



Szymon Węgliński \* D and Gabriel Martysz

Institute of Civil Engineering, Faculty of Civil and Transport Engineering, Poznań University of Technology, 5 Piotrowo Street, 61-138 Poznań, Poland

\* Correspondence: szymon.weglinski@put.poznan.pl

Abstract: In order to protect the natural resources, it is beneficial for the environment when materials that are not valuable, such as waste or recycled materials, are used in engineering. This article presents laboratory studies on the use of mixtures of incineration bottom ash (IBA) from municipal waste incinerators with natural, fine grain and uniform aggregate in cement-bound layers. The mechanical and engineering properties of aggregates were studied, their usefulness was assessed and possible applications are indicated. The chemical composition of the material was found to be consistent with typical IBA from other incineration plants, and leachability studies were carried out, confirming lack of any environmental impact. The authors' own mixtures were prepared based on optimal water content and maximal dry densities of solid particles, and the compressive strength was calculated after 7 and 28 days of hardening. The results indicate that replacing natural aggregates with IBA permits an increase in the compressive values for the specimens using the same amount of CEM I 42.5R while improving the frost resistance of cement–aggregate mixtures.

**Keywords:** MSWI bottom ash; cement stabilization; frost resistance; MSWIBA recycling; sustainable civil engineering



Citation: Węgliński, S.; Martysz, G. Utilization of Municipal Solid Waste Incineration Bottom Ash in Cement-Bound Mixtures. *Sustainability* **2024**, *16*, 1865. https://doi.org/10.3390/su16051865

Academic Editor: Pow Seng Yap

Received: 12 January 2024 Revised: 20 February 2024 Accepted: 22 February 2024 Published: 24 February 2024



**Copyright:** © 2024 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

In processes that relate to broadly understood environmental protection, it is important to strive for a closed cycle of raw materials, including the use of waste materials as input products for production of new materials or for energy generation.

One of the main issues related to the sustainable use of materials is the protection of natural resources. The basic raw materials in construction are natural aggregates, which include sand and other granular materials for rocks. They are the basic component of building materials.

Aggregate mining has a negative impact on the natural environment. The exploitation of aggregate deposits causes, for example, a decrease in groundwater, disruption of the functioning of ecosystems and the need to cut down forests. The diversification of the occurrence of deposits and their distance from the place of development or production plants requires transport, which causes air pollution as a result of the combustion of fossil fuels [1]. The average annual production of aggregates represents six tons per EU citizen [2].

Currently, there is a trend of using granular materials of non-natural origin. The use of recycled aggregates helps to save resources by reducing the extraction of primary raw materials and, moreover, also reduces the generation of waste. The construction sector is one of the largest producers of waste. Recycling of construction raw materials represents a great opportunity to recover inert waste instead of landfilling it [2]. Technical standards divide aggregates into natural, manufactured and recycled [3–6]. Their usefulness depends, among others, on the origin of the rock or its composition (in the case of manufactured and recycled aggregates), grain size and strength parameters. Some granular materials, such as fine sands, can be used for mortars or cement mixtures, but due to their fine grain size and

uniformity—characterized by poor compactibility (e.g., 0/2 mm sands), they cannot be used for embankments or layers of road structures [7], which means they are useless. The materials are stored in heaps and occupy a large area, and due to the presence of small dust fractions, in the wind, they can be moved and mixed with other materials, constituting contamination. Rational use of raw materials requires that they be used in a different way, e.g., as a material for granulating coarse aggregates.

An example of coarse aggregate, which is also an area of potential use of materials in waste management, is a bottom ash resulting from the incineration of municipal waste as part of the waste-to-energy (WTE) strategy. WTE is a widely accepted approach referring to the recovery of heat and electricity from non-recyclable waste, through incineration based processes [8].

In this article, the authors examined mixtures of natural and manufactured aggregates stabilized with Portland cement (CEM I). The tests used aggregates that are considered as waste materials. Natural aggregates were used—0/2 mm sand, which due to their fine and uniform grain size and low compaction (uniformity coefficient Cu < 3 and coefficient of curvature Cc < 1 [9]) are considered unsuitable for embankments and road structures. The second aggregate used is IBA derived from the incineration of municipal waste, the use of which is slowly being popularized and implemented due to the developing network of municipal waste incineration plants and ongoing research on the suitability of the material, taking into account the impact on the natural environment.

Our own pilot mixture proportions were proposed and the obtained compressive strength and frost resistance were compared with the results of typical cement-stabilized natural aggregate. The input materials are natural aggregates—sand 0/2 mm (40-70% share by weight) and sandy gravel 0/8 mm (0-100% share by weight) and manufactured aggregate IBA (30–60\% share by weight). As part of this scientific work, tests were carried out on input materials—IBA aggregate, natural aggregate and cement.

#### 2. Worldwide IBA Application and Polish Conditions

Thermal combustion generates various gaseous emissions and solid residues. These are mainly emissions of  $CO_2$  (carbon dioxide) as well as  $N_2O$  (nitrous oxide),  $NO_x$  (oxides of nitrogen),  $NH_3$  (ammonia) and organic C, measured as total carbon [10]. The two main types of by-products are fly ash (FA) and bottom ash (BA), which account for 80–85% of all residues [11]. Municipal solid waste incineration bottom ash (MSWIBA/IBA) is a porous, greyish and coarse gravel material containing primarily glass, ceramics, minerals, and ferrous and nonferrous materials, along with small amounts of unburned materials and organic carbon. The major compounds are oxides, hydroxides and carbonates. Various spectroscopic analyses have revealed that the main compounds (>10 wt.%) of IBA are SiO<sub>2</sub>, CaO, Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>, whereas Na<sub>2</sub>O, K<sub>2</sub>O, MgO and TiO<sub>2</sub> are present in minor concentrations (0.4–5.0 wt.%); thus, oxides prevail. SiO<sub>2</sub> is a predominant compound in IBA, accounting for up to 49 wt.% [12].

While municipal solid waste, prior to incineration in waste incineration plants, does not classify as hazardous waste; after incineration at a temperature of approx. 850–950 °C, mineral waste remains in which potentially hazardous substances exist. Raw waste is unstable in the natural environment and, therefore, must be treated to make it harmless or undergo stabilization before safe dumping at a landfill site [13].

