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Optimum Fluid Content in Pavement Cold In-Place Recycling Containing Waste Materials

Miloš Kopić, Tiana Milović, Bojan Matić, Stanislav Jovanović and Milan Marinković *

Faculty of Technical Sciences, University of Novi Sad, Trg Dositeja Obradovića 6, 21000 Novi Sad, Serbia
* Correspondence: milan.marinkovic@uns.ac.rs; Tel.: +381-65-5773-817

Abstract: The planning of road infrastructure undergoes major changes, especially in terms of sustainable development. Recycling of pavement structures involves the reuse of materials from existing pavement structures due to its timesaving and environmental benefits, as well as cost reduction. According to the recycling temperature, recycling can be hot and cold. This paper deals with cold in-place recycling and the determination of the optimum fluid content for by-product materials in mixtures compared with one containing natural zeolite. The content of bitumen emulsion and cement—which are the most used materials so far in cold recycling along with foam bitumen—was replaced with fly ash, slag or natural zeolite, and bakelite, respectively, while recycled asphalt pavement from Serbia (Žabalj) was used. Six different mixtures were made. The mixture with the addition of fly ash had the highest optimum fluid content (7.6%) compared with all test mixtures. Mixtures with slag, natural zeolite, and bakelite were in the range of a mixture containing 2% cement. Furthermore, the mixture with 3% cement had the lowest optimum fluid content (5.7%) in comparison to all the mixtures that were tested.

Keywords: reclaimed asphalt pavement (RAP); Portland cement; fly ash; slag; natural zeolite; bakelite; bitumen emulsion; sustainable pavement

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1. Introduction

Due to the rapid population growth around the world [1], there is not only a need for additional development of road infrastructure [2,3] but also its sustainability. Considering the multiple benefits in terms of environmental protection, the rational use of resources and energy consumption, the reduction of air pollution in cities and global warming, and the planning and design of sustainable cities should include the use of recycled materials on a larger scale in the construction and reconstruction of road infrastructure. Recycling road construction materials for reuse in new pavements [4] as well as utilizing binders from less environmentally damaging sources [5] represent the important components for achieving the goal of sustainable pavements.

Over the past decade, there has been an increase in the amount of recycled asphalt, which has led road designers to consider recycling as a sustainable rehabilitation method, especially in the case of severely deteriorated asphalt pavements [6–8]. In the world, there are different classifications of asphalt recycling techniques. Just in the United States, five broad categories are defined to describe the different asphalt recycling methods (full-depth reclamation, cold planning, cold recycling, hot recycling, and hot in-place recycling) according to the Asphalt Recycling and Reclaiming Association [9]. In Europe, the classification of asphalt pavement recycling, regarding the place where mixing is carried out (in-place or in-plant), the temperature of the process (cold or hot) [10,11], the characteristics of the material to be recycled, and the binder type have been proven to be more applicable [12,13].

Cold recycling is a type of recycling in which old pavement layers are removed and crushed into reclaimed asphalt pavement (RAP), and then are mixed with bituminous and/or mineral binders, paved, and compacted at ambient temperatures [4,14]. The use of

cold in-place recycling (CIR) of asphalt pavement up to 100% [15] is an environmentally and economically feasible alternative to hot recycling [16].

One of the binders that can be used in a CIR mixture is cement [17,18]. Global production of cement has very rapidly grown in recent years, and, after fossil fuels and land-use change, it is the third-largest source of anthropogenic emissions of carbon dioxide [19]. Considering that cement production makes up 8% of overall global emissions [20,21], there is an interest in the partial replacement of cement by supplementary cementitious materials (SCMs) with pozzolanic properties and low embodied CO_2 footprints [22], such as waste materials (e.g., fly ash [23], slag [21], or waste glass [24]) and natural pozzolans (e.g., natural zeolite [22,25,26]). Furthermore, the waste materials can also be used in the concrete industry as aggregates [27–30] or fibers [31,32] to reduce the exploitation and costs of raw materials.

From an aspect of cold in-place recycling, Wei et al. [33] obtained as a result of the experiment that there is a difference in optimum fluid content (OFC) depending on the way the samples are compacted. For the specimens made by the vibrating compaction method, they found that OFC was 4.7%, while using the heavy compaction method the OFC value was 5%.

Authors in Brazil [34] varied the content of bitumen emulsion (2%, 3%, and 4%), cement (0%, 1%, and 2%), and water. Also, the authors experimented with the curing speed by curing samples according to standard (25 °C) and expedited procedure (40 °C, 60 °C, and 100 °C). By testing according to the standard curing procedure, they determined that OFC was 6.2% for mixtures with 2% bitumen emulsion, 5.8% for mixtures with 3% emulsion, and 5.3% for mixtures with 4% emulsion.

In South Africa [35], the authors investigated the influence of the percentage of bitumen emulsion and cement on indirect tensile strength. The goal was to choose the best mixture for a road. Ultimately, a residual bitumen content of 2.1% was chosen as optimal. Kezhen et al. [36] investigated the effect of rice husk ash filler on the mechanical properties of bitumen emulsion-based recycled asphalt specimens. In their experimental research, the OFC was 4.5%. Investigations of the specimens showed that the presence of rice husk ash filler improved the water sensitivity and high-temperature performance of asphalt mixtures, but also negatively affected low-temperature performance.

