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# Contribution to the Research on the Application of Bio-Ash as a Filler in Asphalt Mixtures

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**Abstract:** The intensive construction of all categories of roads and the very demanding maintenance of the pavement structures of existing roads due to ever-increasing traffic loads confronts us with a lack of resources and also an increase in cost for the constituent materials of asphalt mixtures. On the other hand, there is another problem: large amounts of waste material in the form of bio-ash, which is obtained by burning waste wood biomass in the production of thermal energy and/or electricity. In order to solve the environmental problem of bio-ash disposal, research was conducted on the use of waste bio-ash as a constituent material in asphalt pavements. As part of this study, the effect of asphalt concrete mix, with bio-ash as a filler, on the release of harmful substances into the environment was investigated. The possibility of using wood bio-ash (BA) as a filler in asphalt mixtures was then determined through physical and mechanical property tests. The properties of the asphalt sample's sensitivity to the action of water (indirect tensile strength ratio—ITSR) and resistance to rutting were tested for asphalt concrete type AC 11 surf with 50% bio-ash in the filler. It was established that asphalt concrete does not release harmful substances into the environment and that the 50% share of bio-ash in the filler results in asphalt that has good resistance to water sensitivity and even greater resistance to rutting.

**Keywords:** pavement structures; wood bio-ash (BA); bituminous mixture filler; environmentally harmful substances; asphalt concrete; water sensitivity; rutting

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## 1. Introduction

Throughout the history of mankind, wood has been used as a fuel for heating and food preparation and also for the development of industry and is considered a biologically renewable energy source. In accordance with the European Directive 2009/28/EC [1], in the last ten years, cogeneration power plants have been constructed for the production of electricity and heat using biomass as a renewable energy source.

According to the data available on their website, Hrvatska elektroprivreda dd [2] has built two biomass cogeneration plants—BE-TO Osijek 3 MWe/10 MWt and BE-TO Sisak 3 MWe/10 MWt. The electricity produced from both power plants is purchased by the energy market operator in accordance with the tariff defined via an ordinance on acquiring the status of a privileged electricity producer for a period of 14 years. The operation of such plants contributes to meeting national targets that are in line with EU directives on renewable energy production and energy efficiency. In addition to large cogeneration plants in Croatia, there are a number of smaller thermal power plants that use biomass as an energy source for central heating and electricity production, so large amounts of bio-ash remain as a byproduct of this process [3].

Biomass combustion systems do not pollute the environment and offer significant environmental protection. Reducing greenhouse gas pollution is a major advantage of using biomass energy. Renewable energy sources (RESs) supply 14% of the total world energy demand [4]. RESs include biomass, hydropower, geothermal, solar, wind, and marine. Renewable energy sources are primary, domestic, and clean energy sources.

The studies conducted so far [4–22] have revealed that bio-ash considerably varies as to its composition and properties, depending on the origin of the biomass, biomass handling, and the combustion technology and temperature. The wide range of compositions and properties of bio-ash requires the testing of individual sources with the purpose of determining the possibility of using bio-ash in road construction as a substitute for traditional materials. The chemical composition of bio-ash, primarily the proportion of CaO and pozzolan, suggests the possibility of the partial or complete replacement of traditional binders, cement, and lime, in the stabilization of soils with a low bearing capacity and in the load-bearing layers of pavements and in concrete. In the absence of binding properties, ash can be used as a replacement for mineral fillers and aggregates (sand or gravel) in asphalt mixtures, concrete, and other pavement layers, depending on the ash fraction. A very important aspect of the use of ash is its environmental impact, so the number of its harmful elements must be studied before using ash [5].

In one study [6], the main objective was to explore the environmental effects of wood ash used in road structures on forest roads. Based on the preliminary results of the present study, the heavy metal content of the road structures containing wood ash, as well as the environmental risk related to it, can be managed. However, the results are preliminary and represent a very short period of time, and thus data that covers the whole study period (two years) is needed before the final results can be reported.

In order for the energy requirements of heat and electricity to be satisfied, those based on biomass will certainly play an important role in the overall energy system and will be necessary in order to meet the growing demands of consumers during the energy crisis. In the form of wood chips, wood residue, shavings, sawdust, and straw obtained from harvesting plants, wood biomass forms the basis for most combustion processes that use the energy value of this waste material. All these combustion systems produce a significant amount of ash, which varies from 5% to 15% of the processed biomass [7]. An assessment of the available data for wood residues and wood chips, the two main sources of woody biomass available for energy production, shows us that the current levels of use are far below the available quantities. The trend of using eco-sustainable and carbon-neutral fuels will contribute to an increase in the production of bio-ash worldwide. This has significant implications for the management and handling of waste, i.e., the assessment of the impact on environmental pollution. The limited understanding of ash behavior (in combination with other materials, its properties, and its long-term environmental impacts) poses a risk in excessive ash-disposal scenarios. The development and improvement of technologies for the production of energy from biomass is essential. Improved technologies that produce smaller amounts of ash through a higher-quality incineration process and increased carbon reduction should result in solving the problems associated with ash management [7]. The use of ash is limited by the presence of heavy metals and other inorganic compounds, which are formed as a result of the thermo-chemical reactions that biomass undergoes during combustion, and this makes the ash harmful to the environment. Variability in the concentration of heavy metals in ash results from differences in the properties of the raw material being burned, so there is no uniform inorganic composition profile for ash, and it needs to be checked. Inefficiency and obsolescence in boilers and furnaces also result in high percentages of unburned organic matter in ash. The use of ash includes the application of ash as a fertilizer in agricultural production, for refueling due to the presence of high unburned carbon contents, and/or as an additive in a wide range of construction materials. The presence of alkali metals, alkaline earth metals, chlorine, sulfur, and silicon affects reactivity and leaching into the environment during the inorganic phases. Research confirms that fly ash has significantly lower concentrations of heavy metals than bottom ash, so a mixture of electrostatic precipitators and fly ash could be suitable for use as an additive in forest soil improvement. This should help maintain nutrient cycling instead of depositing these important nutrients and taking up space. Unburned organic matter has been examined in some studies as a source of fuel, and ash recirculation is suggested, technologically extending the retention time and increasing the turbulence of the material inside the boiler. The presence of carbon in ash limits its application because it reduces its binding properties in certain building materials. The high carbon content also presents a challenge for pelletizing and briquetting, as it reduces the level of necessary cohesion [7].

## 2. State of the Art

Asphalt mixtures are used in the construction of the surface, binding, and base layers of an asphalt pavement structure. They are made of crushed coarse and fine aggregates, filler, bituminous binder, and, in some cases, other additives. Asphalt mixtures are quality mixtures with a complex, highly elastic behavior. Many researchers have investigated the potential of bio-ash for usage in asphalt mixtures [8–22].

