BS EN 12697-22 [51]. This test was performed for all said mixes created with CLMF and mixes created with PAS and CKD. All of the findings were compared to the two control HMAs' rutting resistance. Each mixture was tested at 45 °C for 460 min at 10,000 load cycles. This temperature was chosen based on PD 6691 [52], which states that 45 °C represents moderate to moderately strained locations that require high rut resistance. The test was carried out on a solid slab with dimensions of 400 mm in length, 305 mm in width, and 50 mm in thickness, which was created by compacting a loose bitumen mixture with a roller compactor at room temperature.

To obtain cured conditions, the samples were left in their molds for 24 h at ambient temperature before being cured for 14 days at 40 °C in a ventilated oven [53]. Each bituminous slab sample was conditioned at test temperature for at least 4 h prior to testing, after which a single wheel with a typical vehicle tire pressure of 0.7 MPa was applied to the sample with a forward and backward motion at a frequency of 0.8 Hz. An LVDT was used to track the progress of rutting in the sample over time by tracking the vertical position of the wheel.

2.4.3. Resistance to Fatigue (Four-Point Bending Test)

Fatigue cracking in asphalt mixes is most common when the tensile strain is between 30 and 200 microstrain, according to [53]. According to Brown and Needham [54], the strain levels in a pavement construction are expected to be below 200 microstrain, with the exact amount depending on numerous variables such as the layer thickness, subgrade, type of mixture, and load.

Fatigue cracking has long been thought to be one of the most predominant distresses affecting the serviceability of asphalt pavements. The fatigue properties of all the CBEM and HMA mixes in this study were assessed using a four-point bending test in a controlled strain mode. In this test, fatigue life was defined as the number of cycles applied until the stiffness modulus reached 50% of its initial value. The test includes using two load points to provide a continuous sinusoidal waveform on the top of a prismatic sample (inner clamps). Cutting slabs of cold asphalt mixes and both hot asphalt concrete mixes yielded prismatic samples. All the slabs were formed, compacted, and cured using Thanaya's method [53].

Before testing, each slab was sliced into five samples with dimensions of 400 mm long, 50 mm wide, and 50 mm thick.

3. Results and Discussion

3.1. Influence of Replacing the Traditional Mineral Filler with PSA

Figure 5 displays the ITSM findings for the CBEM which included ranges of PSA; these ranges vary from 0% (6% CLMF) to 6% of the total aggregate weight. In addition, the same figure comprises the findings of the two hot mix asphalts. The results of ITSM demonstrate significant impacts, where a substantial increase for the stiffness modulus with increasing PSA replacement ratio can be observed, and it achieved the ultimate value by replacing all the limestone filler with PSA throughout time. On the other hand, both HMA mixtures showed no significant differences in performance. It can be seen that when the percentage of PSA in CBEM increases, the rate of stiffness enhancement increases proportionally at early ages. The CBEMs comprised of PSA filler reflected an improved value of ITSM with time, particularly at higher percentages of PSA incorporation in the contract of the conventional CBEM that included lime as filler, which reflected low variation in the stiffness with time.