Zhao et al. [37] investigated strength indicators, high-temperature stability, low-temperature crack resistance, water stability, and dynamic modulus. In this paper, six types of cold recycled mixtures (CRM) with varying RAP and reclaimed inorganic binder stabilized aggregate (RAI). The composition of all mixtures, optimum water content, and optimum binder content for all mixtures are shown in Table 1.

Table 1. Mixtures and optimal water content and optimum asphalt content [37].

Mixture	Optimum Water Content (%)	Optimum Asphalt Content (%)
Emulsified asphalt CRM with RAP	4.8	3.8
Emulsified asphalt CRM with composite RAP and RAI	5.6	4.5
Emulsified asphalt CRM with RAI	6.4	5.0
Foamed asphalt CRM with RAP	5.0	3.2
Foamed asphalt CRM with composite RAP and RAI	5.8	3.6
Foamed asphalt CRM with RAI	6.6	4.4

RAP with cement and bitumen emulsion was used for the construction of a rural road in Serbia [38]. In this case, no new aggregate was added. The test determined the OFC of 6%. Significant research in Serbia, concerning the application of RAP, emulsion,

and cement in cold recycling was conducted by Jakovljević [39]. The mentioned author examined six groups of mixtures. The content of cement, bitumen emulsion, crushed stone 0/22 mm, and RAP was different in the composition of individual mixes. The crushed stone content was 100%, 50%, and 20% while based on this the content of RAP was 0%, 50%, and 80%, respectively. Compressive strength, as well as indirect tensile strength after 28 days, were determined. In the second phase, fatigue and modulus were tested for each mixture with the OFC. The optimum content of bitumen emulsion for all combinations of crushed stone and RAP was 3.5%. It should be emphasized that this methodology differs from that applied in this paper.

Previous research in the field of pavement CIR has included the application of cement, bitumen emulsion, and foamed bitumen [40,41]. In order to reduce the negative impact of cement production on the natural environment, SCMs have been used. In the research presented in this paper, 20% of Portland cement (PC) was substituted with solid industrial by-products (fly ash/slag), or with natural pozzolanic material (zeolite) in the same amount. Furthermore, 20% of the bitumen emulsion was replaced with bakelite.

Bitumen emulsion, cement, and foamed bitumen were dominantly used in the majority of research and experiments in the field of CIR. Some research also addresses the use of fly ash and slag in CIR [42,43]. So far, there have been no publications on the application of zeolite and bakelite in CIR, although they are used in hot-mix asphalt (HMA) and warm-mix asphalt (WMA) mixtures [44,45].

This paper deals with (i) the effect of different amounts of PC (2% and 3%), (ii) the effect of partial substitution of 2% PC with fly ash, slag, and natural zeolite, and (iii) the effect of partial substitution of bitumen emulsion with bakelite, by mass, on OFC in sustainable pavement CIR. The mixture with 2% PC represents the reference mixture because most research and regulations limit the cement content to 2%.

2. Materials and Methods

2.1. Component Materials

The research in this paper is a continuation of research that has already been published [46]. For the experimental investigation of the influence of (i) partial substitution of PC with SCMs and (ii) partial substitution of bitumen emulsion with bakelite on the OFC in pavement CIR, the following component materials were used: PC, fly ash, slag, and natural zeolite, presented in Figure 1, as well as filler, bitumen emulsion, bakelite, RAP, and tap water. The locations from which the component materials were taken are marked in Figure 2.

Ordinary Portland cement (CEM I 42.5R, from Lafarge-BFC, Beočin, Serbia) with specific weight and Blaine surface area of 3.126 g/cm^3 and 4188.6 cm^2/g, determined in accordance with EN 1097-7 [47] and EN 196-6 [48], respectively, was used.

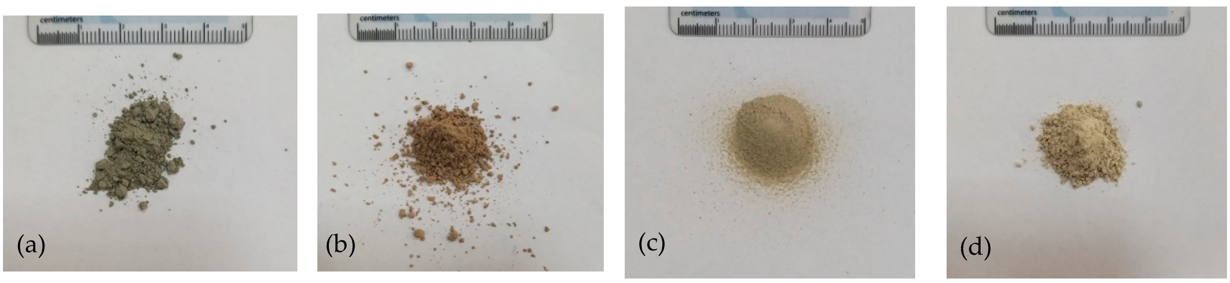

Figure 1. Binders: (**a**) Portland cement, (**b**) fly ash, (**c**) slag, and (**d**) natural zeolite.

Figure 2. Locations from which the component materials were taken.