According to the results of [8], the presence of wood ash can significantly improve the resilient modulus of the asphalt mixture, while stability decreases with increasing wood ash content. However, these values of stability are within the prescribed limits of applicable technical conditions. As the proportion of wood ash increases, permanent deformations decrease, and fatigue resistance improves. Therefore, the authors found that wood ash, as an environmentally sustainable material, is suitable as a replacement material for conventional fillers for the production of hot mix asphalt (HMA) using asphalt concrete AC 10 and bituminous binder PEN 60/70. Wood ash used as a material to replace conventional fillers can not only improve the properties of the asphalt mixture but also reduce environmental pollution. In addition, wood ash can also be used as a bitumen modifier to improve the quality of the asphalt mix.

In [9], samples of bituminous mixtures with 25, 50, 75, and 100% rice husk ash (RHA) as a stone filler substitute for granulometric compositions 4 and 5 (in accordance with Iranian standards) and with 4, 4.5, 5, 5.5, 6, and 6.5% binder were made. The Marshall test was performed, and the Marshall parameters were determined. Subsequently, according to the results for specific gravity and Marshall stability and deformation, a certain optimal bitumen content was obtained for each percentage of RHA. Ten types of bitumen mixture samples were prepared: five samples with granulometric composition number 4 and five samples with granulometric composition number 5. These samples were tested for active water (ITSR), rutting, stiffness modulus, and dynamic creep. An increase in RHA content increases the indirect tensile strength of bituminous mixes when compared to control samples for either saturated or dry samples. For samples made with granulometric composition number 4, with an increase in RHA of 25, 50, 75, and 100%, the amount of TSR increased from 7.7, 13, and 18.1% to 22.8%. The TSR values were 4.1, 7.2, 10.9, and 12.6% for granulometric composition number 5. Increasing the proportion of RHA in the bituminous mixture samples significantly increased the stiffness modulus of the samples. When compared to the control samples, this increase was 54% for granulometry 4 and 117% for granulometry 5. The results clearly show that the samples made from RHA have a higher resistance to rutting, and with increasing RHA, the rutting depth decreases. The results of the dynamic creep test confirm the rutting results. According to these results, the change in flow rate increases with the increase in RHA content, which shows a higher resistance to the occurrence of rutting in the samples made with RHA. The optimal RHA content, considering the requirements and limitations of the prescribed conditions, was determined at 26% (number 4) and 48% for the granulometric composition number 5.

As part of the research by the authors of [10], the properties of SBS (styrene-butadiene-styrene) with a bituminous binder modified with rice husk ash (RHA) and a conventional binder were tested using a rotational viscosity test and a temperature shear test. The properties of the SBS/RHA-modified bitumen mixture were tested for rutting, water penetration, and cracking at low temperatures. The addition of SBS/RHA reduced the penetration value and ductility of the pure bituminous binder and improved the softening point (RB method) of the base bituminous binder. By increasing the proportion of rice husk ash from 0 to 5%, the penetration decreased slightly, and when the proportion was 10% and 15%, the penetration decreased even more. The ductility of the uncured modified bituminous binder decreased slightly with an RHA content of 2–10% and significantly decreased when the RHA content was 15%. The application of SBS/RHA increased the viscosity of the base bituminous binder, and the viscosity increased with the increase in the proportion of RHA. The cycle factor decreased with increasing temperature, and the decreasing trend became slower when the test temperature was higher than 64 °C. The addition of SBS/RHA slightly reduced the resistance to the action of water and the appearance of cracks at low temperatures. The tensile strength ratio (ITSR) of the control and modified bituminous mixture meets the prescribed conditions. It was determined that the ash content of rice husks should not exceed 15%.

The conclusions of the test results [11] are that an increase in the proportion of rice husk ash (RHA) in bitumen causes an increase in the softening point (RB method) and a decrease in the penetration value of bitumen, which indicates that there is an increase in hardness with the bituminous binder. The addition of RHA increases the viscosity of the bituminous binder, improves the penetration index (PI), and lowers the temperature sensitivity of the bitumen. The rheological changes of the bitumen binder with RHA cause an improvement in the physical-mechanical properties of the asphalt mixture (hot mix asphalt—HMA). Bituminous binder with up to 20% RHA increases the stability of the asphalt mixture and also the Marshall quotient (MQ); both are indicators of resistance to rutting. The stiffness modulus of the RHA of the modified asphalt mixtures achieves higher values than those of the standard control mixtures. The fatigue test results of asphalt mixes with RHA confirm an extended service life, which can be attributed to a lower proportion of voids in the mix and/or improved adhesion between aggregate and bitumen.

Based on the data obtained in [12], it was concluded that the value of bitumen penetration decreases if the addition of black rice husk ash (BRHA) is used when compared to the penetration of classic bitumen. The softening point (RB method) increases with increasing BRHA content, and the bitumen becomes more viscous when 6% BRHA is added to it. A PI penetration index of 0.25 indicates high resistance to cracking at low temperatures and the formation of permanent deformation in the form of rutting.

A group of authors [13] investigated the role of fillers in improving the properties of asphalt concrete pavements (ACPs) in resistance to the occurrence of rutting caused by temperature rises, as well as the economic profitability of construction costs. Rock dust of a carbonate composition is usually used as a filler in ACP. This study carried out experimental tests on asphalt concrete containing bagasse ash as a filler. As a reference, the properties of asphalt concrete with stone dust as a filler were taken. Subsequently, samples of asphalt concrete with the addition of 5% bagasse ash in the filler and a stone aggregate with 4.3% of a 60/70 type bitumen binder were tested. During the research, it was established that the use of bagasse ash does not affect the granulometric composition of the asphalt mixture. The measured rutting depths of the asphalt mix with bagasse filler confirm better rutting resistance. Additionally, asphalt pavement using bagasse ash has a lower price of about 0.1% per $m^3$ than conventional pavement.

Based on the results obtained in [14], the efficiency of using fly ash as a substitute for mineral filler in porous bituminous mixtures was evaluated. Modified porous bituminous mixtures with fly ash have a slightly lower content of bituminous binder compared to the controlled versions that use a classic mineral filler. The porous asphalt mix with ash is more resistant to binder leaching than the control mix. While the controlled mixture with a mineral filler has better resistance to rutting, those evaluated with the modified mixture meet the prescribed rutting depth values.

The authors of [15] analyzed the suitability of recycling wood ash (WA) as an alternative filler in bituminous mixtures. The WA used in this study was taken from a landfill located near a food industry plant. Bituminous mortars containing four different amounts of WA were compared to conventional variants with added hydrated lime (HL) in similar amounts. It was found that WA has a lower specific gravity, coarser particles, and a low content of active clay, which affects hydrophilicity. Bituminous mortar with WA was found to have a slightly lower resistance to rutting at high temperatures due to lower stiffness. WA mortars were, however, found to exhibit better fatigue resistance than conventional HL mortars. Bituminous mixtures containing WA had satisfactory Marshall and volumetric properties. The HL and WA mixtures also showed satisfactory resistance to the action of water, which, in both mixtures, decreases with an increase in the amount of filler due to a simultaneous decrease in the proportion of bitumen. According to the proposed methodology, the optimal filler content for HL is 5.5% and 5.6% for WA. WA mixes are more cost-effective as well as more environmentally friendly than HL mixes. By using WA in road construction, a large amount of conventional HL is saved, which reduces the exploitation of natural resources.