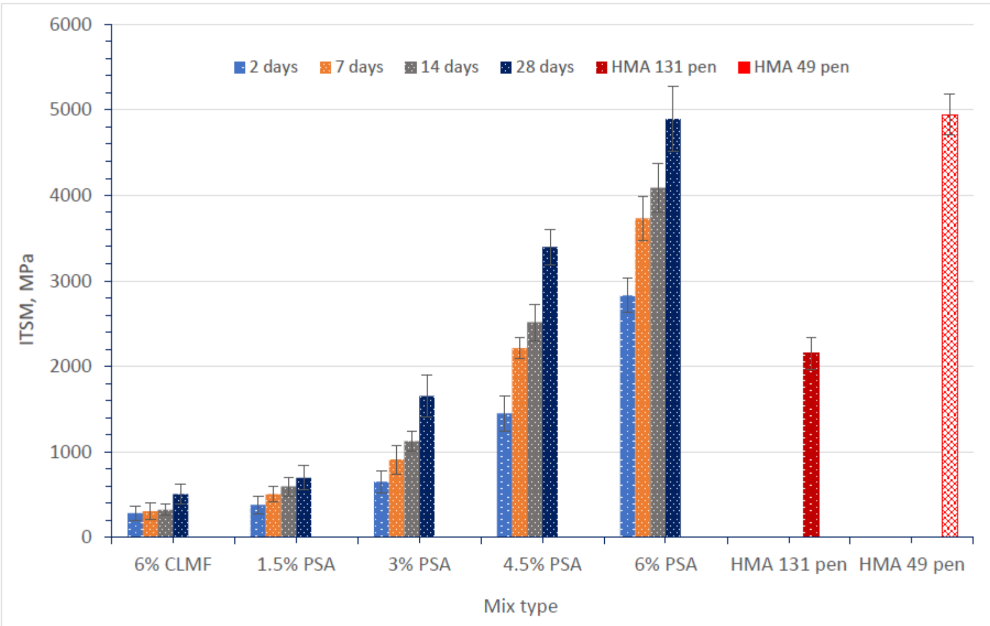

Figure 5. Influence of PSA percentage on ITSM results.

The explanations for this improvement in the stiffness modulus are due to the formation of a new binder in addition to the residual binder generated during the hydraulic reaction of the PSA. In addition, the hydration absorbed the trapped water, which contributed greatly to the mixture's fragility. Furthermore, the addition of PSA to CBEM resulted in a significant increase in the stiffness modulus. This improvement is due to the features of PSA. Its cementitious properties, which include a high-water absorption rate and a high pH, contribute significantly to the emulsion breaking process by forming a strong link between the internal microstructure and participating in the emulsion's braking. These results are in agreement with the findings of Lu, Wang, Leng, and Zhong [4] and Al-Busaltan et al. [1].

The important results from incorporating PSA in the CBEM provided a prompt to discover the full power of PSA by an appropriate activator. Nevertheless, by-product fine materials were found to be very relevant in PSA activating. Figure 6 displays the ITSM findings of cold mix asphalt containing 6% of PSA with different percentages of the CKD activator. CKD was used as an additive in different percentages (0.5%, 1%, 2%, and 4% by total aggregate mass).

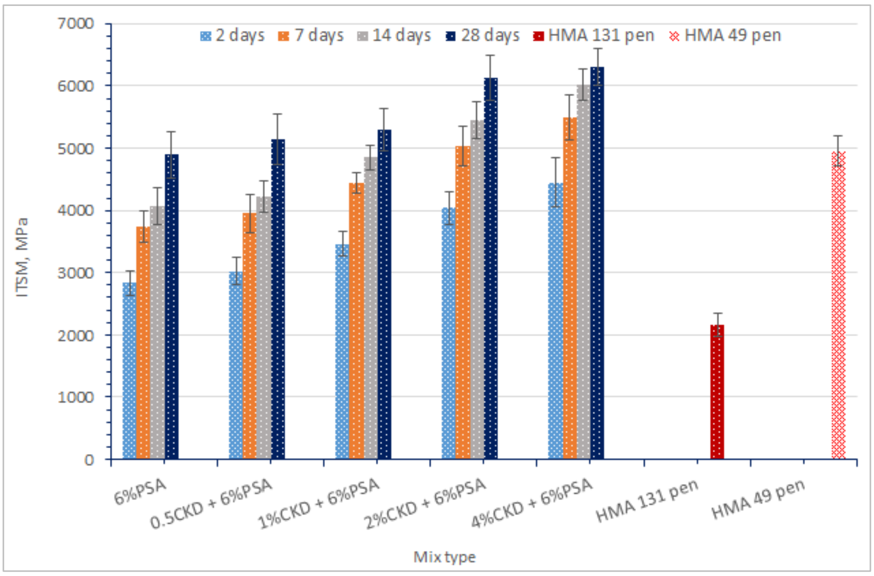

Figure 6. Effect of CKD addition on ITSM with PSA.

Furthermore, a comprehensive comparison has been considered between the conventional hot AC 20 binder course and cold asphalt concrete. The experimental data show a good stiffness value of the cold mix asphalt when using PSA and CKD. Three specimens were produced to ensure the reliability of the results.

It is obviously shown from these Figures that the CKD acts to activate the PSA. The high potassium content of CKD improves the hydration process, accelerating hydration. It can be observed that the addition of CKD increased the degree of hydration; therefore, the primary objective of the CKD to activate the PSA hydration process was achieved. Adding 2% CKD to the CBEM incorporated 6% PSA increased the mix stiffness by about 43% after 2 days, exceeding the desired value for both HMA mixes. Additionally, the HMA mixes' stiffness can be attained after seven days of the curing of CBEM including 2% CKD and 6% PSA.