Applied fly ash was a by-product from the thermal power plant Nikola Tesla B (in Obrenovac, Serbia). Only particles smaller than 0.125 mm were used as SCM. The specific weight and Blaine surface area of the fly ash were 2.313 g/cm^3 and 10,212.47 cm^2/g, determined in accordance with EN 1097-7 [47] and EN 196-6 [48], respectively. The fineness of fly ash was determined by air-jet sieving in accordance with EN 933-10 [49]. Based on the obtained fineness results (18.0%), fly ash belongs to category N according to EN 450-1 [50].

Slag was a by-product from the company Hesteel Serbia (in Smederevo, Serbia) and was also used as SCM with a particle size smaller than 0.125 mm. It was delivered in the form of solid material, and then ground and sieved through a 0.045 mm sieve to reach a Blaine surface area over 2750 cm^2/g in accordance with EN 15167-1 [51]. The specific weight and Blaine surface area of the slag were 2.689 g/cm^3 and 2798.90 cm^2/g, determined in accordance with EN 1097-7 [47] and EN 196-6 [48], respectively. The fineness of slag (60.8%) was determined by an air-jet method by sieving through a 0.045 mm mesh sieve in accordance with EN 933-10 [48].

Natural zeolite, used as a partial replacement of PC in mixtures, was originally from a quarry, "Igroš-Vidojevići" (in Brus, Serbia), with particle size smaller than 0.125 mm. The specific weight and Blaine surface area of the applied zeolite were 2.386 g/cm^3 and 8292.97 cm^2/g, determined in accordance with EN 1097-7 [47] and EN 196-6 [48], respectively. The fineness of the zeolite was 30.1%. The fineness was determined as the mass proportion in percent of the natural zeolite retained when the sample was sieved on a 0.045 mm mesh sieve, by air-jet sieving in accordance with EN 933-10 [49]. Based on the obtained fineness results, zeolite belongs to category N according to EN 450-1 [50]. Natural zeolites as volcanic or volcano-sediment materials have a honeycomb-like structure with extremely small pores and channels that offer a large total specific surface, which represents the base of their high pozzolanic reactivity [52]. For this reason, zeolites are very interesting in the study of sustainable concrete [53] as well as sustainable pavement.

The filler (stone dust) was from the company Karin Komerc MD (in Novi Sad, Serbia). The granulometric composition of the filler was such that more than 95% of the particles pass through a 0.045 mm sieve and, therefore, was used for the granulometric correction of the recycled aggregate. The material met the criteria defined in EN 12620 [54].

A stable cationic bitumen emulsion [55] from Mala Krsna, Serbia was used. The emulsion consists of 60% bitumen and 40% water. According to the EN 13808 [56] standard, bitumen emulsion is classified as C60 B4. To achieve better bonding between the emulsion and cement, but also other binder materials, emulsifiers were added to the emulsion. The emulsion was tested according to the EN 12591 [57] standard and met all required criteria.

Bakelite or waste plastic was obtained from Metalac company (Gornji Milanovac, Serbia). Bakelite is scaly in shape with a dark color and an odor. There are two types

of plastics: thermoplastics and thermosets. The main difference is that thermoplastics can be reused, while thermosets can no longer be used once they have gone through the production process. Bakelite belongs to the group of thermosets. The delivered material (Figure 3a) was in bags and irregular in shape. Bakelite that passed through a 0.125 mm sieve, after which it was further crushed (Figure 3b), was used to make mixtures and had a loss on ignition (LOI) of 98%, and the fineness of 52.1% (by air-jet sieving through a 0.045 mm mesh sieve in accordance with EN 933-10 [49]).

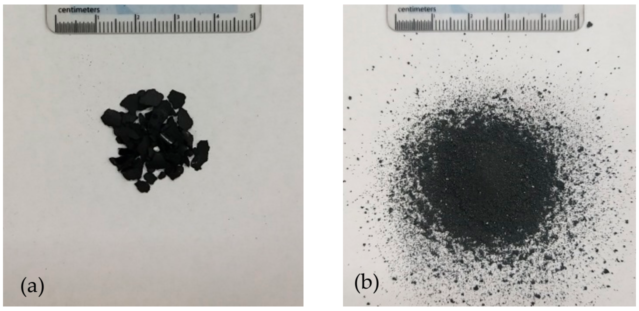

Figure 3. Bakelite (**a**) in delivered form and (**b**) after sieving and crushing.

RAP was originally from the pavement construction in Sveti Nikola Street in Žabalj, Serbia (Figure 4). The length of the road was 1160 m. Site samples were determined based on Benkelman beam deflection measurements. The Benkelman beam measures deflections without damaging the pavement, and therefore is defined as a non-destructive way of testing the pavement structure [58–60]. Pavement deflection depends on the temperature, and it has been seen that the pavement layers vary according to variations in the temperature [61]. Deflection values are collected in divisions for every 50 m interval and the temperature is recorded along.