The purpose of immobilization is to chemically transform hazardous waste into inert or non-hazardous waste in order to mitigate environmental impact in case of uncontrolled infiltration of diluted (leached) compounds [8,14].

Natural weathering is the most cost-effective stabilization treatment method since it results in the chemical stability of IBA. A suitable period of natural weathering of freshly quenched IBA causes chemical and mineralogical changes that lead to neoformed phases and minerals that are thermodynamically more stable and less reactive [15]. One- to three-month exposure to natural weathering is enough to reduce the release of heavy metals from the residue. In this period, some of the chemical and mineralogical characteristics of

the material undergo significant changes, including oxidation of some metals (e.g., Al, Fe, Cu), dissolution and precipitation of the hydroxides and salts of the main cations (e.g., Ca, Al, Na, K), carbonation, neutralization of pH and neoformation of clay-like minerals from glass [16]. Physically, weathering allows the moisture contained in the air to penetrate into the grains, where hydration processes take place. The hydration process involves attaching water to the chemical compounds contained in IBA.

After ageing and stabilization in the open air, the material can be reused as manufactured aggregate. In many countries around the world, combustion products are reused in construction. Especially in road construction, IBA can be used for the following layers as an important alternative allowing conservation of natural aggregates and other natural materials [17,18]:

- Unbound layers—as filler and sub-base material for embankments and base courses [19–24],
- Hydraulically bound layers—for road and floor bases and road structures—as a concrete ingredient [25–29], as well as a substitute for cement [26,30],
- Bituminous layers—added to bituminous mixtures used on local roads [31,32].

Researchers [33] point to the following facts that must be taken into account when deciding to use incineration ashes in construction:

- Bottom ash from municipal solid waste incineration is a material so highly heterogeneous and variable that the results reported cannot guarantee the behavior of the bottom ash obtained under various conditions; it is necessary to continuously control its main chemical and engineering properties,
- Stricter management of selective collection could give rise to a significant change in the composition of the bottom ash, thus, a reduction in the amount of incinerated glass would reduce the bottom ash volume, of which glass is the main component; the resulting bottom ash would mainly become fine dust, thus limiting the application range of the material.

According to the European Directives [34–36] and Polish regulations [37–39], IBA has the status of waste material of 19 01 group: Waste from waste incineration plants, including waste pyrolysis plants and precisely:

- 19 01 11\* Bottom ash and slag containing hazardous substances,
- 19 01 12 Bottom ash and slag other than those mentioned in 19 01 11.

The report, "Secondary materials. Artificial aggregates. Final Report on aggregates obtained from secondary resources (CEN/TC154/TG10/N736)" [40] classifies IBA to the group "B1"—ashes from municipal waste incineration furnaces, excluding fly ashes [41].

In Poland, the allowable composition of ash produced from the incineration of municipal solid waste is defined by the Technical Requirements WT-4 [42].

To get non-hazardous waste status, ash must pass the H1 (explosive property) test, H13 (neutralization) and H14 (ecotoxicity) tests, followed by the "End of waste" procedure when the ash either loses or maintains its waste status. In the first case, IBA becomes a product and now it must meet the REACH requirements in order to be placed on the market. If waste status is maintained, the material may be disposed or used as a secondary aggregate, in accordance with national regulations. Following treatment, ash can be returned to the environmental cycle in the form of, among other things, artificial aggregate or cement filler. Alternative aggregate in the form of IBA has a significant potential, especially with a view to protecting natural materials deposits.

In 2021, 13,674,000 tons of municipal waste were generated in Poland, 4.2% more compared to 2020. This means an increase in the per capita amount of municipal waste generated in Poland from 344 kg in 2020 to 360 kg in 2021 [43]. A part of this amount is recycled, part is composted or fermented, and still another part is landfilled or thermally treated. The percentage shares of the specific waste management methods used in the years 2017–2021 are shown in Table 1.

Year	wt (%)					
	Recycling	Composting	Incineration	Landfilling		
2017	27	7	24	42		
2018	26	8	24	42		
2019	25	9	23	43		
2020	27	12	22	40		
2021	27	13	21	39		

Table 1. Municipal waste management in Poland in 2017–2021 [43].

Significant waste recycling is at a constant level (mean value 26.4%), and the level of composted materials is increasing (almost twice in 2017 and 2013). There are slight decreases in waste that is thermally transformed and in landfills. It is worth making thermal combustion more effective and contributing to the reduction of stored materials that are not safe for the environment. The added value is the possibility of energy recovery as a result of combustion, even in the amount of 10% of the current demand [44].

To sum up the 2020 statistics, thermal treatment facilities disposed of 1,069,000 Mg of waste [45]. Assuming that each Mg of municipal waste produces about 240 kg of IBA, it can be assumed that the incineration processes generated about 257,000 Mg of IBA, which is available for reuse. The maximum throughput capacity of thermal treatment plants located in Poland is shown in Figure 1. All values are in thousand Mg [46].



Figure 1. Maximum efficiency of incineration installations by voivodeship (in thousands of Mg).

The authors suggest the high potential of application of IBA as an aggregate for layers of improved subsoil or mixtures stabilized with binders, as it is used in various countries around the world. Referring to the researchers' comments [33] regarding variable parameters and composition, the obtained aggregates should be carefully controlled by verifying and standardizing the input materials. Due to the relatively young market of incineration plants in Poland and their development, as well as the difficulty of ensuring a homogeneous stream of municipal waste, coherent national regulations should be developed regarding the minimum share of individual types of waste subject to thermal incineration.

According to the authors, the main target of application of IBA should be focused on the road engineering in order to prevent contact of harmful materials with people, e.g., through additives to building materials used in buildings or other places where people stay. The only risk of using IBA aggregate in road construction is the possibility of penetration (leaching) of harmful substances into the soil and groundwater; therefore, the authors proposed the possibility of stabilization with binders to limit the flow of water and the ability of harmful substances to penetrate into the environment by binding them with cement, which creates a homogeneous, compact material. An additional advantageous aspect is the possibility of using fine, homogeneous aggregates, which are often treated as waste material as a fine filler material for coarse aggregate.

