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Podgorica, 18.10.2019.
Broj: 1772

UNIVERZITET CRNE GORE
-Centar za doktorske studije-
PODGORICA

U prilogu vam dostavljamo predlog Vijeća Građevinskog fakulteta Univerziteta Crne Gore, za imenovanje Komisije za ocjenu doktorske disertacije, sa potrebnom dokumentacijom, kandidata mr Katarine Mirković.

S poštovanjem,

SEKRETAR FAKULTETA,

Miro Božović, dipl.prav.

УНИВЕРЗИТЕТ ЦРНЕ ГОРЕ
ГРАЂЕВИНСКИ ФАКУЛТЕТ - ПОДГОРИЦА
Број 1758
Подгорица 17.10.2019. год.

Na osnovu člana 64. Statuta Univerziteta Crne Gore i člana 41. Pravila doktorskih studija Univerziteta Crne Gore, Vijeće Građevinskog fakulteta u Podgorici na sjednici održanoj 16.10.2019.godine, utvrdilo je

PREDLOG

Predlaže se Senatu Univerziteta Crne Gore da imenuje Komisiju za ocjenu doktorske disertacije mr Katarine Mirković,dipl.inž.građ., pod naslovom „Primjena elektro-filterskog pepela u asfaltnim mješavinama", u sastavu:

1. Prof. dr Radomir Zejak,dipl.inž.građ., redovni profesor Građevinskog fakulteta Univerziteta Crne Gore.
2. Prof. dr Goran Mladenović,dipl.inž.građ., vanredni profesor Građevinskog fakulteta Univerziteta u Beogradu.
3. Prof. dr Zvonko Tomanović,dipl.inž.građ., redovni profesor Građevinskog fakulteta Univerziteta Crne Gore.

Komisija je dužna da Vijeću Građevinskog fakulteta i Senatu Univerziteta Crne Gore, podnese izvještaj koji sadrži ocjenu doktorske disertacije, u roku od 45 dana od dana imenovanja Komisije.

- VIJEĆE GRAĐEVINSKOG FAKULTETA U PODGORICI -

DEKAN,

Prof. dr Marina Rakočević

ISPUNJENOST USLOVA DOKTORANDA

OPŠTI PODACI O DOKTORANDU			
Titula, ime, ime roditelja, prezime	Mr Katarina Veljko Mirković		
Fakultet	Građevinski fakultet		
Studijski program	Građevinastvo		
Broj indeksa	5/10		
NAZIV DOKTORSKE DISERTACIJE			
Na službenom jeziku	Primjena elektrofilterskog pepela u asfaltnim mješavinama		
Na engleskom jeziku	Fly Ash Application in Asphlt Mixtures		
Naučna oblast	Građevinastvo - Saobraćajno-urbanistička oblast		
MENTOR/MENTORI			
Prvi mentor	Prof. dr Goran Mladenović, dipl.inž.građ, vanredni profesor	Građevinski fakultet Univerziteta u Beogradu, Republika Srbija	Građevinarstvo – Građenje i održavanje puteva i aerodroma
KOMISIJA ZA PREGLED I OCJENU DOKTORSKE DISERTACIJE			
Prof. dr Zvonko Tomanović, dipl.inž.građ, redovni profesor		Građevinski fakultet Univerziteta Crne Gore, Crna Gora	Građevinarstvo – Geotehnika
Prof. dr Goran Mladenović, dipl.inž.građ, vanredni profesor		Građevinski fakultet Univerziteta u Beogradu, Republika Srbija	Građevinarstvo – Građenje i održavanje puteva i aerodroma
Prof. dr Radomir Zejak, dipl.inž.građ, redovni profesor		Građevinski fakultet Univerziteta Crne Gore, Crna Gora	Građevinarstvo – Materijali i konstrukcije

Datum značajni za ocjenu doktorske disertacije	
Sjednica Senata na kojoj je data saglasnost na ocjenu teme i kandidata	29.01.2015. god.
Dostavljanja doktorske disertacije organizacionoj jedinici i saglasanost mentora	19.09.2019. god.
Sjednica Vijeća organizacione jedinice na kojoj je dat prijedlog za imenovanje komisija za pregled i ocjenu doktorske disertacije	16.10.2019. god.

ISPUNJENOST USLOVA DOKTORANDA
U skladu sa članom 38 pravila doktorskih studija kandidat **je** cjelokupna ili dio sopstvenih istraživanja vezanih za doktorsku disertaciju publikovao u časopisu sa **SCI/SCIE** liste kao prvi autor.

Spisak radova doktoranda iz oblasti doktorskih studija koje je publikovao u časopisima sa SCI/SCIE liste

- **Rad objavljen u časopisu indeksiranom u Science Citation Index listi**

1. Mirković K., Tošić N., Mladenović G.: *Effect of Different Types of Fly Ash on Properties of Asphalt Mixtures,* Advances in Civil Engineering, Article ID 8107264, 2019., 11 pages. https://www.hindawi.com/journals/ace/2019/8107264/ https://doi.org/10.1155/2019/8107264

- **Radovi objavljeni na međunarodnim/regionalnim konferencijama i časopisima koji sadrže djelove disertacije**

2. Mirković K., Mladenović G.,: *Testing of Fly Ash Properties for the Application in Asphalt Mixtures,* The 5th Scientific-Expert Meeting "Road and Environment", Vršac, Serbia, 28-29th September 2017.

3. Mirković K., Mladenović G,: *Characteristics of Asphalt Mixtures $AC11s_{surf}$ with Fly Ash,* The 5th Scientific-Expert Meeting "Road and Environment", Vršac, Serbia, 28-29th September 2017.

4. Mirković K., Mladenović G.,: *Fly Ash Impact on Characteristics of Asphalt Mixtures,* Scientific Journal of Road and Traffic Engineering –Journal of Serbian Road Association No.3 July - September, 2017.