3.2. Wheel Track Test Results at 45 °C

The results of the wheel track test are displayed against the test period in Figure 7, which shows the rutting depth. The CBEM with CLMF has a deeper rut than the other combinations, causing the slab sample to deteriorate. After 10,000 cycles, the rut depths for CBEM with CLMF, 6% PSA, 6% PSA + 0.5% CKD, 6% PSA + 1% CKD, 6% PSA + 2% CKD, 6% PSA + 4% CKD, HMA 131 pen, and HMA 49 pen were 11.8 mm, 0.862 mm, 0.783 mm, 0.701 mm, 0.632 mm, 0.497 mm, 3.349 mm and 2.666 mm, respectively. CLMF's poor effectiveness against rutting means that it is unsuitable for use as a pavement mixture. In comparison to conventional CLMF and both HMA control mixtures, replacing CLMF with PSA in the traditional CBEM resulted in a considerable reduction in rut depth. The PSA reduced the accumulated rutting depth by around 14 times when compared to CLMF. The rutting depth was reduced by roughly 15, 17, and 19 times when CLMF was replaced with 6% PSA + 0.5% CKD, 6% PSA + 1% CKD, 6% PSA + 2% CKD in CBEM. This increase in rutting resistance highlights the benefits of including PSA in CBEM in terms of the hydration process. When CLMF is replaced with 6% PSA + 4% CKD, the rutting depth is reduced even further. When compared to the rutting findings of CBEM-CLMF, CBEM with 6% PSA + 4% CKD showed an increase in resistance to permanent deformation of roughly 24 times. This is owing to the alkali media provided by the CKD, which enhances the cementitious filler's hydration process and results in a dense microstructure.

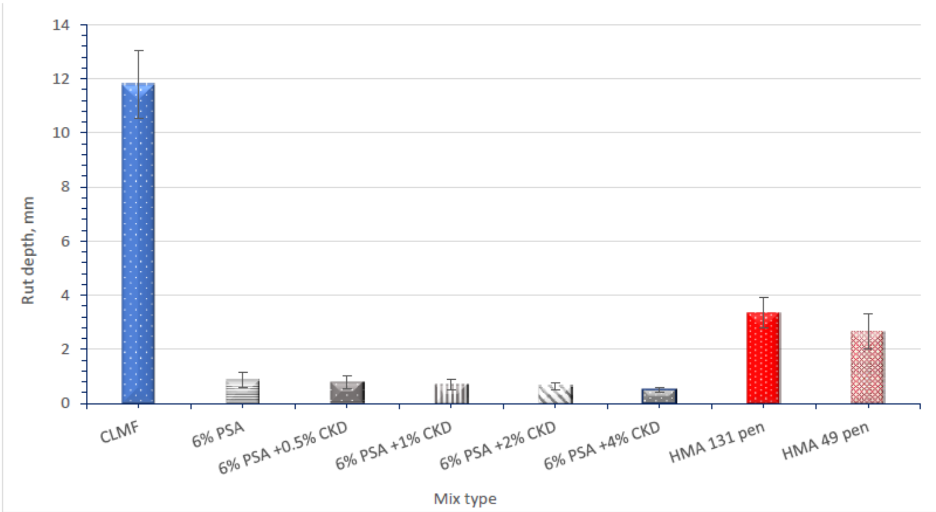

Figure 7. Wheel track results.

3.3. Resistance to Fatigue Results

Under microstrains of 150 at 20 °C and a frequency of 10 Hz, the test was conducted in a strain control mode. Figure 8 shows the fatigue life (Nf) for all mixes based on 150 microstrain. When compared to other mixes, CBEM with CLMF has the lowest fatigue life (low fatigue resistance), with its poor resistance indicated by low stiffness.