### 3. Materials and Methods

# 3.1. Materials

### 3.1.1. Cement

The cement analyzed for the purposes of this research was Portland CEM I 42.5R, produced by GORAZDZE CEMENT (Poland), due to the fact that the binder tested according to the requirements of the standard [47] does not have any additives (blast furnace slags, fly ashes, limestones, burnt shales, etc.) that may affect the properties of stabilized mixtures.

### 3.1.2. Natural Aggregates

Two types of natural aggregate were used in the research:

- Sand 0/2 mm—often considered as a material unsuitable for embankments and stabilization layers due to poor compactibility, which was selected as the material for IBA granulation, marked as  $S_0/2$  and graphically as a blue line (see Section 4),
- Sandy gravel 0/8 mm—a material useful for embankments and for stabilization with binders, selected as a comparative material, marked as RS and graphically as a purple line (see Section 4).

## 3.1.3. IBA

The municipal solid waste incineration bottom ash was taken from the municipal solid waste incineration plant in Bydgoszcz, Poland. Composition tests according to [42] showed that it is a light grey aggregate, composed of incineration bottom ash (96%) and crushed glass fragments (4%) of 0/8 mm in size. Sieve analysis performed in accordance with [48] allowed for the drawing of the grain structure curve (which is presented in Section 4 as a green line). Figure 2 shows IBA micrographs taken under a binocular magnifier  $(15-50\times)$ .





Figure 2. IBA binocular analysis: (a) ceramics, (b) glass, (c) fibrous material.

The tested IBA is characterized by varied granulation and heterogeneity of composition. Variation in fractions and material causes caking. Hard combustion products, ceramics, glass and fibrous materials can be observed.

#### 3.2. Research Experiment, Mixture Design and Sample Preparation

As a part of a pilot analysis of the application of IBA and sand 0/2 mixtures for cement-bound layers, the following tests were carried out:

- Assessment of the chemical composition of IBA—chemical leaching tests and elemental analysis using energy dispersive X-ray spectroscopy with application of a TESCAN VEGA 3 scanning electron microscope with the Bruker EDX attachment for elemental analysis were programmed,
- Original proportions of mixture ingredients were proposed, resulting from practical experience, based on tests of the natural water content of the mixed material and the determination of the optimal water content to obtain the maximum optimal dry density of solid particles of the aggregate—using the Proctor method [49],
- Three series of samples were prepared, differing in the content of natural aggregate and IBA. For each mixture, three different amounts of cement were analyzed (variable by 2 percent) in order to assess the compressive strength and frost resistance of the proposed mixtures,
- Comparative tests were performed for a mixture of useful natural aggregate used for cement-bounded layers in road engineering applications.

Three new mixtures with different contents of mineral material (sand) and IBA were designed and tested to determine their properties. They were marked as follows:

- 60\_IBA/40\_S with 60% IBA and 40% mineral material (sand 0/2) content,
- 45\_IBA/55\_S with 45% IBA and 55% mineral material (sand 0/2) content,
- 30\_IBA/70\_S with 30% IBA and 70% mineral material (sand 0/2) content.

The research procedure was carried out according to the algorithm specified in [50]. The quantities of mixture components were determined according to laboratory formulas. The specimens were compacted in cylindrical molds of equal height and 80 mm diameter. The specimens were made by dynamic compaction using the Standard Proctor method, at the optimum soil–cement moisture content [49]. After demolding, the specimens were cured, depending on the curing time, in moist sand (Figure 3a) and then in water (14 days, see Figure 3b). Compressive strength was determined after 7 and 28 days of curing [50].

The freeze-thaw resistance test involved subjecting the specimens to 14 cycles of freezing and thawing. A single freezing cycle consisted of eight hours of freezing at -23 °C (Figure 3c) and sixteen hours of thawing in water at room temperature [50]. An automatic chamber was used for the tests with automatic change of cycles, including temperature. The compressive strength of the frozen specimens was determined after 28 days of curing.

# 3.3. SEM/EDS Analysis

The analysis of scanning electron microscopy with energy dispersive X-ray spectrometry (SEM/EDS) was used in the determination of the IBA chemical composition. When SEM is combined with elemental microanalysis EDX, it has the advantage that an insight into the elemental distribution of the sample can be directly related to a visual image of the assessed specimen [51].

The IBA material, introduced into the vacuum chamber of the microscope, will be exposed from above with an electron beam. The electron beam when hitting the tested surface is scattered deep into the material and can ionize the sample atoms, knocking secondary electrons from stationary coatings. The gaps formed on the electron coatings are filled rapidly by electrons from the outer atom coatings. Ionized atoms emit X radiation quantum with discrete energy values, characteristic of the elements found in the sample material. Photons of the characteristic X-ray radiation are collected by the EDS detector, placed above the sample, and transmitted by the electronic system to a multi-channel analyzer, in which the impulses are separated depending on the energy. The number of quantum (intensity) of the characteristic X radiation emitted by the tested element within the given time period is proportional to the content of this element in the sample [52].





(**b**)



(c)

**Figure 3.** Specimen care of samples: (**a**) merged in wet sand, (**b**) fully submerged in water, (**c**) during the freezing cycle.

# 4. Results and Discussion

# 4.1. Compressive Strength of Cement

As part of the tests, cement prisms ( $40 \times 40 \times 160$  mm) were prepared in accordance with [53] and the compressive strength was determined. The results are shown in Table 2.

Compressive strength tests were carried out after two hardening periods—after 2 and 28 days. The obtained results allow us to conclude that the tested samples meet the requirements for class 42.5R (2 days: >20 MPa and 28 days: 42.5–62.5 MPa) [40].

Test after 2 Days of Curing			Test after 28 Days of Curing			
Specimen No.	Compressive Load (kN)	Compressive Strength Value (MPa)	Specimen No.	Compressive Load (kN)	Compressive Strength Value (MPa)	
1a	34.99	21.9	4a	70.29	43.9	
1b	33.09	20.7	4b	70.26	43.9	
2a	33.51	20.9	5a	70.51	44.1	
2b	35.59	22.2	5b	69.70	43.6	
3a	34.73	21.7	6a	71.64	44.8	
3b	31.93	20.0	6b	71.96	45.0	
	Average:	$21.2\pm0.86$		Average:	$44.2\pm0.55$	

Table 2. Results of cement performance test.