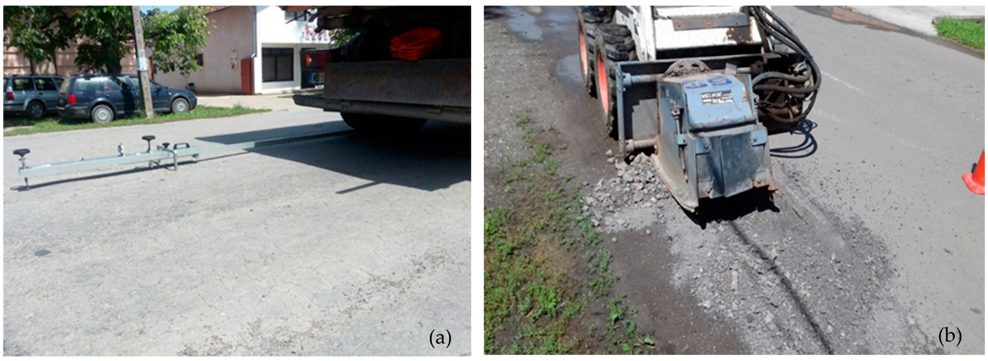

Figure 4. (**a**) Measuring with a Benkelman beam and (**b**) taking RAP samples.

Three homogeneous sections were obtained by analyzing the measured values of deflections based on statistical data. Homogeneous sections are:

- Homogenous section 1 (chainage: from km 0 + 000 to 0 + 200);
- Homogenous section 2 (chainage: from km 0 + 200 to 0 + 400);
- Homogenous section 3 (chainage: from km 0 + 400 to 1 + 160).

In each homogeneous section, a sample of the material was taken to a depth of 20 cm because cold recycling was designed for this depth. Samples of asphalt layers and layers of crushed stone were taken from the existing pavement construction. The thickness of the asphalt layers was 10 cm as well as the thickness of the crushed stone. Asphalt concrete and crushed stones were mixed in a mass ratio of 1:1.

After mixing the material (Figure 5), a representative sample was taken according to the standard [62]. The RAP was dried to a constant mass to evaporate the moisture and

to be able to determine the exact granulometric composition. Moisture-free material is a condition for the determination of the OFC. If the RAP is not completely dried then it would be impossible to determine the OFC, i.e., the obtained value would not be correct.

Figure 5. (**a**) Mixing of recycled asphalt, (**b**) determination of sample, and (**c**) determination of granulometric composition.

The chemical compositions of PC, fly ash, slag, natural zeolite, and filler, presented in Table 2, were determined by energy-dispersive X-ray fluorescence, ED-XRF (Spectro XEPOS C, Kleve, Germany). The system used a silicon drift detector and band-pass filter, and focuses the X-rays from a binary Co/Pd alloy thick-target anode (50 W/60 kV) combining polarized/direct excitation. The sample tray had rotating positions for pellets. The samples for ED-XRF analysis were prepared in accordance with the pressed powder method. The material (5 g) and the binding agent (Cereox wax, Fluxana) were mixed, and then the 32 mm diameter pellets were formed under a 10-ton load applied via a laboratory hydraulic press. Measurements were carried out in a vacuum atmosphere. Spectro XRF Analyzer Pro software version 2.0 was used. LOI was defined as a weight difference of samples between 20 °C and 950 °C.

Table 2. Chemical composition of PC, fly ash, slag, natural zeolite, and filler (wt.%).

	Chemical Composition (wt.%)				
	CEM I 42.5 R	**Fly Ash**	**Slag**	**Zeolite**	**Filler**
Na_2O	0.32	0.33	0.45	0.96	0.90
MgO	2.75	3.28	7.05	2.28	1.04
Al_2O_3	5.10	23.59	8.14	13.84	5.64
SiO_2	21.40	41.73	39.63	60.91	76.36
P_2O_5	0.14	0.13	0.05	0.15	0.08
SO_3	3.47	3.29	1.00	0.00	0.00
Cl^-	0.02	0.00	0.03	0.00	0.00
K_2O	0.76	1.19	0.57	1.00	0.96
CaO	60.66	10.87	39.76	5.12	6.46
TiO_2	0.23	1.50	0.52	0.59	0.53
Cr_2O_3	0.02	0.08	0.01	0.01	0.20
Mn_2O_3	0.09	0.25	0.66	0.09	0.10
Fe_xO_y	2.65	11.11	1.03	4.06	3.00
ZnO	0.05	0.12	0.00	0.01	0.01
SrO	0.10	0.09	0.10	0.27	0.03
BaO	0.04	0.14	0.11	0.71	0.03
LOI	2.20	2.30	0.90	10.00	4.65

As shown in Table 2, the sums of the oxides—which are relevant for pozzolanic reaction—are greater than 70%, allowing the classification of natural zeolite and fly ash into silicate mineral additions (type II), conforming to EN 450-1 [50]. In the case of the slag, the sum of CaO, MgO, and SiO_2 is 86.44%, while the ratio of (CaO + MgO) and SiO_2 is 1.18, which is in accordance with EN 15167-1 [51]. Therefore, slag can be classified as a type II addition in accordance with EN 206-1 [63].