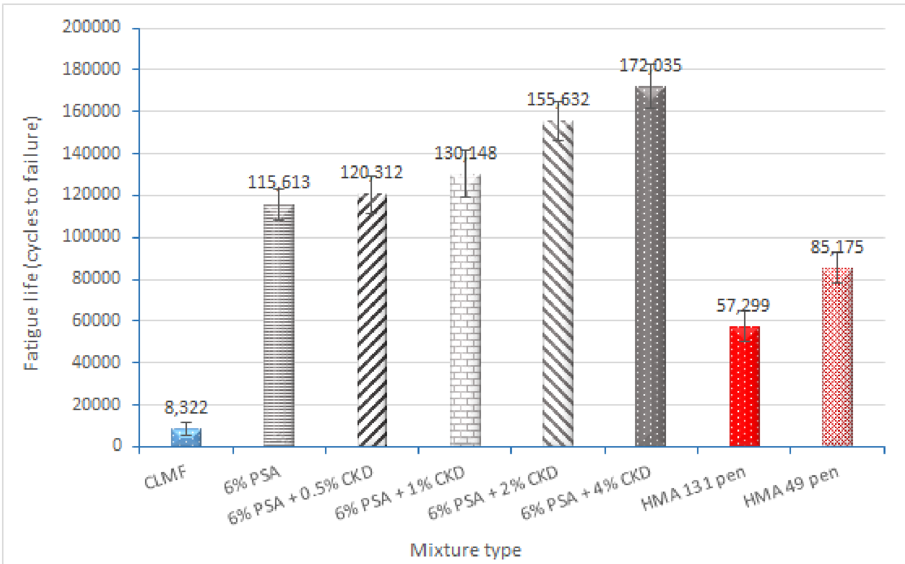

Figure 8. Fatigue performance based on 150 microstrain.

When 6% PSA was substituted for the CLMF in the mix, fatigue resistance increased by 14 times. When compared to traditional cold and hot mixes, the fatigue life of CBEM treated with 6% PSA increased significantly. There was no significant improvement when CLMF was replaced by 6% PSA + 0.5% CKD. However, when CLMF is replaced by 6% PSA + 1% CKD, 6% PSA + 2% CKD, and 6% PSA + 4% CKD, the fatigue life increased by 16, 19, and 21 times compared to mixtures containing CLMF. In addition, 6% PSA + 2% CKD and 6% PSA + 4% CKD mixes improved the fatigue life by around three times in comparison to HMA 131 pen and about two times compared to HMA 49 pen, respectively. This shows that introducing cementitious fillers improves fatigue resistance greatly. The high stiffness modulus performance of the created mixes has resulted in the mixture's great resistance to fatigue cracking.

3.4. SEM Observation

The presence of alkali ions, supplied by CKD, accelerated the dissolution of PSA and formed the hydration products, primarily the ettringite (Figure 9). A similar hydration mechanism was reported by Sadique et al. [55] during the activation of high calcium fly ash with alkali sulfate-rich fly ash and Chaunsali and Peethamparan [56] for the CKD-GGBS system. The progressive formation of hydration products within the developed cold mix system containing the binary blend and consequently increasing ITSM as shown in Figures 5 and 6 was due to the increased activation of the PSA by the calcium and sulfur trioxide content of CKD as per the findings by Sadique and Coakley [57].

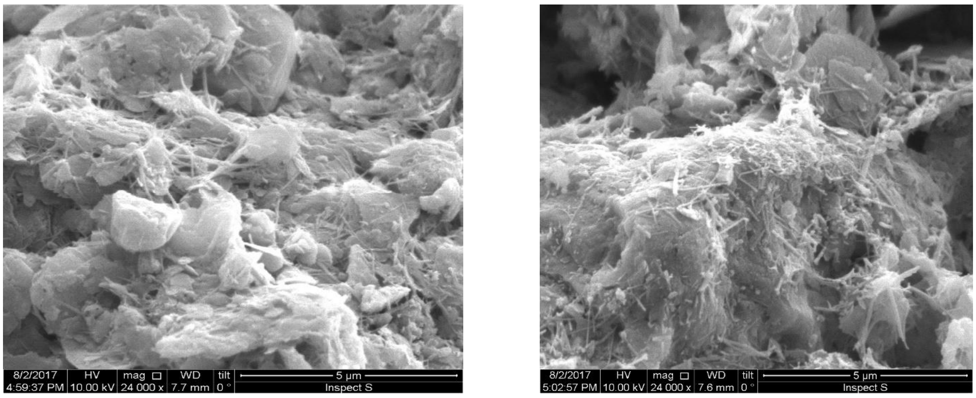

Figure 9. SEM of PSA and CKD at 28 days.