### 4.2. Sieve Analysis of Aggregates

Sieve analysis was performed in accordance with the standard [48]. The gradation of the natural aggregates and IBA is shown in Figure 4.



Figure 4. Granular structure curve of analyzed materials.

The test results allowed determination of the grain size of aggregates (necessary for the preparation of cement–aggregate mixtures) and their compaction. Sand (S\_0/2) is characterized by low compactibility ( $C_U = 1.8$ ) and sandy gravel (RS) is characterized by favorable compactibility ( $C_U = 3.2$ ).

#### 4.3. Chemical Analysis of IBA

Firstly, microscopic viewing was performed with application of a scanning electron microscope (SEM). The results are shown in Figure 5. To assess the chemical composition, leaching tests were performed (in accordance with the standards [54–62]) and elemental analysis was performed using X-ray spectroscopy with energy dispersion for elemental analysis. The results are shown in Tables 3 and 4.



Figure 5. SEM view using EDX analysis, with three location of chemical composition tests.

Parameter	Leaching Values (mg/kg)	Accuracy (mg/kg)	Threshold Values set by Polish Legislation [62] (mg/kg)
As	< 0.01	$\pm 0.002$	<2
Ba	0.085	$\pm 0.017$	<100
Cd	< 0.005	$\pm 0.0001$	<1
Cr	0.45	$\pm 0.11$	<10
Cu	0.23	$\pm 0.05$	<50
Hg	< 0.005	$\pm 0.0012$	<0.2
Mo	0.32	$\pm 0.08$	<10
Ni	0.18	$\pm 0.04$	<10
Pb	<0.01	$\pm 0.003$	<10
Sb	<0.01	$\pm 0.002$	<0.7
Se	<0.01	$\pm 0.002$	<0.5
Zn	0.36	$\pm 0.09$	<50
H <sub>2</sub> O	<1.0	$\pm 0.1$	-
Carbon	49.8	$\pm 10.5$	<800
Chloride—Cl	700	$\pm 140$	<15,000
Fluorides—F	<1.0	$\pm 0.2$	<150
Sulphureous—SO <sub>4</sub>	2800	$\pm 560$	<20,000
TDS	6800	$\pm 1020$	<60,000
pH (20 °C)	7.2	$\pm 0.2$	-

Table 3. Leaching tests of analyzed IBA.

The results of leachability tests confirm the presence of various chemical compounds: chlorides, fluorides and sulphates, as well as heavy metals. The composition of the tested specimen is comparable to various IBAs presented in Section 1. According to the authors, the possibility of chemical stabilization of IBA aggregates with cement will limit the possible penetration of harmful compounds into the soil and groundwater during water flow through the cement-bound mixtures.

	Test No. 1	Test No. 2	Test No. 3
Element		Value: Wt (%)	
Oxygen	15.2	59.1	36.9
Iron	79.0	0.7	0.9
Magnesium	2.0	40.2	6.3
Manganese	2.5	-	-
Silicon	1.3	-	42.4
Calcium	-	-	1.5
Aluminum	-	-	1.2
Carbon	-	-	10.8

Table 4. EDX elemental analysis.

During triplicate testing in elemental analysis using EDX, in test no. 1 (see Figure 6), a significant presence of iron was noticed, and there was also oxygen, gold, manganese, silicon, magnesium and palladium. Test no. 2 (see Figure 7) was dominated by the presence of magnesium and oxygen, with traces of iron, gold and palladium. In the case of test no. 3 (see Figure 8), large amounts of silicon, oxygen and carbon were found, as well as additions of magnesium, calcium, aluminum, gold and palladium. The research confirmed a heterogeneity of the substances contained in IBA—based on a selected single aggregate sample, three X-ray spectroscopic analysis measurements showed a significant diversity of elements depending on the test location (see Figure 5).

#### 4.4. Analysis of the Optimal Water Content and Dry Density of Solid Particles

The proposed mixtures were prepared from a combination of natural aggregate sand 0/2 mm and IBA 0/8 mm. The gradation of the natural aggregates and IBA mixtures are shown in Figure 9.



Figure 6. Elemental composition—quantitative analysis test no. 1.



Figure 7. Elemental composition—quantitative analysis test no. 2.



Figure 8. Elemental composition—quantitative analysis test no. 3.

The recipes for cement-stabilized mixtures, which determine the mass of the input aggregate, binder and water, were developed based on the results of tests using the Proctor method, in which the optimal water content and maximum dry density of solid particles were determined. The list of components is presented in Table 5. Selected graphs obtained based on the results of the Proctor method for 30\_IBA/70\_S mixtures are shown in Figure 10. A summary of the optimal water contents and obtained maximum dry densities of solid particles for all mixtures is shown in Figures 11 and 12.



Figure 9. Granular structure curves of analyzed mixtures of IBA and sand 0/2.

Notation of Mixture	Water Content	Optimal Water Content	Max. Dry Density	Dry Mass of Aggregates	Ma of Ce	ass ement	Mass of Water
	(%)	(%)	(g/cm <sup>3</sup> )	(g)	(%)	(g)	(g)
RS_5C		11.4	1.882	8 360	5.0	418	361
RS_7C	7.65	11.0	1.902	8 360	7.0	585	344
RS_9C		10.8	1.925	8 360	9.0	752	344
60_IBA/40_S/5C		12.9	1.822	8 536	5.0	427	693
60_IBA/40_S/7C	5.43	12.0	1.818	8 536	7.0	598	633
60_IBA/40_S/9C		12.1	1.835	8 536	9.0	768	662
45_IBA/55_S/5C		12.5	1.809	8 509	5.0	425	626
45_IBA/55_S/7C	5.77	11.8	1.802	8 509	7.0	596	584
45_IBA/55_S/9C		11.4	1.811	8 509	9.0	766	567
30_IBA/70_S/5C		12.2	1.799	8 482	5.0	424	480
30_IBA/70_S/7C	6.11	11.5	1.776	8 482	7.0	594	535
30_IBA/70_S/9C		10.8	1.773	8 482	9.0	763	481

Table 5. Mixture components.





Figure 10. Water content and dry densities of solid particles for 30\_IBA/70\_S mixtures.