In [16], biochar was used as a modifier of bitumen obtained from oil processing. Three amounts of biochar: 2%, 4%, and 8%, and two particle sizes: 75–150 μm and <75 μm were tested. Viscoelastic properties, rutting resistance, and fatigue resistance were determined via testing (rolling thin-film oven—RTFO, pressure aging vessel—PAV, and dynamic shear rheometer—DSR). The viscous properties were confirmed with increasing temperature in biochar-modified bitumen. The addition of biochar increased the resistance to rutting. The resistance of bitumen to fatigue cracking was reduced by the addition of biochar, but the size of the biochar particles significantly affected the fatigue resistance. The small-particle biochar-modified binder had better resistance to fatigue cracking than the coarse-particle biochar-modified bitumen. In conclusion, at the end of the tests, an optimal amount of biochar of 2–4% and a particle size of less than 75 μm was determined.

In the search for ways to improve the quality and load capacity of asphalt pavements, taking sustainable development into account, the researchers of [17] used biowaste as a modifier in asphalt pavements. As part of this study, laboratory tests were conducted on the use of peanut or groundnut shell ash (GSA) as a bituminous binder modifier, and its effects on pavement performance were tested. The bituminous binder test results showed that GSA increases viscosity, shear strength, stiffness, and elastic recovery and improves rutting resistance. It was found that up to 10% GSA can be used to modify the bituminous binder without the risk of segregation during storage and transportation. The addition of GSA to the bitumen binder increases the bond between the bitumen and the aggregate and improves the stiffness modulus. GSA-modified bituminous mixtures indicate lower thermal sensitivity and, thus, good resistance to rutting. By applying GSA (up to 15%), the resistance of bituminous mixtures to fatigue and the development of microcracks is improved. Based on tests conducted on bituminous binder and bituminous mixtures containing GSA, an optimal dose of 10% (by the weight of the binder) was determined. GSA landfills cause environmental problems, and by using them in asphalt pavements, they reduce the cost of road construction and maintenance, as well as reducing the environmental problems caused by the GSA landfills.

Based on the conducted research [18], the optimal share of sugarcane bagasse ash (SCBA) was determined to be 0.34%. Amounts of SCBA, ranging from 0.1 to 0.5% by weight of filler, with an optimum asphalt content (OAC) of 5.63%, were tested. The established results showed that modified bituminous mixtures containing SCBA were effective in increasing Marshall stability by 0.6%, deformation by 4.9%, and stiffness modulus by 17.4% when compared to classic, hot-mixed asphalts (HMA). The proportion of voids in the bituminous mixture is higher when compared to unmodified mixtures and has an impact on better resistance to rutting. In order to increase the resistance to rutting, the modified asphalt sample has an optimal void ratio of 4.94%. The use of SCBA as a filler in HMA as an alternative material resulted in better pavement management.

Based on the analysis [19], it was concluded that the physicochemical composition of the filler has a significant influence on bituminous mixtures. Mixtures prepared with red mud (RM), rice straw ash (RSA), glass powder (GP), and carbide lime (CL) fillers, which have higher porosity than conventional stone dust (SD), were found to have a higher optimum bitumen content (OAC). A modified bituminous mix with a copper tailings (CTs) filler has the lowest OAC because this type of filler has the lowest porosity. Good resistance to cracking and rutting was confirmed for all bituminous mixtures except with an RSA filler. Mixtures with fillers made from smaller particles (RM, CT, CL, and GP) have a higher modulus of rigidity and greater resistance to cracking. The mineralogical composition of the filler affects the adhesion of aggregates and bitumen, which results in good resistance to the action of water. After the tests of all 16 combinations, the best results were obtained with the RM filler and then with the CL, CT, SD, GP, and RSA fillers.

Regarding the incorporation of biomass ash as a filler for bituminous mixtures, based on [20], it was determined that biomass ash has acceptable chemical and physical properties for use as a filler. The biomass bottom ash leachate test showed a low concentration in the leachate of heavy metals, as well as carbon and sulfur. The use of biomass ash as a filler in bitumen mixtures with bitumen emulsion develops a mastic that improves tensile strength, improves behavior to plastic deformations, enables greater absorption of bitumen, and has physical properties similar to traditional mixtures.

The result of the use of waste ash from biomass in bituminous mixtures is a reduction in landfills and in the need for new raw materials for asphalt pavement constructions.

In [21], the physical, chemical, and ecological properties of biomass ash for use as a bituminous mixture filler were tested. The main chemical compounds in biomass ash are silicates, potassium carbonate, sylvite, and arcanite. An examination of the pollution of leachate from biomass ash showed a lower concentration of polluting chemical elements than the maximum limits prescribed by regulations. The environmental effects studied in this research show that less harmful emissions are produced by processing ash from biomass when compared to cement, lime, or limestone filler.

The conclusion is that the use of ash from biomass as a filler in bituminous mixtures is technically possible and that there is a significant reduction in environmental impact when compared to other materials that have been investigated.

In 2013, Melotti et al. [22] investigated 27 types of bio-ash used as a filler in asphalt mixtures and found that the use of bio-ash in bituminous mixtures is safe for the environment due to the binder coating on the filler particles.

## 3. Objective of This Study

The development and increase in the number of biomass power plants, as mentioned before, led to the production of increasing quantities of wood bio-ash in Croatia [3]. The positive experience of different applications of wood ash in the construction industry has inspired a growing number of wood ash application studies for usage in pavement structures. One of these studies was a laboratory evaluation of the influence of wood bio-ash (BA), as part of the filler in an asphalt mixture, on the physical-mechanical properties of the mixture, as described in [23]. Different laboratory tests were carried out on the asphalt mixture of asphalt concrete type AC 16 surf, which is used for the construction of leveling layers and wearing layers. A mineral filler (industrial) was used as the basic filler in the AC 16 surf asphalt mixture. As a supplement to the mineral filler, wood bio-ash (BA) was used in different proportions (0%, 25%, 50%, and 75%). The ash was cyclone ash, i.e., fly ash collected on a cyclone deduster. The Marshall stability (MS) and Marshall quotient (MQ) had the highest values in the case of an asphalt mixture with 50% bio-ash (BA) content in the filler, with the lowest deformation-flow ratio, which indicates increased resistance of the asphalt to rutting. The indirect tensile strength values of water-conditioned (ITSw) samples are the highest in the case of an asphalt mixture with a 25% content of bio-ash (BA) in the filler, while, in the case of the dry (ITSd) samples, they are highest in the asphalt mixture with 50% bio-ash (BA) content in the filler, which has an impact on the increased resistance of the asphalt to the appearance of cracks. As the results of the laboratory evaluation of [23] showed, wood ash can replace part of the stone filler in asphalt mixtures with 50% content, improving the performance of the asphalt mixture. According to the described laboratory evaluation [23], research on asphalt mixtures using wood ash in the filler is continuing, and one of these studies is described in this article.