The formation of needle-like ettringite crystals, typical hydrates of the early hydration of the hydraulic binder and an essential and advantageous component of Portland cement systems, is shown in the micrograph of the paste after 28 days of curing. The calcium silicate hydrate (CSH) gel, a component of the cement system that is crucial to the majority of the engineering features of the cementitious binder, enriches the density at the micromolecular level and the surface area of the paste sample. It can be seen that the ettringite has thickened, spread out broadly, and covered the majority of the sample's surface area. At this point in the curing process, cementitious gel coats the majority of the ettringite. Consequently, in order to produce an advanced CBEM with visco-cohesion and interlocking-like bonding that will significantly increase the ITSM with the curing time, the two binders, bitumen and cementitious products, work together inside the CBEM.

4. Conclusions

Based on the results obtained from the experimental program, the following points can be concluded:

The full substitution of conventional mineral filler with waste fly ash (PSA) filler can greatly improve the ITSM of CBEM for the binder course. The newly developed CBEM showed a comparable ITSM to HMA after a short curing time period.

(1) PSA and CKD behave as secondary binding materials in CBEM binder course mixes, where hydration is essential to activate the CaO.
(2) PSA and CKD tend to accelerate the emulsion's breaking and coalescence. The results have shown that combining high alkali material with a high calcium hydroxide filler PSA, improves the performance of PSA significantly.
(3) After 10,000 cycles at 45 °C, CBEM with CLMF performed poorly in wheel track testing, while CBEM containing PSA and CKD showed an improved performance in terms of permanent deformation. At 45 °C, these mixtures form a cohesive dense microstructure by forming hydration products, meaning that they were more resistant to rutting. As a result, this mix is appropriate for usage in hot areas and on heavily trafficked highways.

(4) The addition of PSA and CKD to CBEM improves fatigue performance considerably. In comparison to conventional CBEM with CMLF and CBEM with PSA and CKD, as well as both grades of hot asphalt concrete, it had a longer fatigue life in four-point bending tests at strains of 150 microstrain. This enhancement can be linked to the newly produced mixture's cohesive and better interlocking integral microstructure, as well as the production of a rich binding paste in the hydration products.

Author Contributions: Conceptualization, A.D., S.A.-B. and H.A.N.; Data curation, R.A.-K.; Formal analysis, A.D. and S.A.-B.; Investigation, A.D., R.K.I., J.A. and W.P.; Methodology, M.A.K.; Software, R.K.I.; Supervision, A.D. and H.A.N.; Validation, M.S. and J.M.M.; Writing—original draft, A.D., M.A.K., R.A.-K. and W.P.; Writing—review and editing, J.A., A.D. and J.M.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