Obrazloženje mentora o korišćenju doktorske disertacije u publikovanim radovima

Mr Katarina Mirković je, kao prvi autor, dio rezultata sopstvenih istraživanja vezanih za doktorsku disertaciju objavila u radu koji je publikovan u časopisu indeksiranom na SCI listi, kao i u dva rada predstavljena na regionalnom naučno-stručnom skupu i u jednom radu publikovanom u regionalnom časopisu. U nastavku je dat osvrt na rad objavljen u časopisu *Advances in Civil Engineering,* sa dijelom rezultata sopstvenih eksperimentalnih istraživanja.

Naslov objavljenog rada je *Effect of Different Types of Fly Ash on Properties of Asphalt Mixtures.* Koautori rada su doc. dr Nikola Tošić i prof. dr Goran Mladenović. Rad prikazuje rezultate ispitivanja karakteristika asfaltne mješavine za habajući sloj, AC11s u kojoj je primijenjen elektrofilterski pepeo kao djelimična ili potpuna zamjena za standardni filer – kameno brašno. Primijenjeni pepeli su različitog porijekla i imenovani su kao P, G i K, dok su primijenjeni procenti zamjene 25%, 50%, 75% i 100%.

U okviru uvodnog dijela rada prikazan je kraći pregled dosadašnjuh ispitivanja u svijetu iz predmetne oblasti, sa generalnim zaključcima i osvrtom na oblasti koje treba dalje istraživati. Ovaj dio rada predstavlja sažetak poglavlja 2 doktorske disertacije.

U radu su predstavljene metodologija ispitivanja i karakteristike komponentnih materijala sa akcentom na ispitane karakteristike tri elektrofilterska pepela. Podaci u ovom dijelu rada preuzeti su iz poglavlja 3 i 4 disertacije, u kojima se detaljno predstavljaju i diskutuju rezultati ispitivanja komponentnih materijala, kao i metodologija ispitivanja. Centralni dio rada je posvećen predstavljanju i diskutovanju rezultata ispitivanja asfaltnih mješavina spravljenih sa različitim vrstama pepela i variranim procentima primjene pepela, upoređujući ih sa kontrolnom asfaltnom mješavinom spravljenom sa kamenim brašnom. U radu su predstavljene zapreminske karakteristike mješavina, kao i stabilnost i tečenje po metodi Maršala, otpornost na negativno dejstvo vode i otpornost na trajnu deformaciju. Rezultati predstavljeni u ovom dijelu rada su

preuzeti iz poglavlja 5 doktorske disertacije.
Zaključci izvedeni u radu su dio zaključaka navedenih u disertaciji u podpoglavlju 6.1.2 i oni glase:

- Zapreminska i prividna zapreminska masa asfaltnih mješavina zavise od zapreminske mase pepela, kao i od efekta otvrdnjavanja koji pepeo ima na bitumenski mastiks.
- Dodavanjem bilo kojeg od tri pepela primijenjena u eksperimentu, postižu se zadovoljavajuće zapremisnke karakteristike, pri čemu, šupljine u mineralnoj mješavini i šupljine u asfaltnoj mješavini zavise od vrste i procenta primjene pepela.
- Povećane šupljine u mineralnoj i asfaltnoj mješavini u odnosu na kontrolnu, kod mješavina sa pepelima krupnije gradacije (P i K), ukazuju na potrebu povećanja količine bitumena, dok se pepeo sa finijom gradacijom (G) može razmatrati i kao djelimična zamjena za bitumen.
- Dodatak sva tri pepela povećava stabilnost i smanjuje tečenje mješavine, što se dovodi u vezu sa efektom otvrdnjavanja koji pepeli imaju na bitumenski mastiks. Ovo svojstvo pepela preporučuje ih za upotrebu u toplijim klimatskim područjima gdje je poželjna ugradnja asfaltnih mješavina sa većom stabilnošću.
- Sve mješavine P i G grupe imaju veću otpornost na dejstvo vode od kontrolne mješavine, pri čemu je trend rasta u skladu sa povećanjem procenta primjene pepela. Ovo svojstvo je posebno izraženo kod mješavina G grupe sa izrazito visokim udjelom kalcijum oksida koji omogućava jake veze između zrna agregata i bitumenskog omotača. Kod mješavina K grupe prisutan je suprotan trend, gdje otpornost na dejstvo vode opada sa porastom procenta primjene pepela.
- Generalno, otpornost asfaltne mješavine na trajnu deformaciju zavisi od oblika čestice pepela i teksture njene površine. Sve mješavine K grupe pokazale su izrazitu otpornost na trajnu deformaciju, dok su mješavine P grupe sa manjim procentima zamjene i mješavine G grupe sa većim procentima zamjene pokazale veću otpornost na trajnu deformaciju od kontrolne mješavine.

Datum i ovjera (pečat i potpis odgovorne osobe)

U Podgorici, 17.10.2019.god.

UNIVERZITET CRNE GORE Građevinski fakultet MP

DEKAN

Prilog dokumenta sadrži:

1. Potvrdu o predaji doktorske disertacije organizacionoj jedinici
2. Odluku o imenovanju komisije za pregled i ocjenu doktorske disertacije
3. Kopiju rada publikovanog u časopisu sa odgovarajuće liste
4. Biografiju i bibliografiju kandidata
5. Biografiju i bibliografiju članova komisije za pregled i ocjenu doktorske disertacije sa potvrdom o izboru u odgovarajuće akademsko zvanje i potvrdom da barem jedan član komisije nije u radnom odnosu na Univerzitetu Crne Gore

VIJEĆU GRAĐEVINSKOG FAKULTETA

UNIVERZITETA CRNE GORE

PODGORICA

PREDMET: ***Zahtjev za ocjenu doktorske disertacije mr Katarine Mirković, dipl.inž.grad.***

Poštovani,

U skladu sa Pravilima studiranja na doktorskim studijama Univerziteta Crne Gore podnosim zahtjev za ocjenu doktorske disertacije pod nazivom

Primjena elektrofilterskog pepela u asfaltnim mješavinama

Završetkom doktorske disertacije i objavom rada u časpisu sa SCI/SCIE liste koji sadrži rezultate dijela sopstvenih istraživanja sprovedenih u okviru izrade doktorske disertacije, ispunila sam uslove za njenu predaju. Ovim putem se obraćam Komisiji za doktorske studije Građevinskog fakulteta da inicira predlog Komisije za ocjenu doktorske disertacije.