Figure 11. Summary of the optimal water content of analyzed mixtures depending on the amount of cement.



**Figure 12.** Summary of maximum dry density of solid particles of analyzed mixtures depending on the amount of cement.

The natural water content was determined for each mixture, and then the necessary amount of water was added to obtain the optimal water content during forming and compacting the specimens.

As the amount of cement increases, the optimal water content of the proposed mixtures (excluding 60\_IBA/40\_S) and the comparative aggregate (sandy gravel, RS) decreases. Mixtures resulting from the combination of IBA and sand 0/2 show higher (and in one case equal) optimal water content with the same level of cement admixture than the comparative mixture, which may mean that the IBA content, which is a more porous material than natural aggregate, requires sufficient amount of additional water. With a lower IBA content in the mixtures, the optimal water content decreases (although it is still higher than in the case of the comparison mixture without the application of IBA).

Mixtures based on IBA are characterized by lower values of maximum dry density of solid particles than the reference mixture (sandy gravel RS). The situation is related to the density of the input materials (fine sand 0/2 and IBA, which is characterized by low density due to high porosity). The trend is correct for all tested mixtures. It should be noted that mixtures with 5% and 7% binder added showed lower densities than the same mixtures with 9% addition. The change in tendency may be related to the increase in the

### 4.5. Compressive Strength

The obtained compressive strength and freeze–thaw resistance values are summarized in Table 6. Frost resistance index (FRi), according to [50], is the ratio of the compressive strength values measured after a period of 28 days of hardening: specimens subjected to freezing in relation to reference specimens which were immersed in water.

amount of natural aggregate in the stabilized mixture. In the case of the reference mixture, an increase in the amount of binder increases the maximum dry density of solid particles.

**Compressive Strength after: Frost Resistance Index** Notation of Mixture 28 Days: (FRi) 28 Days 7 Days 14 Days + 14 Freezing MPa MPa MPa RS\_5C  $0.62\pm0.04$  $0.36\pm0.17$ 0.30  $1.21\pm0.12$ 60\_BA/40\_S/5C  $1.79\pm0.11$  $3.59 \pm 0.63$  $3.22 \pm 0.50$ 0.90 45\_BA/55\_S/5C  $1.77\pm0.20$  $2.89 \pm 0.04$  $2.51\pm0.23$ 0.8730\_BA/70\_S/5C  $1.27 \pm 0.13$  $2.63\pm0.29$  $2.11 \pm 0.09$ 0.80 RS\_7C  $1.53 \pm 0.16$  $2.93\pm0.25$  $2.53\pm0.22$ 0.86 60\_BA/40\_S/7C  $3.37\pm0.22$  $5.22\pm0.21$  $3.29\pm0.21$ 0.63 45\_BA/55\_S/7C  $2.82 \pm 0.56$  $5.08 \pm 0.33$  $4.74 \pm 0.14$ 0.93 30\_BA/70\_S/7C  $3.21\pm0.34$  $4.42\pm0.26$  $3.91\pm0.31$ 0.88 RS\_9C  $2.62\pm0.14$  $5.21 \pm 0.19$  $4.52\pm0.53$ 0.87 60\_BA/40\_S/9C  $5.01\pm0.65$  $5.38 \pm 0.64$  $4.40\pm0.56$ 0.82 45\_BA/55\_S/9C  $4.62\pm0.11$  $7.09 \pm 0.10$  $6.64\pm0.08$ 0.94 30\_BA/70\_S/9C  $3.69 \pm 0.15$  $7.50\pm0.09$  $7.42\pm0.15$ 0.99

Table 6. Compressive strength of specimens with the addition of cement.

The cement-stabilized reference soil mixtures achieved significantly lower compressive strength values than the mixtures including the tested mixtures of IBA with sand. Particular attention should be paid to the specimens subjected to the freeze-thaw resistance test.

Among the results obtained, for 7 day specimens, the standard deviation of the mean is in the range of 2.4–19.9%, with an average of 8.9%. For 28 day samples, the standard deviation is 1.2–17.5%, with an average of 6.9%. For samples after the freeze–thaw test, the standard deviation is 1.2–47.2%, with an average of 10.8%. Particularly significant differences were found in the case of specimens after freezing resulting from the low compressive strength of the reference mixture, which failed prematurely.

For the sake of simplicity of the analysis of results, a comparative compressive strength index was proposed, where standardized values (equal to 1.0) were determined for the reference mixture. Each value of the obtained compressive strength is related to the value obtained by the reference mixture in the analyzed cycle considered. The values obtained for the respective mixtures containing an addition of IBA were calculated relative to the values for the reference soil (Figures 13–15).



Figure 13. The comparative compressive strength index with 5% of cement.



Figure 14. The comparative compressive strength index with 7% of cement.



Figure 15. The comparative compressive strength index with 9% of cement.

#### 4.6. Early Compressive Strength Standardization of Results

On evaluation after 7 days of curing, the specimens containing IBA achieved at least 40% higher compressive strength for a binder addition of 9%, and even nearly three times higher compressive strength for cement addition of 5% than the soil specimens without IBA. As can be seen, the greater the amount of ash in the mixture, the more favorable the comparative index, which indicates that IBA increases the skeleton strength and bearing capacity. In practice, the recommended curing and hardening time (during which the layer cannot be loaded, usually at least 3–7 days) may be shortened due to the higher compressive strength and load-bearing capacity. Therefore, it is possible to load the completed layer in advance, e.g., with the traffic of construction vehicles. In the case of the reference mixture, due to lower strength values, the load on the layer may cause damage and destruction of the load-bearing structure of the mixture, which will contribute to weakening the final stiffness of the layer structure.

#### 4.7. Equivalent Compressive Strength Standardization of Results

The specimens tested after 28 days of curing, similarly to the 7 day specimens, achieved significantly higher strength values than the reference mixtures. In the case of the addition of 5% cement, the compressive strength increased by 2.2–3.0 times and with the addition of 7% cement—by 1.5–1.8 times. Higher values were achieved for mixtures in which more than half of the composition was IBA. The results obtained for the mixtures containing IBA and 9% cement content showed strength improvement ranging from 3% (for 60% IBA addition) to 44% (for 30% IBA addition). As can be seen, the difference between 30% and 60% IBA content is not very significant for samples with a binder addition of at least 7%.