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

References

1. Al-Busaltan, S.; Al Nageim, H.; Atherton, W.; Sharples, G. Mechanical properties of an upgrading cold-mix asphalt using waste materials. *J. Mater. Civ. Eng.* **2012**, *24*, 1484–1491. [CrossRef]
2. Abdel-Wahed, T.; Dulaimi, A.; Shanbara, H.K.; Al Nageim, H.J.S. The impact of cement kiln dust and cement on cold mix asphalt characteristics at different climate. *Sustainability* **2022**, *14*, 4173. [CrossRef]
3. Dulaimi, A.; Shanbara, H.K.; Al-Rifaie, A.J.C.; Materials, B. The mechanical evaluation of cold asphalt emulsion mixtures using a new cementitious material comprising ground-granulated blast-furnace slag and a calcium carbide residue. *Constr. Build. Mater.* **2020**, *250*, 118808. [CrossRef]
4. Lu, D.; Wang, Y.; Leng, Z.; Zhong, J. Influence of ternary blended cementitious fillers in a cold mix asphalt mixture. *J. Clean. Prod.* **2021**, *318*, 128421. [CrossRef]
5. Leech, D. *Cold Asphalt Materials for Use in the Structural Layers of Roads*; Project Report 75; Transport Research Laboratory: Berkshire, UK, 1994.
6. Phillips, P. *Evaluation, Prediction and Enhancement of HAUCphalt-A Cold Mix Asphalt Technology*; University of Ulster: Belfast, Northern Ireland, 1998.
7. Dong, Q.; Yuan, J.; Chen, X.; Ma, X. Reduction of moisture susceptibility of cold asphalt mixture with Portland cement and bentonite nanoclay additives. *J. Clean. Prod.* **2018**, *176*, 320–328. [CrossRef]
8. Cui, P.; Schito, G.; Cui, Q. VOC emissions from asphalt pavement and health risks to construction workers. *J. Clean. Prod.* **2020**, *244*, 118757. [CrossRef]
9. Thives, L.P.; Ghisi, E. Asphalt mixtures emission and energy consumption: A review. *Renew. Sustain. Energy Rev.* **2017**, *72*, 473–484. [CrossRef]
10. Rubio, M.d.C.; Moreno, F.; Martínez-Echevarría, M.J.; Martínez, G.; Vázquez, J.M. Comparative analysis of emissions from the manufacture and use of hot and half-warm mix asphalt. *J. Clean. Prod.* **2013**, *41*, 1–6. [CrossRef]
11. Autelitano, F.; Bianchi, F.; Giuliani, F. Airborne emissions of asphalt/wax blends for warm mix asphalt production. *J. Clean. Prod.* **2017**, *164*, 749–756. [CrossRef]
12. Autelitano, F.; Giuliani, F. Analytical assessment of asphalt odor patterns in hot mix asphalt production. *J. Clean. Prod.* **2018**, *172*, 1212–1223. [CrossRef]
13. Jain, S.; Singh, B. Cold mix asphalt: An overview. *J. Clean. Prod.* **2021**, *280*, 124378. [CrossRef]
14. Dash, S.S.; Chandrappa, A.K.; Sahoo, U.C. Design and performance of cold mix asphalt—A review. *Constr. Build. Mater.* **2022**, *315*, 125687. [CrossRef]
15. Nassar, A.I.; Mohammed, M.K.; Thom, N.; Parry, T. Characterisation of high-performance cold bitumen emulsion mixtures for surface courses. *Int. J. Pavement Eng.* **2018**, *19*, 509–518. [CrossRef]
16. Kadhim, M.A.; Al-Busaltan, S.; Nemaa, Z.K.; Abo Almaali, Y.; Saghafi, B.; Al-Kafaji, M.; Al-Jawad, O.; Al-Yasari, R. Evaluating the cracking performance indices of half-warm mix asphalt comprising waste glass. *Int. J. Pavement Res. Technol.* **2021**, *8*, 1–15. [CrossRef]
17. Ling, C.; Hanz, A.; Bahia, H. Measuring moisture susceptibility of cold mix asphalt with a modified boiling test based on digital imaging. *Constr. Build. Mater.* **2016**, *105*, 391–399. [CrossRef]
18. Zhu, C.; Zhang, H.; Huang, L.; Wei, C. Long-term performance and microstructure of asphalt emulsion cold recycled mixture with different gradations. *J. Clean. Prod.* **2019**, *215*, 944–951. [CrossRef]