Uz zahtjev prilažem:

- pismenu saglasnost mentora da rad zadovoljava kriterijume doktorske disertacije;
- štampani primjerak doktorske disertacije;
- fotokopije objavljenih radova tematski vezanih za doktorsku disertaciju;
- CD sa cjelokupnim sadržajem doktorske disertcije u PDF formatu i
- pismenu Izjavu o autorstvu (prilog 1 iz Uputstva o oblikovanju doktorske disertacije).

S poštovanjem,

U Podgorici, 19.09.2019. godine

Podnosilac

Mr Katarina Mirković, dipl.inž.grad.

[signature]

UNIVERZITET CENE GORE

GRAĐEVINSKI FAKULTET

PODGORICA

Na osnovu člana 37. Pravila doktorskih studija Univerziteta Crne Gore dajem sledeću

SAGLASNOST

Rad pod nazivom „Primjena elektrofilterskog pepela u asfaltnim mješavinama", autora mr Katarine Mirković, dipl.inž, građ., stručnog saradnika Građevinskog fakulzeta Univerziteta Crne Gore, zadovoljava kriterijume doktorske disertacije, propisane Statutom Crne Gore i Pravilima doktorskih studija.

U Beogradu, 9.09.2019.god.

MENTOR

Prof. dr Goran Mladenović, dipl.inž.građ.

УНИВЕРЗИТЕТ ЦРНЕ ГОРЕ

ГРАЂЕВИНСКИ ФАКУЛТЕТ - ПОДГОРИЦА

19. 09. 2019.

Spisak radova koji sadrže rezultate i djelove doktorske disertacije mr Katarine Mirković:

- Radovi objavljeni u časopisima sa SCI/SCIE liste

1. Mirković K., Tošić N., Mladenović G.: *Effect of Different Types of Fly Ash on Properties of Asphalt Mixtures,* Advances in Civil Engineering, Article ID 8107264, 2019., 11 pages.

- Radovi objavljeni na međunarodnim/regionalnim konferencijama i časopisima

2. Mirković K., Mladenović G.,: *Testing of Fly Ash Properties for the Application in Asphalt Mixtures,* The 5^{th} Scientific-Expert Meeting "Road and Environment", Vrsac, Serbija, 28-29^{th} September 2017.

3. Mirković K., Mladenović G,: *Characteristics of Asphalt Mixtures $AC11s_{surf}$ with Fly Ash,* The 5^{th} Scientific-Expert Meeting "Road and Environment", Vrsac, Serbija, 28-29^{th} September 2017.

4. Mirković K., Mladenović G.,: *Fly Ash Impact on Characteristics of Asphalt Mixtures,* Scientific Journal of Road and Traffic Engineering –Journal of Serbian Road Association No.3 July - September, 2017.

U Beogradu, 09.09. 2019.

MENTOR

Prof. dr Goran Mladenović, dipl.inž.građ.

Izjava o autorstvu

Potpisana Katarina Mirković

Broj indeksa/upisa 5/10

Izjavljujem

da je doktorska disertacija pod naslovom

Primjena elektrofilterskog pepela u asfaltnim mješavinama

- rezultat sopstvenog istraživačkog rada,
- da predložena disertacija ni u cjelini ni u djelovima nije bila predložena za dobijanje bilo koje diplome prema studijskim programima drugih ustanova visokog obrazovanja,
- da su rezultati korektno navedeni, i
- da nijesam povrijedio/la autorska i druga prava intelektualne svojine koja pripadaju trećim licima.

U Podgorici, 19. 09. 2019. godine

Potpis doktoranda

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UNIVERZITET CRNE GORE GRAĐEVINSKI FAKULTET / / Katarina Mirković PRIMJENA ELEKTROFILTERSKOG PEPELA U ASFALTNIM MJEŠAVINAMA doktorska disertacija Podgorica, 2019. godine UNIVERSITY OF MONTENEGRO FACULTY OF CIVIL ENGINEERING / / Katarina Mirković FLY ASH APPLICATION IN ASPHALT MIXTURES Doctoral Dissertation Podgorica, 2019 Doktorand: Ime i prezime: Katarina Mirković, dipl. inž. građ. Datum i mjesto rođenja: 20. 08. 1964.

god, Podgorica, Crna Gora Postdiplomske studije: Građevinski fakultet Univerziteta Crne Gore Postdiplomske magistarske akademske studije, Studijski program Građevinarstvo, Saobraćajni smjer, 2010. god. Mentor: prof. dr Goran Mladenović, dipl. inž. građ. Vanredni profesor Građevinskog fakulteta Univerziteta u Beogradu Komisija za ocjenu podobnosti doktorske teze i kandidata: prof. dr Goran Mladenović, dipl. inž.

građ. Vanredni profesor Građevinskog fakulteta Univerziteta u Beogradu prof. dr Radomir Zejak, dipl. inž. građ. Redovni profesor Građevinskog fakulteta Univerziteta Crne Gore prof. dr Milun Krgović, dipl. inž. met. Redovni profesor Metalurško-tehnološkog fakulteta Univerziteta Crne Gore prof. dr Zvonko Tomanović, dipl. inž. građ.

Redovni profesor Građevinskog fakulteta Univerziteta Crne Gore Komisija za ocjenu doktorske disertacije: Komisija za odbranu doktorske disertacije: Datum odbrane:

Dear Dr. Mirković,

I am pleased to let you know that your article has been published in its final form in "Advances in Civil Engineering."