The aim of this research is to examine the resistance to rutting of asphalt mixtures using bio-ash (50% in the filler; type AC 11 surf asphalt concrete) and to determine the water sensitivity of the asphalt specimens. The aim is also to confirm that wood bio-ash, as a filler covered with bitumen and embedded in the asphalt layer of the roadway, is not harmful to the environment, i.e., that there is no release of hazardous substances during road exploitation.

## 4. Materials

A bituminous mixture of asphalt concrete type AC 11 surf 50/70 (in accordance with HRN EN 13108-1 [24]), which is used for the construction of wearing pavement layers, was used for laboratory tests.

### *4.1. Components of the Asphalt Mixture*

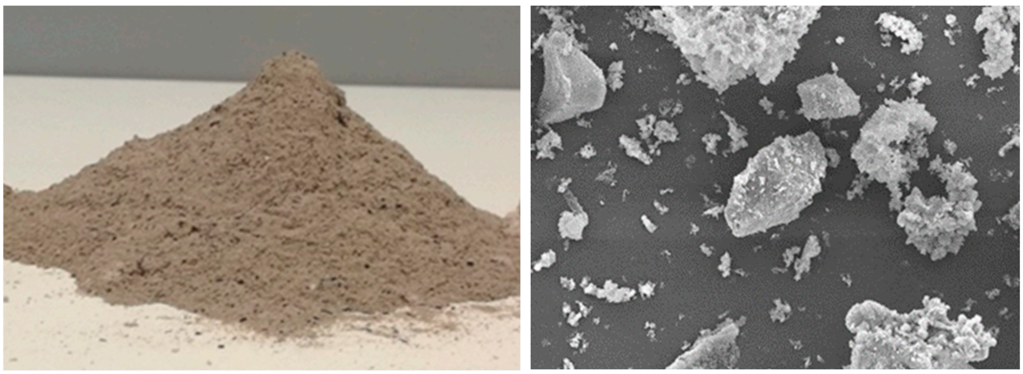
The basic constituent materials of bituminous mixtures are crushed coarse and fine aggregates of a silicate composition. The properties of the aggregate were determined in accordance with the standard HRN EN 13043 [25], and the aggregate granulometric composition (see Table 1) in accordance with the standard HRN EN 12697-2 [26].

**Table 1.** Granulometric composition of the aggregate (HRN EN 12697-2).

| Sieve opening size (mm) | 0.063 | 0.09 | 0.125 | 0.25 | 0.5 | 0.71 | 1 | 2 | 4 | 5.6 | 8 | 11.2 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cumulative passing (%) | 6.6 | 7.0 | 9 | 13 | 19 | 22 | 25 | 35 | 54 | 61 | 78 | 98 |

A total of 50% industrial filler of a carbonate composition and 50% bio-ash filler were used in the AC 11 surf asphalt mixture, and for the control bituminous mixture, 100% of industrial filler of a carbonate composition was used.

The bio-ash used was cyclone ash (Figure 1), which is fly ash collected on a cyclone deduster; an assessment of the relevant fine particles is shown in Table 2 in accordance with the standard HRN EN 12697-2 [27].



(**a**) (**b**)

**Figure 1.** (**a**) Cyclone ash from wood biomass and (**b**) SEM microphotograph (magnification SEM_MAG = 1000×).

**Table 2.** Assessment of fine ash particles (HRN EN 933-10).

| Sieve opening size (mm) | 2.0 | 0.125 | 0.063 |
|---|---|---|---|
| Cumulative passing (%) | 100 | 78 | 49 |

The chemical composition of the wood ash is important in determining the ash impact in an asphalt mixture; therefore, the composition is shown by the portion of individual components (mass %): MgO = 3.06, $Al_2O_3$ = 0.44, $SiO_2$ = 4.05, $P_2O_5$ = 2.90, $SO_3$ = 1.59, $K_2O$ = 2.82, and CaO = 46.91. The main chemical component is calcium oxide CaO, which can increase the adhesion between the bitumen and the aggregate and, thus, decrease the aggregate nakedness. The morphology of wood ash is also important for better understanding the effect of ash on the properties of asphalt mixtures; therefore, scanning electron microscopy (SEM) was used for the images of the shape and size of the ash particles (Figure 1) and shows the ash composition of the particle with different shapes and sizes. Ash particles are irregular in shape and have a rough, porous surface, and the particle sizes are also very uneven.

The binder in the asphalt mixture is road construction bitumen, type 50/70, with the soluble part forming 5.8% (of a total soluble and insoluble part of 6.0%). The bitumen's properties are determined in accordance with the HRN EN 12591 standard [28]. The properties of road construction bitumen 50/70, produced by MOL, Hungary, Szazhalombatta refinery, are given in Table 3.

**Table 3.** Properties of road construction bitumen 50/70.

| Property | Test Method | Specification |
|---|---|---|
| Density at 25 °C ($Mg/m^3$) | MSZ EN ISO 3838 | 1.020 |
| Softening point (°C) | MSZ EN 1427 | 46–54 |
| Penetration at 25 °C (mm $10^{-1}$) | MSZ EN 1426 | 50–70 |
| Resistance to hardening at 163 °C | | |
| – Change of mass (%) | MSZ EN 12607-1 | Max. +/−0.5 |
| – Retained penetration (%) | MSZ EN 1426 | Min. 50 |
| – Increase in softening point (°C) | MSZ EN 1427 | Min. 9 |
| Flashpoint (°C) | MSZ EN ISO 2592 | Min. 230 |
| Solubility (%) | MSZ EN 12592 | Min. 99 |

The stone aggregate fractions used (0/2, 2/4, 4/8, and 8/11 mm); the mineral filler and the cyclone bio-ash filler meet the properties prescribed by the HRN EN 13043 standard [25].

### 4.2. Asphalt Mixture Properties and Preparation

In the laboratory, after mixing (at a temperature of 160 °C ± 5 °C for 3 min in accordance with HRN EN 12697-35 [29]), the component materials of the asphalt mix samples were prepared with a roller compactor in accordance with HRN EN 12697-33 [30]. Then, from the same bitumen mixtures, samples were prepared with an impact compactor in accordance with HRN EN 12697-30 [31] using a compactor with a metal stand, a compaction temperature of 150 °C ± 5 °C, and a compaction energy of 2 × 35 blows. The physical and mechanical properties of the asphalt mixtures with and without bio-ash in the filler are shown in Table 4.