19. Kadhim, M.A.; Al-Busaltan, S.F.; Almuhanna, R.R. Characterize cold bituminous emulsion mixtures incorporated ordinary portland cement filler for local surface layer. *J. Univ. Babylon* **2018**, *26*, 247–263.
20. Al Nageim, H.; Al-Busaltan, S.F.; Atherton, W.; Sharples, G. A comparative study for improving the mechanical properties of cold bituminous emulsion mixtures with cement and waste materials. *Constr. Build. Mater.* **2012**, *36*, 743–748. [CrossRef]
21. Li, G.; Zhao, Y.; Pang, S.S.; Huang, W. Experimental study of cement-asphalt emulsion composite. *Cem. Concr. Res.* **1998**, *28*, 635–641. [CrossRef]
22. García, A.; Lura, P.; Partl, M.N.; Jerjen, I. Influence of cement content and environmental humidity on asphalt emulsion and cement composites performance. *Mater. Struct.* **2013**, *46*, 1275–1289. [CrossRef]
23. Gautam, P.K.; Kalla, P.; Jethoo, A.S.; Agrawal, R.; Singh, H. Sustainable use of waste in flexible pavement: A review. *Constr. Build. Mater.* **2018**, *180*, 239–253. [CrossRef]
24. Rubio, M.C.; Martínez, G.; Baena, L.; Moreno, F. Warm mix asphalt: An overview. *J. Clean. Prod.* **2012**, *24*, 76–84. [CrossRef]
25. Dulaimi, A.; Al-Busaltan, S.; Sadique, M. The development of a novel, microwave assisted, half-warm mixed asphalt. *Constr. Build. Mater.* **2021**, *301*, 124043. [CrossRef]
26. Toraldo, E.; Martinez-Arguelles, G.; Mariani, E. Effect of reinforced fibers on stiffness of recycled asphalt half-warm emulsion mixes. In *Sustainability, Eco-Efficiency, and Conservation in Transportation Infrastructure Asset Management*; CRC Press: Boca Raton, FL, USA, 2014; pp. 53–59.
27. Abdullah, M.E.; Zamhari, K.A.; Hainin, M.R.; Oluwasola, E.A.; Yusoff, N.I.M.; Hassan, N.A. High temperature characteristics of warm mix asphalt mixtures with nanoclay and chemical warm mix asphalt modified binders. *J. Clean. Prod.* **2016**, *122*, 326–334. [CrossRef]
28. Du, S.J.C.; Materials, B. Mechanical properties and reaction characteristics of asphalt emulsion mixture with activated ground granulated blast-furnace slag. *Constr. Build. Mater.* **2018**, *187*, 439–447. [CrossRef]
29. Dulaimi, A.; Al Nageim, H.; Ruddock, F.; Seton, L. Assessment the performance of cold bituminous emulsion mixtures with cement and supplementary cementitious material for binder course mixture. In Proceedings of the 38th International Conference on Cement Microscopy, Lyon, France, 17–21 April 2016; pp. 283–296.
30. Ge, Z.; Li, H.; Han, Z.; Zhang, Q. Properties of cold mix asphalt mixtures with reclaimed granular aggregate from crushed PCC pavement. *Constr. Build. Mater.* **2015**, *77*, 404–408. [CrossRef]
31. Shalan, A.A.; Al-Sahaf, N.A.; Al-Hdabi, A. Investigation of binder course cold asphalt emulsion mixture properties containing cement and GGBS. In Proceedings of the International Conference on Civil and Environmental Engineering Technologies, Najaf, Iraq, 23–24 April 2019; IOP Publishing: Bristol, UK, 2019; p. 012014.
32. Shanbara, H.K.; Dulaimi, A.; Al-Mansoori, T.; Al-Busaltan, S.; Herez, M.; Sadique, M.; Abdel-Wahed, T. The future of eco-friendly cold mix asphalt. *Renew. Sustain. Energy Rev.* **2021**, *149*, 111318. [CrossRef]
33. Russo, F.; Veropalumbo, R.; Oreto, C.; Biancardo, S.A.; Abbondati, F.; Viscione, N.J.C. Verifying the mechanical performance of cold and hot asphalt mastics containing jet grouting waste as a filler. *Coatings* **2021**, *11*, 751. [CrossRef]
34. Amoori Kadhim, M.; Al-Busaltan, S.; Dulaimi, A.; Sadique, M.; Al Nageim, H.; Al-Kafaji, M.; Al-Yasari, R. Developing a sustainable, post treated, half warm mix asphalt for structural surface layer. *Constr. Build. Mater.* **2022**, *342*, 127926. [CrossRef]
35. Dulaimi, A.; Al Nageim, H.; Ruddock, F.; Seton, L. Microanalysis of alkali-activated binary blended cementitious filler in a novel cold binder course mixture. In Proceedings of the 38th International Conference on Cement Microscopy, Lyon, France, 17–21 April 2016; pp. 189–205.
36. Malaiskiene, J.; Kizinievic, O.; Kizinievic, V.; Boris, R.J.C.; Materials, B. The impact of primary sludge from paper industry on the properties of hardened cement paste and mortar. *Constr. Build. Mater.* **2018**, *172*, 553–561. [CrossRef]
37. Baloochi, H.; Aponte, D.; Barra, M. Waste paper ash as a hydraulic road binder: Hydration, mechanical and leaching considerations. *J. Environ. Manag.* **2022**, *314*, 115042. [CrossRef] [PubMed]
38. Siddique, R.; Rajor, A. Use of cement kiln dust in cement concrete and its leachate characteristics. *Resour. Conserv. Recycl.* **2012**, *61*, 59–68.
39. Rahman, M.; Rehman, S.; Al-Amoudi, O. Literature review on cement kiln dust usage in soil and waste stabilization and experimental investigation. *Int. J. Res. Rev. Appl. Sci.* **2011**, *7*, 77–87.
40. Al-Merzah, S.; Al-Busaltan, S.; Al Nageim, H. Investigating the feasibility of using cement Kiln dust in cold bituminous emulsion asphalt. In Proceedings of the 4th International Congress on Civil Engineering, Architecture and Urban Development, Tehran, Iran, 27–29 December 2016.
41. Read, J.; Whiteoak, D. *The Shell Bitumen Handbook*, 5th ed.; Shell Bitumen: London, UK, 2003.
42. European Committee for Standardization. *BS EN 13108: Part 1. Bituminous Mixtures Materials Specification-Asphalt Concrete*; British Standard Institution: London, UK, 2016.
43. Nikolaids, A. Construction and performance of dense cold bitumen mixtures as strengthening over layer and surface layer. In Proceedings of the 1st European Symposium on Performance and Durability of Bituminous Materials, London, UK, March 1994.
44. Konsta-Gdoutos, M.S.; Shah, S.P. Hydration and properties of novel blended cements based on cement kiln dust and blast furnace slag. *Cem. Concr. Res.* **2003**, *33*, 1269–1276. [CrossRef]
45. Deb, P.; Singh, K.L. Accelerated curing potential of cold mix asphalt using silica fume and hydrated lime as filler. *Int. J. Pavement Eng.* **2022**, 1–21. [CrossRef]
46. Asphalt Institute. *Asphalt Cold Mix Manual, Manual Series No.14 (MS-14)*, 3rd ed.; Asphalt Institute: Lexington, KY, USA, 1997.