#### 4.8. Standardization after Freeze–Thaw Resistance Test

A significant, at least 5 times improvement, in compressive strength was obtained for specimens stabilized with the lowest, i.e., 5%, cement addition. An almost 9 times improvement was observed in the case of the highest proportion of IBA in the mixture ( $60_BA/40_S$ ). For the specimens with 7% CEM, a 40–87% improvement in the compressive strength was noted. For mixtures with 9% binder addition, improvement of ca. 50–65% was observed for the mixtures with IBA content of 45% and 30% by weight. In the case of a high amount of cement addition (>7%), the application of IBA as a replacement aggregate is not justified. The reference mixture based on natural aggregate achieves comparable compressive strength values as mixtures with IBA. Varying the amount of IBA does not have a significant impact on the results obtained.

#### 4.9. Freeze-Thaw Resistance Values

The reference specimens achieved very low compressive strength when stabilized with 5% cement addition. The literature [50] states that non-cohesive soils are fully frost resistant, however, a small addition of cement (e.g., 5% shown in the research conducted) does not ensure the required frost resistance (FRi value of at least 0.6). Specimens with low compressive strength are susceptible to frost damage, which in practice may result in loss of stiffness and premature failure and destruction of structural layers.

The values obtained for the other reference mixtures were in the order of 0.87. In seven out of nine cases, the mixtures containing IBA achieved higher, i.e., more desirable values, than the reference mixtures. Two cases with lower values were the mixtures with 60% IBA content.

With regard to the study in [63], in which the researchers stabilized bottom ash with 2–10% cement I content, replacing 40% and 70% natural aggregate with ash gave approximately 5 times lower compressive strength for specimens after 7 and 28 days of curing. It must be noted, however, that 0/20 mm aggregate was used in these tests, which had a considerable effect on the obtained values, due to the load-bearing capacity and hardness of the coarse grains contained in the mixture.

### 5. Conclusions

This article focuses on the application potential of aggregates, which in practice are considered as unsuitable for road engineering—uniformly fine sand 0/2 mm and manufactured material from municipal waste incineration, i.e., bottom ash.

The authors proposed application of sand 0/2 as a filler for IBA. The prepared mixtures were stabilized with Portland cement CEM I 42.5R. The mixtures proposed in this study had 30%, 45% and 60% of natural aggregate (sand) replaced by ash. These mixtures were tested for freeze–thaw resistance and compressive strength as part of the study.

Reuse of waste materials is beneficial to the natural environment conservation. IBA leachability studies demonstrated that the amount of the harmful substances released as a result does not exceed the allowable limits and does not pose an environmental hazard. Stabilization of IBA with cement appears to be useful, as it will significantly mitigate the risk of leaching harmful substances from the bound layers into soil. Below are the main findings of this study:

- (1) The analyzed municipal waste incineration ash from Bydgoszcz as a result of leaching test contains magnesium, calcium, iron and aluminum oxides, which is typical of furnace bottom ash, which is known from other research. EDX studies showed heterogeneity of this material; specifically, variable aggregate composition demonstrated in a three-fold test of a single sample (at different testing locations) that different concentrations of the same and variable basic elements were found.
- (2) Optimal water content for IBA and sand 0/2 mixtures is higher than reference mixtures, regardless of the amount of cement admixture.
- (3) Based on density of IBA, the maximum dry density of solid particles decreases as the amount of IBA decreases. For the three cement additions, comparable maximum density values were found without significant differences due to the increase in the amount of binder. In the case of the reference mixture, the maximum density increases with the increase in the amount of cement.
- (4) IBA addition significantly improved the freeze-thaw resistance of the analyzed mixtures, which is especially true for the 5% addition of CEM I 42.5R cement in relation to reference sandy gravel, which, when stabilized with cement, would not ensure the freeze-thaw resistance required by the standards (>0.6). All the IBA containing mixtures achieved freeze-thaw resistance index values above 0.63, which passes the technical standards for mixtures for road construction layers in Poland.
- (5) The reference cement-stabilized soil mixture achieved a compressive strength value representative of treated subgrade layers given in [50]. With the same CEM I addition of approx. 8.5%, the 45\_R/55\_S and 30\_R/70\_S mixtures including an IBA addition achieved compressive strengths corresponding to the classes for paving grade road base mixes (high quality of compressive strength and frost resistance).
- (6) With an increasing quantity of IBA in the mix, a smaller improvement in compressive strength is observed.

**Author Contributions:** Conceptualization, S.W. and G.M.; methodology, S.W.; software, S.W.; validation, S.W. and G.M.; formal analysis, G.M.; investigation, S.W.; resources, G.M.; data curation, S.W. and G.M.; writing—original draft preparation, S.W.; writing—review and editing, G.M.; visualization, S.W.; supervision, G.M.; project administration, S.W. and G.M. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by the Polish Ministry of Science and Higher Education under grant 0413/SBAD/5447.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

**Data Availability Statement:** The data presented in this study are available on request from the corresponding author.