Katarina Mirković, "Effect of Different Types of Fly Ash on Properties of Asphalt Mixtures," Advances in Civil Engineering, vol. 2019, Article ID 8107264, 11 pages, 2019. https://doi.org/10.1155/2019/8107264.

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Best regards,

Yassmin Mabrouk Fathy
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Advances in Civil Engineering
Volume 2019, Article ID 8107264, 11 pages
https://doi.org/10.1155/2019/8107264

Research Article

Effect of Different Types of Fly Ash on Properties of Asphalt Mixtures

Katarina Mirković [1], Nikola Tošić [2], and Goran Mladenović [2]

[1] *University of Montenegro, Faculty of Civil Engineering, Bulevar Džordža Vašingtona b.b., 81000 Podgorica, Montenegro*
[2] *University of Belgrade, Faculty of Civil Engineering, Bul. kralja Aleksandra 73, 11000 Belgrade, Serbia*

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Academic Editor: Faiz U.A. Shaikh

In order to preserve natural resources, the use of waste and alternative materials in the construction and maintenance of roads is increasingly investigated. This paper presents the results of testing wearing course asphalt mixtures (AC 11s SURF 50/70) made with various percentages of fly ash, used as a partial or complete substitute for mineral filler. The properties of fly ash were determined to assess their suitability for use in asphalt mixtures. The experimental research was performed on asphalt samples containing fly ash from three different sources, with 25%, 50%, 75%, and 100% of mineral filler substitution. The control mixture was prepared with 100% of mineral filler. The paper presents the volumetric composition, stability, and flow of asphalt mixtures tested on standard Marshall's samples, water sensitivity, and resistance to permanent deformation. The results of this study indicate that a satisfactory volumetric composition can be achieved by adding fly ash, while the bulk density and voids of the mineral and asphalt mixture generally depend on the type of fly ash and its content. The stability and flow of mixtures with fly ash are favourable compared with the control mixture. The water sensitivity of mixtures with fly ash is generally lower compared with the control mixture and depends on the type and percentage of fly ash. The resistance to permanent deformation of the asphalt mixtures depends on the fly ash type and percentage. The results obtained in this study are an important step towards broader implementation of fly ash in asphalt mixtures.

1. Introduction

The construction and maintenance of roads requires a large amount of high-quality materials. In order to preserve natural resources, a number of studies have been carried out to prove the usability of different waste and alternative materials in concrete and asphalt pavements such as steel slag, waste rubber, waste polyethylene, recycled concrete, and asphalt aggregate, as well as construction and demolition waste [1–6].

One of the most promising of these materials is fly ash, the by-product of coal combustion which is generated during the production of electricity in thermal power plants. Globally, almost one billion tonnes of fly ash is generated annually [7] and its landfilling represents a significant environmental problem. As a material with pozzolanic activity, fly ash has been widely used in a variety of applications such as concrete, soil improvement, and road construction, namely, for embankments and both unbound and bound subbase and base layers [8–12].

Research on the possibility of its application in asphalt mixtures began around the middle of the previous century, as part of efforts to contribute to sustainable engineering, with the aim of obtaining mixtures of satisfactory properties, reducing the harmful effects of landfilling and preserving natural resources. It has been intensified in the last few decades and has concentrated on two approaches.

One approach is focused on the use of fly ash in bitumen mortar as a substitute for a certain amount of bitumen [13] in order to improve its properties, mainly resistance to permanent deformation, stiffness, viscosity at high temperatures, and temperature sensitivity [13–15].

Sobolev et al. [13] studied the effect of fly ash on the rheological performance of bitumen and mastic using a

Dynamic Shear Rheometer (DSR). The study included two types of asphalt binders and two types of fly ash, Class C and Class F, as defined in ASTM C618 [16], which differentiates the ashes based on the total content of silica, aluminium, and iron oxide ($SiO_2 + Al_2O_3 + Fe_2O_3$). A microstructural investigation of bitumen with fly ash using a scanning electron microscope (SEM) demonstrated a crack-arresting effect induced by fly ash particles at low temperatures. The investigation of the rheological performance of mastics using DSR confirmed that fly ash can be used as a bitumen extender, replacing up to 15% of bitumen to improve its resistance to permanent deformation at high temperatures. The best improvement was achieved by adding 15% of Class F fly ash or 30% of Class C fly ash.

Sharma et al. [14] investigated the use of fly ash with different fly ash-to-bitumen (FA/B) ratios in mastics, ranging from 0,6 to 1,2. The authors found that the softening point, viscosity and complex modulus of mastics were increasing with increasing fly ash content, while the phase angle was decreasing, indicating improved shear resistance with increasing fly ash content. They also found that mastics become less sensitive to the FA/B ratio with increasing temperature.

As representatives of the first approach to the usability of fly ash in asphalt mixtures, these studies share joint conclusions regarding the improvement of mastics properties, possible reduction of the bitumen content, and hence, reduced cost and environmental impact.

The studies which consider the application of fly ash in asphalt mixtures and belong to the second approach, included different classes of fly ash, with varying chemical composition and content of fly ash in the mixture. The main objective of these studies was to determine the optimum amount of mineral filler replacement as well as the impact of fly ash on the volumetric composition, optimum bitumen content, mechanical properties, and performance of asphalt mixtures [13, 14, 17–22].

Sharma et al. [14] found that the Optimum Bitumen Content (OBC) in asphalt mixtures with Class C fly ash as a filler depends on the filler content and Rigden voids. The OBC decreases with increasing fly ash content. Mistry and Roy [21] came to a similar conclusion for dense bitumen macadam mix with Class F fly ash. The OBC was slightly decreasing with increasing fly ash content by up to 6% and that when adding up to the 4% of fly ash to dense bitumen macadam asphalt mix, the OBC can be reduced by 7.5% compared with the control mixture with 2% of hydrated lime as a filer. However, Androic et al. [20] showed an increase in the air void content for fly ash asphalt mixtures compared with a mixture with stone filler, indicating the need for increased bitumen content in mixtures with fly ash.