**Table 4.** Physical-mechanical properties of the asphalt mixtures AC 11 surf 50/70.

| | With 50% BA Filler | With 100% Mineral Filler | Test Method |
|---|---|---|---|
| Bulk density of a compacted asphalt specimen (Mg/m$^3$) | 2.492 | 2.471 | HRN EN 12697-6 [32] |
| Maximum density of an asphalt mixture (Mg/m$^3$) | 2.586 | 2.597 | HRN EN 12697-5 [33] |
| Air Void Content (%) | 3.6 | 4.9 | HRN EN 12697-8 [32] |
| Voids filled with bitumen (%) | 79.6 | 74.4 | HRN EN 12697-8 [34] |
| Marshall Stability (kN) | 11.7 | 10.6 | HRN EN 12697-34 [35] |
| Deformation (mm) | 2.6 | 2.2 | HRN EN 12697-34 [35] |
| Tangential deformations (mm) | 1.3 | 1.2 | HRN EN 12697-34 [35] |
| Total deformations (mm) | 2.9 | 3.5 | HRN EN 12697-34 [35] |
| Marshall Quotient (kN/mm) | 4.5 | 4.8 | HRN EN 12697-34 [35] |

## 5. Test Methods

Firstly, laboratory tests were carried out to determine the environmental impact of asphalt samples with a BA filler, i.e., the leaching properties of harmful substances were tested.

### *5.1. Testing the Leaching of Hazardous Substances from a Bituminous Mixture with Bio-Ash*

This test was for the purpose of confirming that the wood bio-ash BA in the composition of the bituminous mixture did not have a harmful effect on the environment as a result of the release of hazardous substances from the pavement. The eluate prepared in accordance with HRN EN 12457-4 [36] was tested using the equipment specified in the standards (Figure 2) in demineralized, ultra-pure water and a chemical reagent of recognizable analytical purity.

**Figure 2.** Test equipment for testing the leaching of hazardous substances.

The property tests were carried out using the corresponding methods on the eluate of the crushed asphalt sample and on the eluate of the submerged asphalt sample in the form of a Ø100 mm cylinder (Table 5).

**Table 5.** Testing the eluate of a crushed asphalt sample and of a submerged asphalt sample (cylinder Ø100 mm).

| Characteristic | Test Method |
|---|---|
| Concentration of hydrogen ions (pH) | HRN EN ISO 10523 [37] |
| Fluorides | RU-MET-001 |
| Sulfates | SM23 Ed2017, 4500 E |
| Chlorides | HRN ISO 9297 [38] |
| Phenolic index | HRN ISO 6439 [39] |
| Content of the total dissolved solids (TDS) | HRN EN 15216 [40] |
| Dissolved organic oxide (DOC) | HRN EN 1484 [41] |
| Arsenic (As), Barium (Ba), Cadmium (Cd) | HRN EN ISO 17294-2 [42] |
| Total chromium (Cr), Copper (Cu), Mercury (Hg) | HRN EN ISO 17294-2 |
| Molybdenum (Mo), Nickel (Ni), Lead (Pb) | HRN EN ISO 17294-2 |
| Antimony (Sb), Selenium (Se), Zinc (Zn) | HRN EN ISO 17294-2 |

### *5.2. Testing the Water Sensitivity of Asphalt Samples*

Frequent damage to and the deterioration of asphalt pavements are caused by the action of water during road use. The water sensitivity of asphalt is manifested as a reduction in the connection between the bituminous binder and the aggregate under traffic loads, resulting in the degradation of the driving surface of the pavement.

Table 6 shows the average dimensions and densities of the dry samples before conditioning, and Table 7 shows the average dimensions and densities of the water-saturated samples before vacuuming. After vacuuming the water-saturated samples, no changes in dimensions (height, diameter) were found, and there was no change in the volume or swelling of the asphalt samples.

**Table 6.** Average dimensions and densities of dry samples before conditioning.

| Asphalt Sample | Average Height (mm) | Average Diameter (mm) | Average Dry Sample Mass (g) | Average Sample Density (Mg/m$^3$) |
|---|---|---|---|---|
| Dry (four pieces) With 50% BA filler | 64.2 | 101.5 | 1232.2 | 2.412 |
| Dry (four pieces) With 100% mineral filler | 64.5 | 101.4 | 1238.0 | 2.419 |

**Table 7.** Average dimensions and densities of water-saturated samples before vacuuming.

| Asphalt Sample | Average Height (mm) | Average Diameter (mm) | Average Sample Mass (g) | Average Sample Density (Mg/m$^3$) |
|---|---|---|---|---|
| Wet (four pieces) With 50% BA filler | 63.2 | 101.5 | 1220.5 | 2.415 |
| Wet (four pieces) With 100% mineral filler | 64.0 | 101.4 | 1237.2 | 2.419 |

In accordance with HRN EN 12697-12 [43], four wet test samples were conditioned for 72 +/− 2 h at 40 °C +/− 2 °C in water; the other four dry test samples were conditioned for 72 +/− 2 h at 20 °C +/− 5 °C in air, and all eight asphalt samples were tested using method A at 15 °C.

The sensitivity of the asphalt pavement layer to the action of water (ITSR) is expressed by the ratio of the indirect tensile strengths of the wet and dry asphalt samples.

$$ITSR = ITSw/ITSd \tag{1}$$

where

ITSR = indirect tensile strength ratio (%);
ITSw = indirect tensile strength of wet asphalt sample (kPa);
ITSd = indirect tensile strength of dry asphalt sample (kPa).

*5.3. Testing the Rutting Resistance*

The bituminous mixture with 50% bio-ash content in the filler and the 100% mineral filler were tested for rutting resistance in accordance with procedure B of HRN EN 12697-22 [44] using a small-sized device in the air (Figure 3). Compaction was carried out using controlled compaction energy on a roller compactor with a segment of a smooth steel roller, after which the samples with a diameter of 200 mm were conditioned for a period of 5 days at ≤25 °C and at a test temperature of 60 °C +/−1 °C. The tests were carried out on two samples (Ø200 mm), for which the density and height were determined in accordance with HRN EN 12697-6 [31] and HRN EN 12697-29 [45], respectively. The number of test wheel passes was 20,000 passes in total. The test wheel load on the asphalt surface was 700 N, while the length of the measuring path was 200 mm, and the load frequency was 26.5 per minute.