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

### References

- Babiak, M.; Węgliński, S.; Kosno, J. Regeneration of bituminous pavements with modifier received from the raw organic material. In *Revitalization of Urbanized Areas*, 1st ed.; Buczkowski, W., Szymczak-Graczyk, A., Eds.; PZITB: Poznań, Poland, 2016; pp. 431–444.
- 2. Tost, M.; Ammerer, G. Sustainable Supply of Aggregates in Europe; Final Report 12/2022; Montanuniversität Leoben: Leoben, Austria, 2022.
- 3. EN 12620:2002+A1:2008; Aggregates for Concrete. CEN: Brussels, Belgium, 2008.
- 4. *EN 13043:2013;* Aggregates for Bituminous Mixtures and Surface Treatments for Roads, Airfields and Other Trafficked Areas. CEN: Brussels, Belgium, 2013.
- 5. EN 13139:2013; Aggregates for Mortar. CEN: Brussels, Belgium, 2013.
- 6. *EN 13242:2013;* Aggregates for Unbound and Hydraulically Bound Materials for Use in Civil Engineering Work and Road Construction. The Polish Committee for Standardization: Warsaw, Poland, 2010.
- 7. Węgliński, S. Evaluation of the suitability of cement-stabilized even-grained soils for road engineering. *Przegląd Bud.* 2024; 1–2, *in press.*
- 8. Sofi, M.; Sabri, Y.; Zhou, Z.; Mendis, P. Transforming Municipal Solid Waste into Construction Materials. *Sustainability* **2019**, *11*, 2661. [CrossRef]
- 9. *EN ISO 14688-2:2018;* Geotechnical Investigation and Testing. Identification and Classification of Soil. Part 2: Principles for a Classification. CEN: Brussels, Belgium, 2018.
- 10. Johnke, B. Waste incineration—An important element of the integrated waste management system in Germany. *Waste Manag. Res.* **1992**, *10*, 303–315. [CrossRef]
- 11. Seniunaite, J.; Vasarevicius, S.; Grubliauskas, R.; Zigmontiene, A.; Vaitkus, A. Characteristics of Bottom Ash from Municipal Solid Waste Incineration. *Annu. Set Environ. Prot.* **2018**, *20*, 123–144.
- 12. Cho, B.H.; Nam, B.H.; An, J.; Youn, H. Municipal Solid Waste Incineration (MSWI) Ashes as Construction Materials—A Review. *Materials* **2020**, *13*, 3143. [CrossRef]
- 13. Kukielska, D. Aggregates from waste materials. Comprehensive management of municipal and industrial waste. *Aggreg. Prod. Transp. Appl.* **2017**, *3*, 17–19.
- 14. Mikuła, J.; Łach, M.; Mierzwiński, D. Utilization methods of slags and ash from waste incineration plants. *Ecol. Eng.* **2017**, *18*, 37–46. [CrossRef]
- 15. del Valle-Zermeno, R.; Chimenos, J.M.; Giro-Paloma, J.; Formosa, J. Use of weathered and fresh bottom ash mix layers as a subbase in road constructions: Environmental behavior enhancement by means of a retaining barrier. *Chemosphere* **2014**, 117, 402–409. [CrossRef] [PubMed]
- 16. Chimenos, J.M.; Fernández, A.I.; Miralles, L.; Segarra, M.; Espiell, F. Short-term natural weathering of MSWI bottom ash as a function of particle size. *Waste Manag.* 2003, 23, 887–895. [CrossRef] [PubMed]
- 17. Chen, D.; Zhang, Y.; Xu, Y.; Nie, Q.; Yang, Z.; Sheng, W.; Qian, G. Municipal solid waste incineration residues recycled for typical construction materials—A review. *RSC Adv.* 2022, *12*, 6279–6291. [CrossRef]
- 18. Lynn, C.J.; Ghataora, G.S.; Dhir, R.K. Municipal incinerated bottom ash (MIBA) characteristics and potential for use in road pavements. *Int. J. Pavement Res. Technol.* **2017**, *10*, 185–201. [CrossRef]
- Hu, Y.; Li, G.; Zhong, Y. Utilization of municipal solid waste incineration bottom ash as road construction materials. In Proceedings of the International Conference on Mechanic Automation and Control Engineering, Wuhan, China, 26–28 June 2010; pp. 1370–1373.
- 20. Kraszewski, C. Technical and Chemical Properties of Slags from Municipal Waste Incineration (IBA) and the Possibilities of Their Use in Road Construction; Road and Bridge Research Institute: Warsaw, Poland, 2014.
- 21. Saveyn, H. (Ed.) Study on Methodological Aspects Regarding Limit Values for Pollutants in Aggregates in the Context of the Possible Development of End-of-Waste Criteria under the EU Waste Framework Directive; European Commission: Luxembourg, 2014.
- 22. Abdulahi, M.M. Municipal solid waste incineration bottom ash as road construction material. AU JT 2009, 13, 121–128.
- 23. Izquierdo, M.; Vazquez, E.; Querol, X.; Barra, M.; Lopez, A.; Plana, F. Use of bottom ash from municipal solid waste incineration as a road material. *Int. Ash Util. Symp.* 2001, *4*, 32–38.
- 24. Vaitkus, A.; Skultecke, J.; Sernas, O. A Test Road with Unbound Base and Sub-Base Course from MSWI Bottom Ash Mixtures. *Buildings* 2023, *13*, 1311. [CrossRef]
- 25. Shi, C.; Li, J.; Sun, S.; Han, H. Research on Pavement Performance of Cement-Stabilized Municipal Solid Waste Incineration Bottom Ash Base. *Materials* **2022**, *15*, 8614. [CrossRef]
- Keppert, M.; Reiterman, P.; Pavlik, Z.; Pavlikova, M.; Jerman, M.; Cerny, R. Municipal solid waste incineration ashes and their potential for partial replacement of Portland cement and fine aggregates in concrete. *Cem. Lime Concr.* 2010, 15, 187–193.