Mistry and Roy [21] found that for up to 4% of fly ash, the Marshall stability was lower for the control mixture with 2% of hydrated lime. The Marshall stability was increasing with fly ash content by up to 6% of fly ash and then decreasing for higher fly ash contents. Androic et al. [20] reached a similar conclusion, but obtained the highest stability with 3% of fly ash. However, Kar et al. [17] found that stability was consistently lower for asphalt mixtures with fly ash compared with the control mixture for bitumen contents ranging from 4% to 7%.

Likitlersuang and Chompoorat [18] found that the ratio of stability and flow of mixtures with a fly ash content ranging from 1% to 5% was almost constant and similar to the control mixture, which is confirmed by findings of Mistry and Roy [21].

Sobolev et al. [19] found that asphalt mixtures with fly ash have a higher modulus compared with traditional mixtures with stone dust, which is the result of an increased complex modulus of asphalt mastics with fly ash. Similar findings are reported by Sharma et al. [14].

Sharma et al. [14] found that the Indirect Tensile Strength (ITS) was increasing with fly ash content for all four types of fly ash that were tested. Likitlersuang and Chompoorat [18] also obtained a slight increase of ITS with increasing fly ash content at temperatures of 25°C and 55°C. However, results of Kar et al. [17] indicate that asphalt mixtures with fly ash have slightly lower ITS compared with the control mixture. Sharma et al. [14] also found that the horizontal tensile strain at failure was largest for mixtures with 7% of fly ash and that fly ash with high CaO contents showed the largest strains, indicating higher resistance to low-temperature cracking.

The Indirect Tensile Strength Ratio (ITSR) is a common parameter used to evaluate moisture susceptibility of asphalt mixtures and presents the ratio of ITS of specimens conditioned in water and that of dry specimens. Alternatively, Indian specifications [22] use the Retained Stability (RS) as measure of moisture susceptibility of asphalt mixtures. Sharma et al. [14] found that both ITSR and RS of fly ash asphalt mixtures were equal to or higher than the corresponding values of traditional asphalt mixtures with stone filler. Both ratios were decreasing with increasing fly ash content. The largest improvement in resistance to moisture was obtained with fly ash with the largest CaO content. This is confirmed by Likitlersuang and Chompoorat [18] who also found an increase in the ITSR for mixtures with fly ash. However, Kar et al. [17] found that RS for mixtures with fly ash is slightly lower for conventional mixture with stone filler but also satisfied requirements according to specifications [22].

Sharma et al. [14] is the only reference presenting the resistance to permanent deformation of asphalt mixtures with fly ash obtained using the static creep test. The authors found that mixtures with fly ash have a favourable rutting resistance compared with the mixture with stone filler. However, the testing was performed at a relatively low temperature of 30°C which is not fully representative for rutting resistance, and the static creep test is inferior to simulation tests available nowadays, such as the wheel-tracking test.

Based on the literature review, it can be concluded that the addition of fly ash to asphalt mixtures may lead to an improvement in mixture properties and performance. However, findings from previous studies are very often contradictory, leading to the conclusion that the validity of some of the presented results may be limited to specific fly ashes used in these studies.

The objective of the study presented in this paper was to investigate the impact of three fly ashes with significantly different chemical compositions on the properties of asphalt mixtures in order to get a more general conclusion regarding the possibility of using fly ash in asphalt mixtures. In addition, the objective was to evaluate the impact of different percentages of substitution of mineral filler, ranging from 25% to full substitution, on properties and performance of asphalt mixtures, including volumetric composition and resistance to water and permanent deformation.

2. Materials and Methods

In this study, a wearing course asphalt mixture with maximum aggregate size of 11 mm (AC 11s SURF 50/70) was used as the control mixture. The mixture was made of quartz-latite stone aggregate (Quarry "Štitarica," Mojkovac, Montenegro), available in fractions 0/4, 4/8, and 8/11 mm, limestone mineral filler produced by the company "Šišković", Podgorica, Montenegro, and road bitumen B 50/70 (Oil Refinery "Pančevo," Serbia). In addition, fly ash from three thermal power plants (TPPs) in the region was used as a partial or full replacement of mineral filler: TPP "Pljevlja"-Pljevlja, Montenegro; TPP "Gacko"-Gacko, Bosnia and Herzegovina; and TPP "Kosovo B"-Priština, Kosovo (this designation is without prejudice to positions on status and is in line with UNSCR 1244/1999 and the ICJ opinion on the Kosovo declaration of independence), denoted as fly ash "P" (from TPP "Pljevlja"), "G" (from TPP "Gacko"), and "K" (from TPP "Kosovo B").

2.1. Fly Ash. In order to determine the properties of sampled fly ashes and assess their suitability for use in asphalt mixtures, an extensive testing program was carried out (Table 1), to test chemical, physical, and mechanical properties of fly ash, as well as their environmental impact. Mechanical and physical properties tested included tests used typically for mineral fillers in asphalt mixtures. Testing for the presence of heavy metals, radioactivity, and leaching test was performed in order to assess the potential negative environmental impact of fly ash use.

Table 2 shows the classification of sampled fly ashes according to their chemical composition, while Tables 3 and 4 present the results of the physical and mechanical properties of ashes, which are relevant for assessing the suitability of their use in asphalt mixtures.

The fly ash P is a Class F aluminosilicate ash that is pozzolanically active, recommending it for use in the cement industry. A significant percentage of CaO (21.08%), unusual for Class F fly ash, makes it also suitable for use in asphalt mixtures. The smooth texture and spherical shape of the particles make it suitable for embedding [23], but the silicate component gives it hydrophilicity, i.e., weaker affinity with bitumen. In addition, this fly ash is more coarsely graded compared with the standard mineral filler.