Additionally, the mineralogical characterization of natural zeolite was performed to determine the main type of zeolite in the presented tuff. For the stated purpose, XRD analysis (Philips PW1710 device, Philips, Amsterdam, The Netherlands) was used under the following experimental conditions: monochromatic Cu Kα radiation with 1.5418 Å wavelength in the 5–55° of 2θ range, scan rate 0.02° and 0.5 s per step, at a voltage of 40 kV, and a current of 30 mA. According to the XRD results shown in Figure 6, the following minerals are presented in the zeolitic tuff sample: clinoptilolite (as the main zeolite mineral), smectite, mica group minerals, quartz, feldspar, analcime, and amphibole. The results of mineralogical characterization are in accordance with literature data, i.e., the presence of clinoptilolite as the main zeolite mineral in zeolitic tuff from the quarry "Igroš", as well as the presence of smectite, mica group minerals, quartz, feldspar, and analcime, is also confirmed by Kašić et al. [64], while the presence of amphibole in subordinate quantities is determined by Cocić et al. [65].

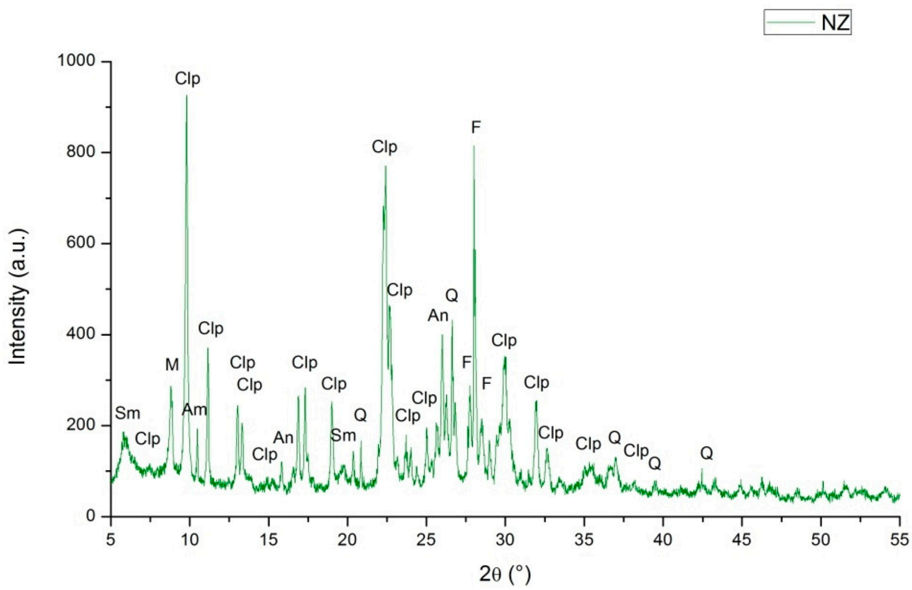

Figure 6. XRD diffractogram of natural zeolite (Clp—clinoptilolite, Sm—smectite, M—mica group minerals, Q—quartz, F—feldspar, An—analcime, and Am—amphibole).

Today, more than 50 types of natural zeolite minerals are known [52], and clinoptilolite is the one that is the most used as SCM [22,52,66]. However, smectite is a type of clay mineral whose swelling ability can affect the properties of fresh and hardened cement-based composites [67].

2.2. Mixture Compositions and Methodology

A total of six different mixtures was the subject of the following research, while the details about the proportion of PC, by-products, and natural zeolite in mixtures are provided in Table 3.

Table 3. Proportions of PC, by-products, natural zeolite, and bakelite in mixtures.

	Cement (%)	Addition	Proportion of Addition (%)
Mixture 1	2		
Mixture 2	1.6	Fly ash	0.4 (instead of 20% cement)
Mixture 3	1.6	Slag	0.4 (instead of 20% cement)
Mixture 4	1.6	Zeolite	0.4 (instead of 20% cement)
Mixture 5	2	Bakelite	20% instead of bitumen emulsion
Mixture 6	3		

In general, there is no single method in the world for the design of cold in-place recycling. Many of the methods are improvements on the design methods associated with hot mix asphalt mixtures [4].

The methodology applied in this research is based on the Wirtgen guideline for cold recycling [68] and guidelines from South Africa [69]. The size of the test specimens is defined by the mentioned manuals. The diameter of the tested specimens is 150 mm. In-place recycling design consists of the following steps:

- Testing of sampled RAP and possible correction of granulometric composition;
- Determining OFC;
- Determination of mechanical characteristics of samples made with optimum fluid content (ITS, etc.).

This paper presents the first two steps of designing a cold recycling mixture. Tests of RAP have shown that the granulometric composition of the sample did not meet the limit values, so it needed to be corrected (Table 4). The granulometric curve of the RAP sample was compared with the granulometric curve from the Wirtgen manual [70]. The granulometric curve did not satisfy the passages at the smallest sieve openings. When designing cold recycled mixtures, there is often a shortage of small grains because they are bound in bitumen.

Table 4. Granulometric composition of RAP and composition correction.