47. Serfass, J.-P.; Poirier, J.-E.; Henrat, J.-P.; Carbonneau, X. Influence of curing on cold mix mechanical performance. *Mater. Struct.* **2004**, *37*, 365–368. [CrossRef]
48. Jenkins, K.J. Mix Design Considerations for Cold and Half-Warm Bituminous Mixes with Emphasis of Foamed Bitumen. Ph.D. Thesis, Stellenbosch University, Stellenbosch, South Africa, 2000.
49. European Committee for Standardization. *BS EN 12697: Part 26. Bituminous Mixtures-Test Methods for Hot Mix Asphalt-Stiffness*; British Standards Institution: London, UK, 2012.
50. Wang, Z.; Zhao, Z.; Yang, C.; Yang, X.; Chen, S.; Zou, Y. Performance evaluation of asphalt modified with steel slag powder and waste tire rubber compounds. *Sustainability* **2022**, *14*, 8615. [CrossRef]
51. European Committee for Standardization. *BS EN 12697: Part 22. Bituminous Mixtures-Test Methods for Hot Mix Asphalt-Wheel Tracking Test Methods for Hot Mix Asphalt*; British Standards Institution: London, UK, 2003.
52. European Committee for Standardization. *PD 6691: Guidance on the Use of BS EN 13108 Bituminous Mixtures–Material Specifications*; British Standards Institution: London, UK, 2010.
53. Thanaya, I. Improving the performance of cold bituminous emulsion mixtures (CBEMs) incorporating waste materials. In *Performance of Bituminous and Hydraulic Materials in Pavements*; Routledge: Oxfordshire, UK, 2003.
54. Brown, S.F.; Needham, D. A study of cement modified bitumen emulsion mixtures. In Proceedings of the Association of Asphalt Paving Technologists (AAPT), Reno, NV, USA, 10–13 January 2000.
55. Sadique, M.; Al-Nageim, H. Hydration kinetics of a low carbon cementitious material produced by physico-chemical activation of high calcium fly ash. *J. Adv. Concr. Technol.* **2012**, *10*, 254–263. [CrossRef]
56. Chaunsali, P.; Peethamparan, S. Evolution of strength, microstructure and mineralogical composition of a CKD–GGBFS binder. *Cem. Concr. Res.* **2011**, *41*, 197–208. [CrossRef]
57. Sadique, M.; Coakley, E. The influence of physico-chemical properties of fly ash and CKD on strength generation of high-volume fly ash concrete. *Adv. Cem. Res.* **2016**, *28*, 595–605. [CrossRef]