A high proportion of CaO in fly ash G (74.44%) and somewhat less in fly ash K (45.98%) recommend them for use in asphalt mixtures due to improved adhesion of aggregate and bituminous binder, with positive effects on mixture stability. However, fly ashes G and K do not meet the requirements of ASTM C 618-19 [16] for Class C in terms of the minimum content of $SiO_2 + Al_2O_3 + Fe_2O_3$ of 50%. Fly ash G is an alkaline fly ash with a high content of CaO, whereas fly ash K has a high sulphate content and is a highly alkaline fly ash without pozzolanic properties.

All three fly ashes meet the requirements of relevant standards related to ecological suitability, presented in Table 1.

To determine the suitability of fly ash for use in asphalt mixtures, the following tests were performed on fly ash samples (Table 4):

(i) Particle size distribution
(ii) Particle density
(iii) Voids of dry compacted filler/fly ash (Rigden voids), which indicate the capacity of the filler/fly ash to hold bitumen
(iv) Increase in the softening point defining the degree of stiffening of the mixture of mineral filler/fly ash and bitumen (ΔR&B)
(v) Bitumen number (BN) that represents the apparent viscosity of the mixture of mineral filler/fly ash and water

Voids of the dry compacted mineral filler are usually in the range of 28–45%, while for limestone filler, the range is narrower, 30–34% [24]. All three fly ashes have a higher percentage of Rigden voids than the mineral filler. The void content in fly ash P is slightly higher than the upper limit for voids in a mineral filler, while fly ashes K and G have substantially higher void contents (55% and 59%, respectively), not unusual for fly ash [25], indicating higher absorption of oil components from the bitumen, which will increase stiffness of the mastics and can negatively affect the properties and ageing of bitumen and the bituminous mixture [26].

Increase of the ring and ball softening point (ΔR&B) indicates an increase of bitumen 70/100 stiffness to which 37.5% (v/v) of specific filler is added. Mineral fillers have an increase in the softening point between 8°C and 25°C, with a most common value of 15°C.

The increase in ΔR&B for fly ash P is lowest and within the range, while ΔR&B of the fly ash G is slightly above the upper limit, and ΔR&B of the fly ash K is significantly outside the ΔR&B range for mineral filler. This means that the fly ash P causes the lowest increase in the mastic (bitumen) stiffness, compared with the other two fly ashes, which is favourable for the longevity of asphalt mixtures and their resistance to cracking.

BN indicates the amount of water which needs to be added to the fly ash in order to achieve apparent viscosity adequate for the production of asphalt mixtures. Fly ash G has a BN similar to mineral filler as a result of high CaO

Table 1: Overview of tests conducted on fly ash specimens.

Mechanical properties/method	Density *EN 1097-7*	Voids in dry compacted filler (fly ash) *EN 1097-4*	Increase of the ring and ball softening point *EN 13179-1*	Bitumen number *EN 13179-2*
Chemical composition/method	Na_2O, MgO, Al_2O_3, SiO_2, SO_3, K_2O, CaO, TiO_2, Fe_2O_3, Gd_2O_3, R *SEM method*			
Ecological suitability/method	Presence of heavy metals *EN 13656; AMA-12; EN 12457-4*	Radioactivity *Gamma-spectrometric analysis*		Leaching test *EN 12457-4, EN 12506, EN 13370, EPA 2007*
Content of organic matters/method	Loss on ignition-LOI *EN 12879*			
Mineral properties/ method	Determination of amorphous and crystalline phase contents			
Physical properties	Colour, shape, and size of particles, surface texture *SEM method*	Gradation *EN 933-10*		

Table 2: Classification of tested fly ashes by chemical composition.

Fly ash	Chemical composition (%)			Classification		
	SiO_2	CaO	$SiO_2 + Al_2O_3 + Fe_2O_3$	ASTM C 618	Mining Institute Belgrade	pH classification
P	42.83	21.08	69.78~70	F	Calcium-silicate	Aluminosilicate, pozzolanic active
G	6.04	74.44	$13.85 \ll 50$	/	Calcium	Alkaline
K	16.91	45.98	$26.15 \ll 50$	/	Calcium-silicate	High sulphate content, highly alkaline, no pozzolanic properties

Table 3: Physical properties of fly ash specimens and standard mineral filler.

Property	Fly ash origin			Mineral filler
	P	G	K	
Colour	Grey	Pale dark yellow	Dark yellow	White
Particle shape	Mostly spherical	Combined spherical and irregular	Combined spherical and irregular	Angle-shape to prismatic
Surface texture	Mostly smooth	Mostly rough	Mostly rough	Mostly rough

Table 4: Mineral filler and fly ash properties.

Property		Method	Min. filler Šišković	Fly ash		
				P	G	K
Particle size distribution (passing through sieve opening, mm) (%)	0.063	EN 933-10	80.8	42	44	75
	0.09		90.2	57	67	80
	0.125		92.8	70	89	84
	0.25		99.6	74	91	86
	0.71		100	84	94	92
	2.0			100	100	100
Particle density (mg/m^3)		EN 1097-7	2.711	2.272	2.966	2.821
Voids of dry compacted mineral filler (%)		EN 1097-4	31.5	46	59	55
Increase of the softening point (ΔR&B test) (°C)		EN 13179-1	10.3	17.0	28.8	38.6
Bitumen number (BN) (ml)		EN 13179-2	21	45	27	46

content, while other two fly ashes have substantially higher BN values.

2.2. Mineral Filler. The mineral filler used in this study represents the standard material used for fillers in asphalt mixtures in the region. The properties of the mineral filler are shown in Table 4. Figure 1 shows the unified particle size distribution of the mineral filler and three fly ash samples.

Fly ash G completely satisfies EN 13043 particle size distribution requirements for mineral filler, while fly ashes P and K have substantially coarser particle size distribution.