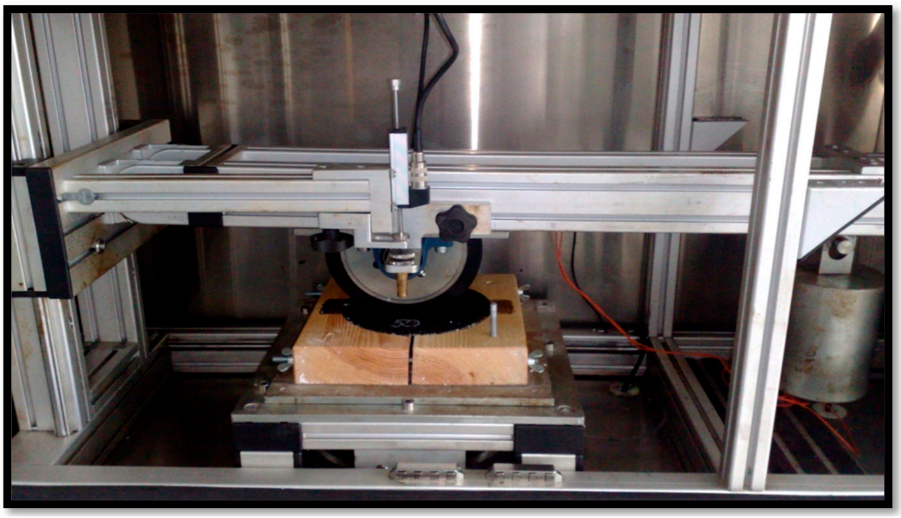


**Figure 3.** Wheel Tracker.

The following parameters were obtained:

- tracking speed WTSair in mm/1000 cycles;
- proportional (%) rut depth PRDair at 10,000 cycles (20,000 passes);
- rut depth RDair in mm after 10,000 cycles (20,000 passes).

The following properties were determined for the bituminous mixtures (AC 11 surf 50/70) (Table 8):

- the density of the test sample with 50% bio-ash filler and the density of the control sample with 100% mineral filler;

- the height of the test sample with 50% bio-ash in the filler and the height of the control sample with 100% mineral filler (0% bio-ash).

**Table 8.** Density and height of test sample I and II and control samples.

| Asphalt AC 11 Surf 50/70 | Sample with 50% BA Filler | Sample with 100% Mineral Filler |
|---|---|---|
| Density (Mg/m$^3$) | 2.484 | 2.508 |
| Height (mm) | 38.7 | 42.5 |

## 6. Results and Discussion

### *6.1. The Results of Hazardous Substance Leaching from Bituminous Mixture with BA*

All hazardous substance leaching from the bituminous mixtures with BA was satisfactory, i.e., they are below the maximum allowed concentrations in accordance with the regulations [46–48]. The results for the crushed asphalt sample given in Table 9 show that milled asphalt aggregate with bio-ash as a filler is not harmful to the environment and can be treated fully in accordance with the HRN EN 13108-8 standard [49]. The concentration of hydrogen ions (pH) was 10.3 pH unit/20 °C.

**Table 9.** Test results of crushed asphalt sample eluate.

| Characteristic, (Unit of Measurement) | Results | Max. Allowed Concentration [46–48] |
|---|---|---|
| Fluorides (mg/kg dry matter) | <1.00 | 10 |
| Sulfates (mg/kg dry matter) | <30.0 | 1000 |
| Chlorides (mg/kg dry matter) | <1.00 | 800 |
| Content of total dissolved solids (TDS) (mg/kg dry matter) | 290 | 4000 |
| Dissolved organic oxide (DOC) (mg/kg dry matter) | 32.0 | 500 |
| Arsenic (As) (mg/kg dry matter) | <0.005 | 0.5 |
| Barium (Ba) (mg/kg dry matter) | 0.363 | 20 |
| Cadmium (Cd) (mg/kg dry matter) | <0.005 | 0.4 |
| Total chromium (Cr) (mg/kg dry matter) | 0.006 | 0.5 |
| Copper (Cu) (mg/kg dry matter) | <0.005 | 2 |
| Mercury (Hg) (mg/kg dry matter) | <0.002 | 0.01 |
| Molybdenum (Mo) (mg/kg dry matter) | <0.005 | 0.5 |
| Nickel (Ni) (mg/kg dry matter) | <0.005 | 0.4 |
| Lead (Pb) (mg/kg dry matter) | 0.015 | 0.5 |
| Antimony (Sb) (mg/kg dry matter) | <0.005 | 0.06 |
| Selenium (Se) (mg/kg dry matter) | <0.005 | 0.1 |
| Zinc (Zn) (mg/kg dry matter) | <0.005 | 4 |

The test results of intact/whole asphalt samples (cylinder) are shown in Table 10, which confirms that the asphalt layer with 50% bio-ash filler should not be harmful to the environment during the exploitation cycle of the pavement. The concentration of the hydrogen ions (pH) was 9.2 pH unit/20 °C.

**Table 10.** Test results of the submerged asphalt sample eluate (cylinder).

| **Characteristic, (Unit of Measurement)** | **Results** | **Max. Allowed Concentration [46–48]** |
|---|---|---|
| Fluorides (mg/kg dry matter) | 0.433 | 10 |
| Sulfates (mg/kg dry matter) | 13.3 | 1000 |
| Chlorides (mg/kg dry matter) | <12.0 | 800 |
| Content of total dissolved solids (TDS) (mg/kg dry matter) | <12.0 | 4000 |
| Dissolved organic oxide (DOC) (mg/kg dry matter) | 30.7 | 500 |
| Arsenic (As) (mg/kg dry matter) | <0.005 | 0.5 |
| Barium (Ba) (mg/kg dry matter) | 0.077 | 20 |
| Cadmium (Cd) (mg/kg dry matter) | <0.005 | 0.4 |
| Total chromium (Cr) (mg/kg dry matter) | <0.005 | 0.5 |
| Copper (Cu) (mg/kg dry matter) | <0.005 | 2 |
| Mercury (Hg) (mg/kg dry matter) | <0.002 | 0.01 |
| Molybdenum (Mo) (mg/kg dry matter) | <0.005 | 0.5 |
| Nickel (Ni) (mg/kg dry matter) | <0.005 | 0.4 |
| Lead (Pb) (mg/kg dry matter) | 0.016 | 0.5 |
| Antimony (Sb) (mg/kg dry matter) | <0.005 | 0.06 |
| Selenium (Se) (mg/kg dry matter) | <0.005 | 0.1 |
| Zinc (Zn) (mg/kg dry matter) | <0.005 | 4 |

The test established that bio-ash as a filler in the asphalt mixture does not release harmful ingredients that endanger the environment and confirms its usability in asphalt layers in road construction.

*6.2. The Impact of the BA Filler on the Results of Indirect Tensile Strength/Ratio—ITS/ITSR*

The indirect tensile strengths of the conditioned dry samples (ITSd) and water-saturated samples (ITSw) were determined in accordance with the HRN EN 12697-23 standard [50]. The results of the indirect tensile strength of the dry samples (ITSd) and the water-saturated samples (ITSw), together with the ITSR results, can be found in Figure 4.