Sieve Size (mm)	RAR (g)	Filler (g)	Corrected Mass (g)	Passes through Sieves (g)	Passes through Sieves (%)	Passes through Sieves for Unit Sample (g)	Remains on Sieves (g)
75.00	0.00		0.00	15,027.43	100	5500	0.0
63.00	0.00		0.00	15,027.43	100	5500	0.0
45.00	0.00		0.00	15,027.43	100	5500	0.0
31.50	48.43		48.43	14,979.00	99.68	5482.28	17.7
22.40	1787.25		1787.25	13,191.75	87.78	4828.15	654.1
16.00	1961.75		1961.75	11,230.00	74.73	4110.15	718.0
11.00	1245.75		1245.75	9984.25	66.44	3654.21	455.9
8.00	1130.00		1130.00	8854.25	58.92	3240.63	413.6
4.00	2301.25	0	2301.25	6553.00	43.61	2398.38	842.3
2.00	1534.50	0	1534.50	5018.50	33.39	1836.76	561.6
1.00	926.75	0	926.75	4091.75	27.23	1497.57	339.2
0.50	449.50	0	449.50	3642.25	24.24	1333.05	164.5
0.25	213.50	0	213.50	3428.75	22.82	1254.91	78.1
0.06	128.75	3300	3428.75	0.00	6.00	330.00	924.9
bottom							330.0
Sum	11,727.43	3300					5500.0

RAP was tested according to EN 933-10:2009 [49] and EN 933-2:2020 [71].

The optimum fluids concept is often applied to cold recycled mixtures with the aim of optimizing the packing of the solid particles. The OFC represents the content of fluids at the closest packing of the aggregate particles or the maximum dry density [72].

The amount of participation of individual fractions of the aggregate was determined by calculation, taking into account that the weight of individual samples is 5500 g (Table 4). The ratio of RAP and additional fine-grained material (filler) in the corrected mixture, was 78%:22%. The corrected granulometric curve of the mixture is shown in Figure 7. After correcting the granulometric composition, the OFC was examined. The OFC means the amount of fluid at which the maximum dry bulk density is achieved. Total fluids consist of two parts. The first part includes water that is added to the mixture without additives, and the second part is water in the bitumen emulsion. For each different binder added to recycling mixtures, the OFC must be determined in order to be able to design the mixture and examine the mechanical properties of the mixture.

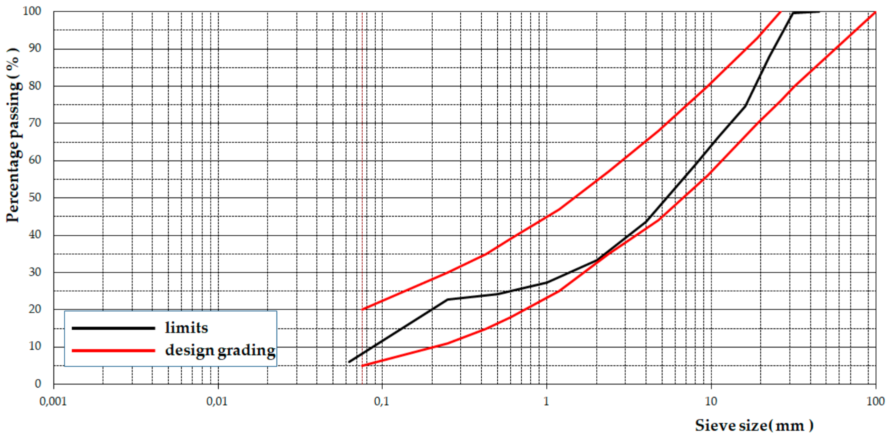

Figure 7. Corrected granulometric composition of mixtures and limits.

It is recommended to make a minimum of three mixtures, with three different fluid contents, to be able to determine the OFC from the diagram of fluid content-bulk density.

The initial content of fluids is determined based on experience, based on the type and composition of RAP, and additional binders. Sometimes it is necessary to make more than three mixtures when the OFC cannot be read from the diagram with three different percentages of fluid content. This happens when the bulk density constantly increases or constantly decreases with increasing fluid content.

The test began by mixing water and bitumen emulsion in a mass ratio (1:1) per fluid content, Table 5.

Table 5. Composition of test mixtures for OFC (W—water and BE—bitumen emulsion).

	Cement (g)	Addition		4%		6%		8%	
		Type	Mass (g)	W	BE	W	BE	W	BE
Mixture 1	110	/	0	110	110	165	165	220	220
Mixture 2	88	Fly ash	22	110	110	165	165	220	220
Mixture 3	88	Slag	22	110	110	165	165	220	220
Mixture 4	88	Zeolite	22	110	110	165	165	220	220
Mixture 5	88	Bakelite	22/33/44	110	88	165	132	220	176
Mixture 6	165	/	0	110	110	165	165	220	220

Samples weighing 5500 g were used to test the OFC so that the quantities of material were determined based on that mass (e.g., for samples to which 6% of the fluid content

was added, a total of 330 g of fluid was required, which was divided into 165 g of water and 165 g bitumen emulsion). All mixtures were prepared with 4%, 6%, and 8% fluids to determine the OFC.

During the preparation of samples, water and bitumen emulsion were added to the previously prepared mixture of RAP, filler, and cement (zeolite/fly ash/slag/bakelite). After combining all the ingredients, the material was mixed until a homogeneous mixture was obtained. Furthermore, the mixture was compacted according to the requirements of the modified Proctor test and packed in a mold, and then the samples were taken out using an extractor. Removed samples were left in the oven to dry to constant mass at the temperature of 40 °C, and they were periodically checked by measuring their mass and comparing them with the previous measurements. The approximate drying time of the samples was 72 h. Based on a certain OFC, the amount of water required for the best workability and compaction of the mixture is determined in the following way:

$$W_{OCC} = W_{OFC} - W_{RED} \quad (1)$$

where:

W_{OCC}—Water content for optimum compaction and workability (% by mass)
W_{OFC}—Optimum fluid content—OFC (% by mass);
W_{RED}—Reduction amount (% by mass).