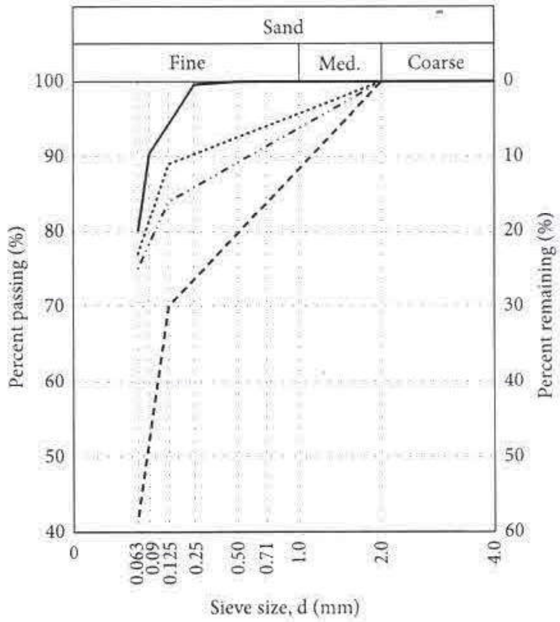

Figure 1: Particle size distribution of mineral filler and three fly ash samples.

2.3. Stone Aggregate. Crushed quartz-latite stone aggregate was used in this study. The particle size distribution and physical properties of the stone aggregate fractions are shown in Figure 2 and Table 5, respectively.

2.4. Bitumen. The basic properties of bitumen B 50/70, used in this study, are shown in Table 6.

2.5. Asphalt Mixtures. One control mixture (with mineral filler only) and 12 mixtures with partial or full replacement of mineral filler with fly ash were tested. The mineral filler participated in the total aggregate matrix with 4%. The replacement of mineral filler with fly ash was 25%, 50%, 75%, and 100% which is 1%, 2%, 3%, and 4% of the aggregate matrix. The codes of the mixtures used in this study are given in Table 7, with the replacement percentage denoted by the subscript. Table 7 also provides the material composition of the mixtures.

The OBC of 5.6% for the control mixture AC 11s was determined using the Marshall method (EN 12697-34) [28]. The same OBC was then applied for mixtures with fly ash.

For all prepared mixtures, the volumetric composition and the physical and mechanical properties were determined by the application of methods shown in Table 8.

Water sensitivity was tested according to EN 12697-12A [29] and expressed by a ratio (ITSR) of ITS of Marshall specimens, compacted with 2×35 blows and immersed in water for 70 h at 40°C, and the ITS of dry specimens.

Testing of the resistance to permanent deformation of asphalt mixtures was performed on a small device, in air, at a temperature of 60°C, after 10 000 cycles (20,000 passes) in accordance with procedure B of EN 12697-22 [30]. For all 13 mixtures, two slabs $320 \times 260 \times 50$ mm were tested for each mixture. In order to simulate field conditions, prior to the compaction of slabs, asphalt mixtures were conditioned at a temperature of 135°C for 4 h.

3. Results and Discussion

This section presents the results of experimental tests performed in this study, namely, volumetric composition, stability, and flow of asphalt mixtures, water resistance, and resistance to permanent deformation. Subsequently, the results are discussed in detail for each experimental test.

Taking into consideration the scope and aim of the experimental program, the discussion of the results comprises a descriptive comparative analysis with the aim of assessing the influence of different fly ashes and fly ash percentages on the properties of asphalt mixtures. Such a discussion offers a first step and empirical basis for further, more detailed, experiments and analyses.

3.1. Volumetric Composition, Stability, and Flow of Asphalt Mixtures. The volumetric properties, stability, and flow of asphalt mixtures with fly ash and the control mixture with stone filler are presented in Table 9.

The apparent and bulk densities of mixtures with fly ash depend on the density and content of fly ash. The apparent and bulk densities of P mixtures decreased with increasing fly ash content, mainly due to the substantially lower density of ash P compared with the other two fly ashes and mineral filler. Contrary to this, the apparent and bulk densities of G mixtures increased with increasing fly ash content, while for K samples, the opposite trend was found. The results for K mixtures can be explained by the stiffening effect of fly ash K on the bitumen mastics and its highest ΔR&B value (Table 4).

Voids in the mineral aggregate (VMA) in all G mixtures have essentially the same value, within the range of the control mixture. VMA for P and K mixtures is higher than for the control mixture and for both groups there is a constant increasing trend with the percentage of replacement, more pronounced for P mixtures due to their coarser gradation. Air voids in P, G, and K mixtures follow the same trend within the group as VMA. All G and K mixtures meet specifications for air voids (below 6.5%). However, all P mixtures except P_{25} have air voids higher that the specified upper limit. The mixture P_{100} has the largest voids in the asphalt mixture, almost 64% higher than the control mixture.

The addition of fly ash improves the stability of asphalt mixtures up to 16% (mixture P_{75}). The only exceptions are the K_{100} mixture which has the lowest stability of all tested mixtures (4% lower than the control mixture) and the P_{50} mixture for which stability is equal to the control mixture. The flow of all mixtures with fly ash is smaller for the control mixture, with the P group having the lowest values. The P_{100} mixture has the lowest flow deformation, 40% smaller than