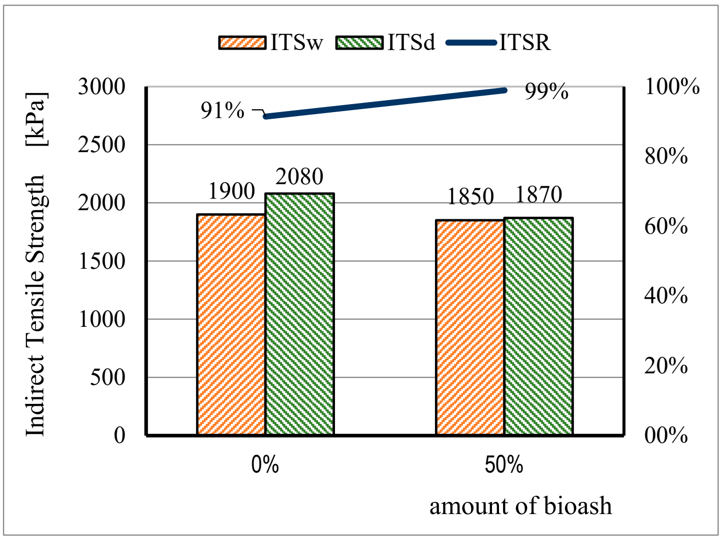



**Figure 4.** Results of indirect tensile strength (ITS) and ratio (ITSR).

The ratio of the indirect tensile strengths of the wet and dry asphalt samples with BA ($ITSR_{BA}$) and without BA (ITSR) are

$$ITSR_{BA} = ITSw/ITSd = 1850/1870 = 98.9\%$$

$$ITSR = ITSw/ITSd = 1900/2080 = 91.4\%$$

The prescribed condition, according to the technical regulations [51], is ITSR $\geq$ 90% in the most demanding category, which confirms that the asphalt concrete with 50% bio-ash in the filler achieves a good resistance to the action of water, better than asphalt with 100% mineral filler, with an increase of 8.2% achieved.

*6.3. The Impact of the BA Filler on the Results of Rutting Resistance*

Table 11 shows the recorded average rut depths of both the sample with 50% bio-ash filler and of the control sample without a bio-ash filler. The results indicate that bio-ash BA improves the asphalt sample's resistance to rutting.

**Table 11.** Results of the rut depth.

| Number of Passes | The Depth of the Rut for Asphalt 50% BA (mm) | The Depth of the Rut for Asphalt 100% Mineral Filler (mm) |
|---|---|---|
| 0 | 0.00 | 0.00 |
| 2000 | 1.00 | 1.04 |
| 4000 | 1.16 | 1.16 |
| 6000 | 1.25 | 1.24 |
| 8000 | 1.30 | 1.30 |
| 10,000 | 1.36 | 1.37 |
| 12,000 | 1.40 | 1.42 |
| 14,000 | 1.43 | 1.46 |
| 16,000 | 1.45 | 1.49 |
| 18,000 | 1.48 | 1.52 |
| 20,000 | 1.50 | 1.54 |

During the rutting process, after every 2000 passes on the abscissa (x-axis), the depth of rutting in millimeters is recorded on the ordinate (y-axis), and a curve is drawn. Figure 4 shows the rutting curves (mean value of rutting) of the asphalt with BA and the asphalt without BA (the control sample). From Figure 5, it is evident that the rut depth RDair after 10,000 cycles (20,000 passes) is 2.7% higher for the asphalt sample without bio-ash BA as a filler. The asphalt with bio-ash is more resistant to the deformation of wheel tracks (rutting).

An additional part of the research was conducted in order to confirm the influence of BA filler on rutting. The results were compared with the rutting parameters tested in the same laboratory on eight samples of asphalt concrete (AC 11 surf 50/70) with the classic mineral filler (of carbonate origin). Eight asphalt mixtures were selected according to the bulk density of the compacted asphalt specimen, which is comparable (within reproducibility) to the mixture with bio-ash. These eight samples were made as part of the preparation for the preliminary laboratory work for the mixed formulas of the asphalt mixtures, and they differ from each other in terms of the type of component materials and the composition and physical/mechanical properties of the mixture.

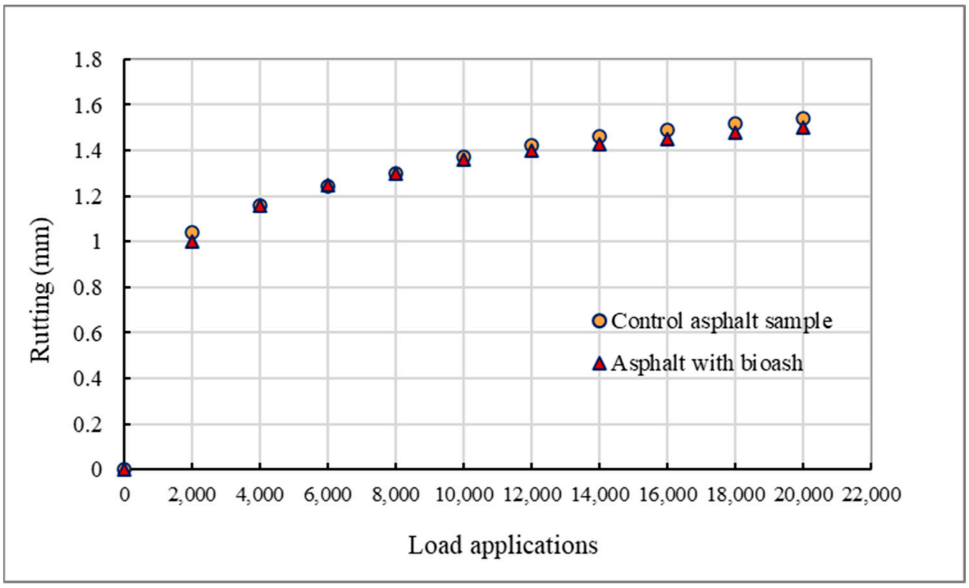



**Figure 5.** Impact of asphalt type on rutting depth.

Table 12 shows the results of the tested parameters for resistance to rutting: an asphalt sample with 50% BA filler and a sample with 100% mineral filler, and the median values of eight samples of the same type of asphalt mixture (AC 11 surf 50/70) with a classic mineral filler without BA.

**Table 12.** Results of rutting parameters.

| The Rutting Parameter | Asphalt Sample 50% BA | Asphalt Sample 100% Mineral Filler | Asphalt Compared 8 Samples without BA | Max. Allowed Value [51] |
| --- | --- | --- | --- | --- |
| | (1) | (2) | (3) | |
| Tracking speed WTSair (mm/1000 cycles) | 0.03 | 0.04 | 0.03 | 0.07 |
| Relative (%) rut depth PRDair at 10,000 cycles | 3.88 | 3.66 | 3.68 | 7.00 |
| Rut depth RDair (mm) after 10,000 cycles | 1.50 | 1.54 | 1.36 | - |

Figures 6–8 show comparisons of rutting speed (WTSair), relative rutting depth (PRDair), and rutting depth (RDair) of

(1) an asphalt sample with 50% BA filler;
(2) an asphalt sample with 100% mineral filler;
(3) the median values of eight samples of the same type of asphalt mixture (AC 11 surf 50/70) using a classic mineral filler without BA.