3. Test Results and Discussion

The results of the OFC testing and maximum dry density value for different mixtures are shown in Table 6. Figure 8 shows the results of the Proctor test for variable molding fluid contents.

Table 6. OFC and maximum bulk density of the mixtures.

	Cement (%)	Proportion of Addition (%)	Maximum Dry Density (g/cm^3)	OFC (%)
Mixture 1	2		2.163	6.5
Mixture 2	1.6	0.4 Fly ash (instead of 20% cement)	2.182	7.6
Mixture 3	1.6	0.4 Slag (instead of 20% cement)	2.133	6.4
Mixture 4	1.6	0.4 Zeolite (instead of 20% cement)	2.162	6.5
Mixture 5	2	Bakelite (instead of 20% bitumen emulsion)	2.123	6.4
Mixture 6	3		2.131	5.7

Mixture 1 (containing 2% PC) was considered the reference mixture. The mixture containing 3% cement had the lowest OFC (5.7%). In comparison with the reference mixture, it had a lower OFC value of 25%. The highest OFC (7.6%), as well as maximum dry density (2.182 g/cm^3), contained the mixture with the addition of fly ash. Santos et al., 2011 [73] confirmed that fly ash requires a larger amount of liquid. According to Kim et al. [74], higher optimum water content could be associated with the pozzolanic reaction of fly ash and releasing the capillary tension from the greater exposed surface of the finer fly ash particles. The above-mentioned statement is in accordance with the obtained OFC value (7.6%) and a very high specific surface area of the applied fly ash in this study (10,212.47 cm^2/g). The remaining four mixtures had approximately the same value of OFC.

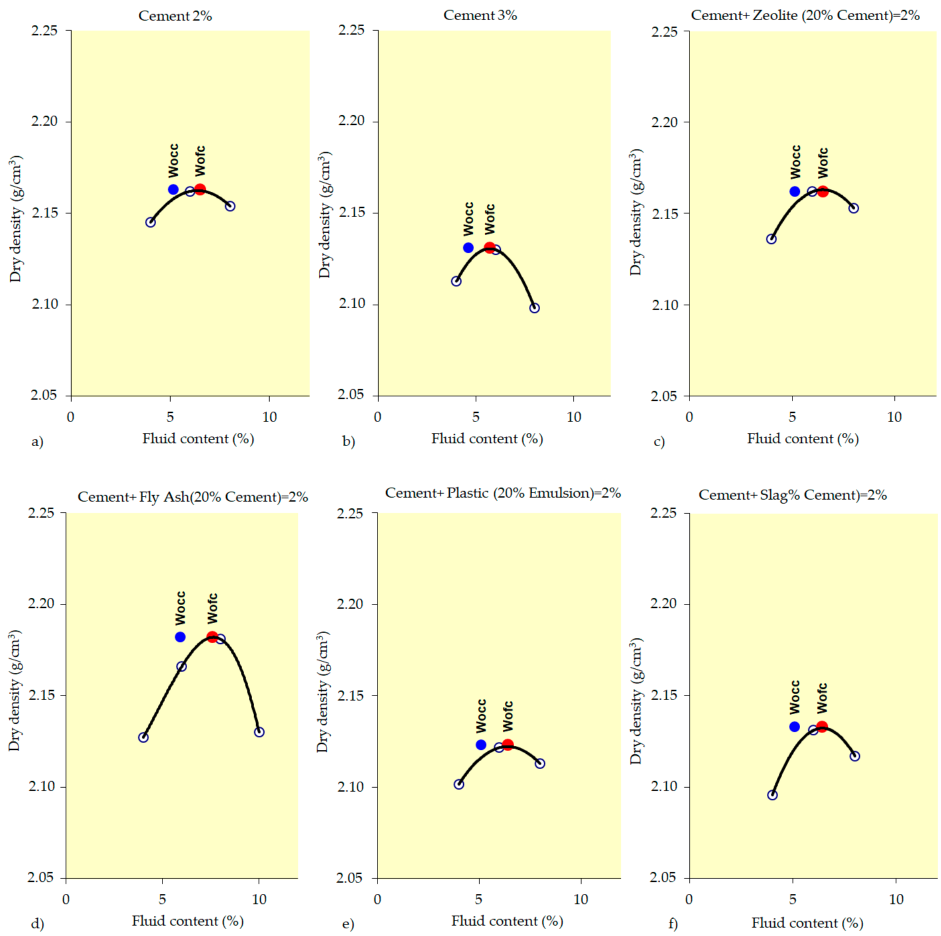

Figure 8. OFC for tested mixtures with (**a**) 2% cement, (**b**) 3% cement, (**c**) cement and zeolite, (**d**) cement and fly ash, (**e**) cement and plastic, and (**f**) cement and slag.