- 27. Sikora, J.; Niemiec, M.; Szeląg-Sikora, A.; Kuboń, M.; Mruk, B.; Marczuk, A. Use of slag from thermal treatment of municipal waste for concrete purposes. *Przemysł Chem.* **2019**, *98*, 1104–1107. [CrossRef]
- 28. Lynn, C.J.; Dhir, R.K.; Ghataora, G.S. Municipal incinerated bottom ash characteristics and potential for use as aggregate in concrete. *Constr. Build. Mater.* **2016**, 127, 504–517. [CrossRef]
- 29. Tang, Q.; Gu, F.; Chenm, H.; Lu, C.; Zhang, Y. Mechanical evaluation of bottom ash from municipal solid waste incineration used in roadbase. *Adv. Civ. Eng.* **2018**, *2018*, 5694908. [CrossRef]
- Czop, M.; Łaźniewska-Piekarczyk, B. Use of Slag from the Combustion of Solid Municipal Waste as A Partial Replacement of Cement in Mortar and Concrete. *Materials* 2020, 13, 1593. [CrossRef] [PubMed]
- 31. Chen, J.S.; Chu, P.Y.; Chang, J.E.; Lu, H.C.; Wu, Z.H.; Lin, K.Y. Engineering and environmental characterization of municipal solid waste bottom ash as an aggregate substitute utilized for asphalt concrete. *J. Mater. Civ. Eng.* **2008**, *20*, 432–439. [CrossRef]
- 32. Gress, D.; Zhang, X.; Tarr, S.; Pazienza, I.; Eighmy, T. Physical and environmental properties of asphalt-amended bottom ash. *Transp. Res. Rec.* **1992**, *1345*, 10–18.
- 33. Forteza, R.; Far, M.; Segui, C.; Cerda, V. Characterization of bottom ash in municipal solid waste incinerators for its use in road base. *Waste Manag.* 2004, 244, 899–909. [CrossRef] [PubMed]
- 34. European Union. Directive 2008/98/EC of the European Parliament and of the Council of 19 November 2008 on waste and repealing certain Directives. *Off. J. Eur. Union* 2008, *312*, 3–30.
- 35. European Union. Directive 2018/851 of the European Parliament and of the Council of 30 May 2018 amending Directive 2008/98/EC on waste. *Off. J. Eur. Union* 2018, 150, 109–140.
- 36. European Union. Commission Implementing Decision 2019/2010 of 12 November 2019 establishing the best available techniques (BAT) conclusions, under Directive 2010/75/EU of the European Parliament and of the Council for waste incineration. *Off. J. Eur. Union* **2019**, *312*, 55–91.
- 37. Regulation of the Minister of Development of 22 January 2016 on the requirements for conducting the thermal transformation of waste and methods of dealing with waste generated as a result of this process. J. Laws 2016, 108, Warsaw 2016.
- 38. Regulation of the Minister of the Environment of 9 December 2014 on the waste catalogue. J. Laws 2014, 1923, Warsaw 2014.
- 39. Act of 14 December 2012 on waste. J. Laws 2013, 21, Warsaw 2013.
- 40. *CEN/TC154/TG10/N736 2012;* Secondary Materials. Artificial Aggregates. Final Report on Aggregates Obtained from Secondary Resources. Eurostat: Luxembourg, 2016.
- 41. Góralczyk, S.; Kukielska, D. Secondary sources as a resource base for the production of aggregates. *Sci. Work. Min. Inst. Wrocław Univ. Sci. Technol. Stud. Mater.* **2013**, *136*, 49–59.
- 42. WT-4; Unbound Mixtures for National Roads. Technical Requirements. General Director of National Roads and Motorways. Warsaw: Warsaw, Poland, 2010.
- 43. Domańska, W. (Ed.) Environment 2022; Statistics Poland: Warsaw, Poland, 2022.
- 44. Krawczyk, Z. Waste incineration-necessity or optimal solution. Chemik 2011, 65, 727-745.
- 45. Eurostat Statistics Explained. 2023; Municipal Waste Statistics: Luxembourg, 2023.
- Waszczyłko-Miłkowska, B.; Kamińska-Borak, J. Thermal Treatment of Municipal Waste in Poland in 2020; The Institute of Environmental Protection–National Research Institute: Warsaw, Poland, 2021.
- 47. EN 197-1:2011; Cement. Part 1: Composition, Specifications and Conformity Criteria for Common Cements. CEN: Brussels, Belgium, 2011.
- 48. *EN 933-1:2012;* Tests for Geometrical Properties of Aggregates—Part 1: Determination of Particle Size Distribution–Sieving Method. CEN: Brussels, Belgium, 2012.
- EN 13286-2:2004; Unbound and Hydraulically Bound Mixtures—Part 2: Test Methods for Laboratory Dry Density and Water Content–Proctor Compaction. CEN: Brussels, Belgium, 2004.
- 50. *PN-S-96012:1997;* Roads–Subbase and Subgrade of Cement Stabilized Soils. The Polish Committee for Standardization: Warsaw, Poland, 1997.
- 51. Turner-Walker, G.; Syversen, U. Quantifying histological changes in archaeological bones using BSE-SEM image analysis. *Archaeometry* **2002**, *44*, 461–468. [CrossRef]
- Wassilkowska, A.; Czaplicka-Kotas, A.; Zielina, M.; Bielski, A. An analysis of the elemental composition of micro-samples using EDS technique. *Tech. J. Cheistry* 2014, 111, 133–148.
- 53. EN 196-1:2016; Methods of Testing Cement. Part 1: Determination of Strength. CEN: Brussels, Belgium, 2016.
- 54. EN 12457-4:2002; Characterization of Waste-Leaching. CEN: Brussels, Belgium, 2002.
- 55. *EN 1484:1998*; Water Analysis–Guidelines for the Determination of Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC). CEN: Brussels, Belgium, 1998.
- 56. EN 15216:2022; Environmental Matrices–Determination of Total Dissolved Solids (TDS) in Water and Eluates. CEN: Brussels, Belgium, 2022.
- 57. ISO 10304-1:2007; Water Quality. Determination of Dissolved Anions by Liquid Chromatography of Ions. Part 1: Determination of Bromide, Chloride, Fluoride, Nitrate, Nitrite, Phosphate and Sulfate. ISO: Vernier, Geneva, 2007.
- 58. ISO 10523:2008; Water Quality. Determination of pH. ISO: Vernier, Geneva, 2008.
- 59. *ISO* 12846:2012; Water Quality. Determination of Mercury Method Using Atomic Absorption Spectrometry (AAS) with and without Enrichment. ISO: Vernier, Geneva, 2012.

- 60. ISO 17294-2:2016; Water Quality. Application of Inductively Coupled Plasma Mass Spectrometry (ICP-MS). Part 2: Determination of Selected Elements including Uranium Isotopes. ISO: Vernier, Geneva, 2016.
- 61. EN 15934:2012; Sludge, Treated Biowaste, Soil and Waste. Calculation of Dry Matter Fraction after Determination of Dry Residue or Water Content. CEN: Brussels, Belgium, 2012.
- 62. Regulation of the Minister of the Environment of November 18, 2014 on the conditions to be met when discharging sewage into water or land and on substances particularly harmful to the aquatic environment. *J. Laws* **2014**, 1311, Warsaw 2014.
- 63. Paine, K.A.; Dhir, R.K.; Doran, V.P.A. Incinerator bottom ash: Engineering and environmental properties as a cement bound paving material. *Int. J. Pavement Eng.* **2002**, *3*, 43–52. [CrossRef]

**Disclaimer/Publisher's Note:** The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.