By using 50% BA filler in an asphalt mixture of type AC 11 surf 50/70, we achieved good resistance to the appearance of ruts.

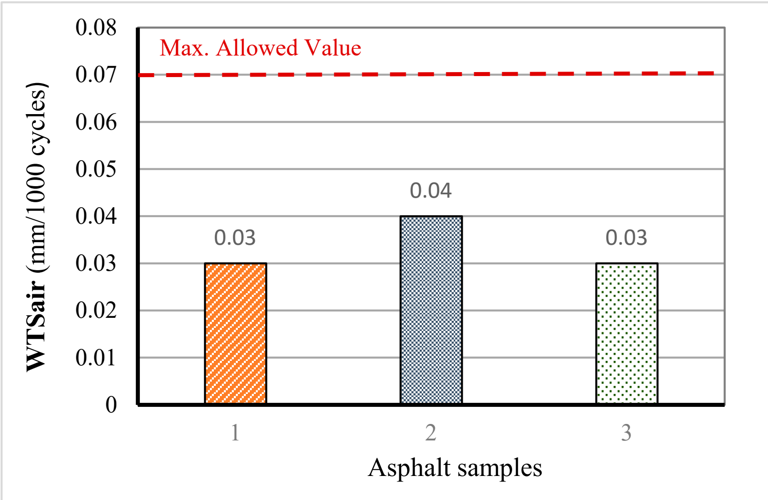



**Figure 6.** Rutting speed WTSair.

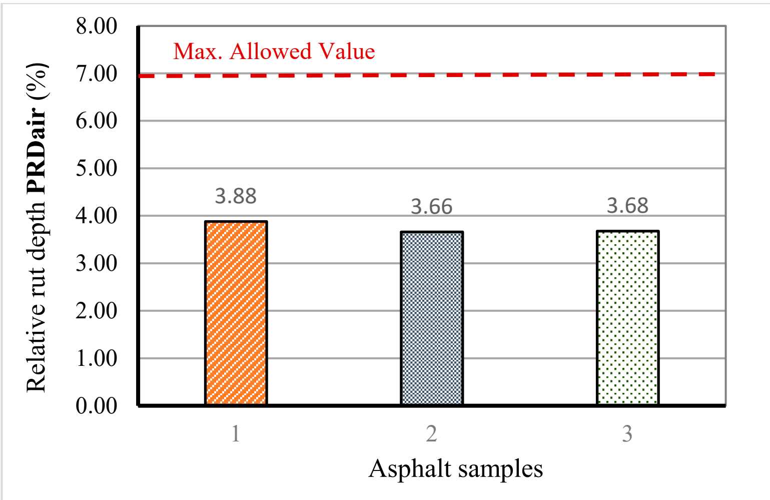



**Figure 7.** The relative rut depth PRDair.

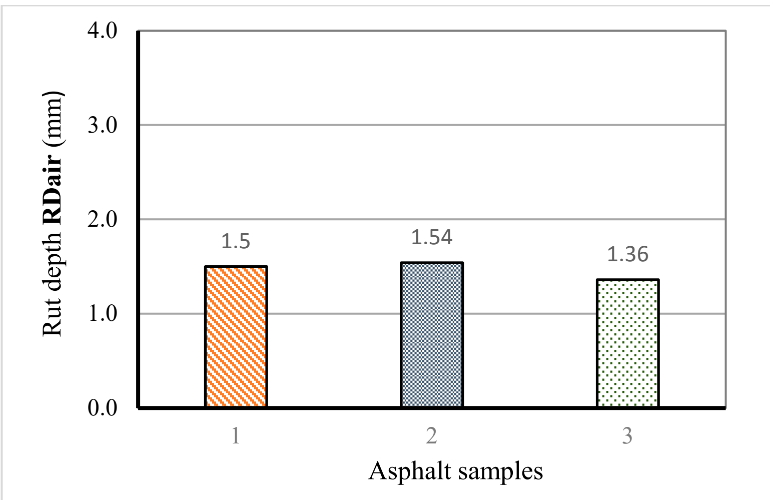



**Figure 8.** Rut depth RDair.

## 7. Conclusions

The research has examined the application of wood bio-ash (BA) as a substitute for 50% of the conventional mineral filler made from stone material with a carbonate composition.

A bituminous mixture of asphalt concrete with 50% bio-ash as a filler was tested for its impact on environmental pollution, its water sensitivity, and its resistance to the occurrence of rutting. This research confirmed the possibility of using BA in the asphalt layers of the pavement for the following reasons:

1. Due to the hazardous substances (sulfates, content of total dissolved solids (TDS), and total chromium) contained in BA, it cannot be deposited in landfills, and the same applies if it is combined with other similar waste [23]. The tests conducted on a crushed asphalt sample and a compacted asphalt roller with BA as a filler confirm that there are no illegal amounts of discharge; therefore, there is no harmful impact on the environment (Tables 6 and 7). Bio-ash, covered with bitumen in the asphalt pavement, is protected from the release of harmful substances into the environment;
2. A ratio of the indirect tensile strength (ITSR = ITSw/ITSd) of 98.9% confirms the good resistance of asphalt with BA to the action of water, which has a direct impact on the durability of the pavement and, therefore, road maintenance costs;
3. By testing the resistance to rutting of the asphalt samples with and without BA, good rutting parameters (WTSair = 0.03 mm/1000 cycles; PRDair = 3.88%; RDair = 1.5 mm) were confirmed. It is important that the addition of bio-ash does not worsen the resistance of the asphalt mixture to the occurrence of plastic deformations, and multiple is used for environmental protection, which also results in positive economic indicators.

Laboratory tests have confirmed the justification of using wood ash as a filler in bituminous mixtures, achieving the following advantages:

- A reduction in the amount of BA waste in landfills and its harmful impact on the environment;
- Enables the use of less industrial stone filler, which is extracted from quarries, the opening and extending of which harm the natural environment;
- Good resistance of the asphalt to the action of water and good resistance to rutting, which directly implies better management of the asphalt pavement.

In the end, it should be mentioned that the variability of the WA composition should always be considered, and the possibility of application should be confirmed with full laboratory tests. For the type of asphalt from which a recipe using bio-ash was created in the laboratory at the asphalt plant, the bio-ash from the same power plant/heating plant or from more than one plant should be used if the same biomass is used as the energy source.

The knowledge gained through this research has confirmed that the goal of achieving sustainability and innovation in road construction can be achieved by using new component materials in asphalt mixes.

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**Informed Consent Statement:** Informed consent was obtained from all subjects involved in the study.

**Data Availability Statement:** The authors agree to submit test results as appropriate.

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