In the case of mixtures containing 20% partial replacement of PC, the mixture with natural zeolite had the same value of OFC as reference one, and approximately the same maximum dry density value as the reference mixture (with 2% PC). Although natural zeolites have the ability to adsorb and release water over 30% of their dry weight when they are ground into the powder [52], the mixture containing natural zeolite (mixture 4) as a partial replacement of PC had the same OFC as mixture 1. A possible reason for the above-mentioned could be the order of mixing the component materials, i.e., first, cement was mixed with natural zeolite and then added to RAP, after which a mixture of water and bitumen emulsion was added to the obtained mixture of dry ingredients. If the dry mixture of PC and natural zeolite had first been combined with water, the natural zeolite would probably have absorbed the water to a greater extent, which happens, e.g., in the case of preparing the mortars. The mixture with slag had a slightly lower value of OFC (for cca 2%), while the mixture with fly ash had a higher value of OFC (for cca 17%) compared with the reference value. The slightly reduced value of OFC in the case of the mixture containing slag could be related to the low value of the specific surface of the slag (2798.90 cm^2/g). PC, natural zeolite, and fly ash had for cca 50%, 196%, and 265% higher Blaine surface area compared with slag, respectively, and therefore a higher water demand, as binders.

The mixture containing bakelite had the same OFC (6.4%) as the mixture with added slag and similar maximum dry density values, while it had the lowest maximum dry density of all tested mixtures. The same trends can be observed for the optimal water content—W_{occ} (marked with blue dots) and OFC—W_{ofc} (marked with red dots), as shown in Figure 8.

The mixture with 2% cement was adopted as a reference mixture against which all other mixtures were compared. The reason why this mixture was chosen as a reference is that most of the research in the field of cold in-place recycling examined specimens with a cement content of up to 2%, and the values obtained can be compared. A comparison of the results of our experiment with the results of other authors is given in Table 7.

Table 7. OFC for mixes of individual authors.

Author	Cement Content (%)	Additives	Optimum Fluid Content (%)
Wei et al. [33]	1.5	none	4.7 (vibrating compaction method)
Wei et al. [33]	1.5	none	5.0 (heavy compaction method)
Jenkins, K.J. [35]	1	none	2.1
Kezhen et al. [36]	<1.5	none	4.5
Zhao et al. [37]	1.5	none	3.8
Jakovljević [39]	/	none	3.5
Smiljanić et al. [38]	2	none	6
Mixture 1	2	none	6.5
Mixture 2	1.6	fly ash (0.4%)	7.6
Mixture 3	1.6	slag (0.4%)	6.4
Mixture 4	1.6	zeolite (0.4%)	6.5
Mixture 5	2	bakelite	6.4
Mixture 6	3	none	5.7

It must be emphasized that OFC value depends on many factors and that it is not easy to compare the values obtained from experiments. Factors that affect OFC values are RAP granulometric composition, type of cement, type of bitumen emulsion, number of fine particles in RAP, amount and type of waste, and additional materials. Considering only the amount of cement as the parameter, it can be concluded that for 2% of cement, OFC is cca 6%. By comparing the results of our experiment and other available research, it can be concluded that the dominant influence on the OFC value has the partial replacement of cement with waste material itself and the type of used waste material.

4. Conclusions

Based on the presented experimental results of determining OFC in CIR that contained different amounts of Portland cement, as well as by-products, natural zeolite, and bakelite, the following conclusions can be drawn:

- Mixtures with 3% cement had the lowest optimal content (5.7%), while the mixture with the addition of fly ash had the highest one (7.6%) and the highest value of dry density (2.182 g/cm^3) in comparison with the remaining tested mixtures;
- The mixture containing bakelite had the lowest maximum dry density (2.123 g/cm^3) of all tested mixtures;
- With an increase in cement content from 2% to 3% percent, there was a decrease in OFC of cca 25%;
- The mixture with 2% cement and the mixture containing 20% natural zeolite as a partial replacement for cement, had the same OFC (6.5%) and approximately the same maximum dry density value;

- The mixture with slag had a slightly lower value of OFC (for cca 2%), while the mixture with fly ash had a higher value of OFC (for cca 17%) compared with the mixture with 2% cement;
- The mixture containing bakelite had the same OFC (6.4%) as the mixture with added slag and similar maximum dry density values.

The obtained results show that mixtures with fly ash require more liquid compared with other mixtures with 2% cement in order to obtain optimal compaction. The use of substitute waste materials reduces the cost of mixtures and, at the same time, does not lead to a significant change in the need for fluid content. In future experimental research, the plan is to change the percentages of fly ash, slag, and zeolite, as well as plastic, i.e., bakelite in CIR.

In this paper, a cement and bitumen emulsion was replaced with 20% waste materials. Future research will include varying the content of waste materials as a substitute for cement and emulsion. Replacement of cement with zeolite, fly ash, or slag could be carried out in the following percentages: 5%, 10%, 15%, 25%, and 30%. The same could be achieved with bakelite as a replacement for bituminous emulsion. Varying the content of replacement materials would show how changing their amount affects the optimal fluid content and mechanical properties. Also, the order of mixing the components proved to be a very important factor in certain scientific research that addresses the topic of cold recycling. Furthermore, the immobilization of potential heavy metals from waste materials and RAP in CIR will be examined and compared with the mixture containing zeolite as a well-known adsorbent.

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