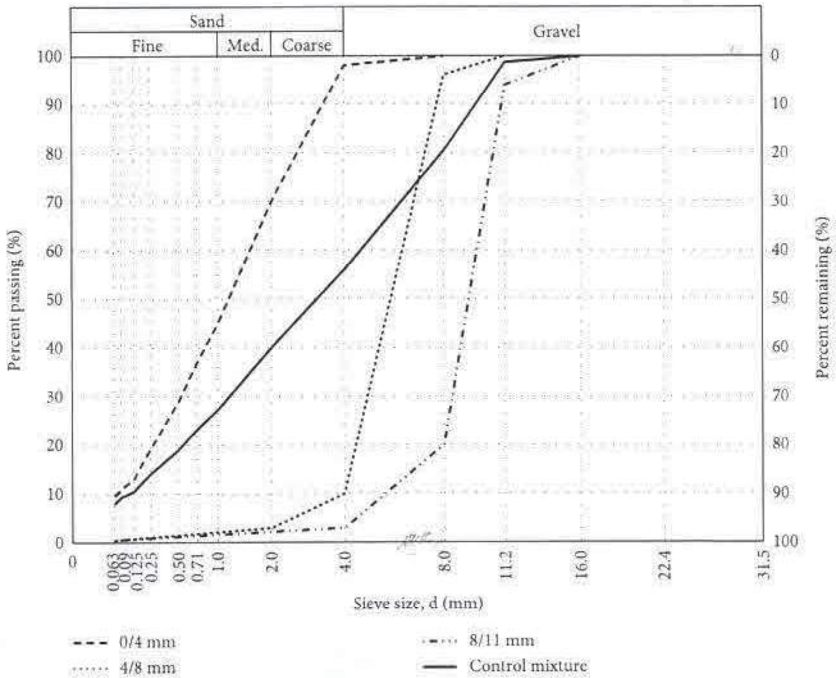

FIGURE 2: Particle size distribution of stone aggregate fractions and control mixture.

TABLE 5: Physical properties of stone aggregate fractions and criteria according to SRPS U.E4.014.

Property	Method	Stone aggregate fraction (mm)					
		0/4		4/8		8/11	
		Value	Criterion	Value	Criterion	Value	Criterion
Content of particles smaller than 0.09 mm (%)	EN 933-1	11	Max 10	0	Max 1	0	Max 1
Density-pycnometer method (Mg/m^3)	EN 1097-6	2.730	—	—	—	—	—
Density-wire basket method (Mg/m^3)	EN 1097-6	—	—	2.724	—	2.719	—
Sand equivalent (%)	EN 933-8	65.4	min 60	—	—	—	—
Fineness modulus	SRPS U.E4.014	2.64	1.95–3.00	—	—	—	—
Water absorption (%)	EN 1097-6	—	—	0.6	Max 1.6	—	—

TABLE 6: Bitumen properties.

Property	Method	Value
Penetration at 25°C (0.01 mm)	EN 1426	61
Ring and ball softening point (°C)	EN 1427	50.1
Penetration index	EN 12591-A	−0.1
Relative density	EN 15326	1.011

the control mixture. The G and K mixtures have balanced values within the group, typically 16.7% lower than the control mixture.

Higher stability and lower flow value of almost all mixtures with fly ash is the result of the stiffening effect that fly ash has on mastic and the mixture, compared with the mineral filler. The effect becomes more pronounced at higher temperatures, which recommends the use of fly ash in warmer climates. Increasing stability and flow reduction is consistent with findings of Androic [20] and Çelik [27] but opposite to findings of Kar et al. [17] who obtained a decrease in Marshall stability and an increase in flow.

3.2. Water Resistance. The ITS test is useful to evaluate the resistance to cracking of asphalt layers, as well as the sensitivity of mixtures to moisture damage. ITSR is determined according to EN 12697-12A [29] as the ratio of ITS of specimens in a water saturated state and specimens in a dry state and is used in this study for the evaluation of moisture susceptibility of all mixtures. The obtained ITS and ITSR values are shown in Table 10. It should be noted that the reported values are averages of three measurements with a very low scatter of results (the coefficient of variation was below 5% for all mixtures).

The ITS of fly ash asphalt mixtures depends on fly ash type and content. The ITS of all P mixtures is consistently higher than the control mixture (up to 9.3%), while for all K mixtures, the ITS is lower than the control mixture (up to 10.3%). For G mixtures, there is a decreasing trend with an increase of fly ash content and for contents below 50% the ITS is higher than for the control mixture, while for higher replacement percentages, it is lower. This significant difference in ITS results can be related to the chemical

Table 7: Coding and composition of asphalt mixtures.

Groups of mixtures	Control mix	P group				G group				K group			
Mixture code	CM	P_{25}	P_{50}	P_{75}	P_{100}	G_{25}	G_{50}	G_{75}	G_{100}	K_{25}	K_{50}	K_{75}	K_{100}
Component material	Material proportion by mass (%)												
Mineral filler	3.78	2.83	1.89	0.94	—	2.83	1.89	0.94	—	2.83	1.89	0.94	—
Fly ash "P"	—	0.94	1.89	2.83	3.78	—	—	—	—	—	—	—	—
Fly ash "G"	—	—	—	—	—	0.94	1.89	2.83	3.78	—	—	—	—
Fly ash "K"	—	—	—	—	—	—	—	—	—	0.94	1.89	2.83	3.78
Stone aggregate 0/4 mm	47.20												
Stone aggregate 4/8 mm	21.71												
Stone aggregate 8/11 mm	21.71												
Bitumen B 50/70	5.60												

Table 8: Testing of volumetric composition with corresponding methods.

Property of asphalt mixture	Method
Asphalt specimen density (mg/m^3)	EN 12697-6-procedure B
Asphalt mixture maximum density (mg/m^3)	EN 12697-5-procedure C
Voids in asphalt specimen (%)	EN 12697-8
Voids in mineral mixture (%)	
Stability (kN)	EN 12697-34
Flow (mm)	

composition of fly ashes and their stiffening effect on asphalt mixtures, which can be assessed based on ΔR&B values. The observed trends are clearly visible in Figure 3 which plots ITS versus fly ash percentage. The full line represents the control mix value, and the blue, red, and green dashed lines represent linear regression relationships for P, G, and K mixtures, respectively.

Fly ash K is predominantly calcic (CaO content is 45.98%) and is characterized by its extremely high ΔR&B value (38.6°C) which indicates its significant stiffening effect of mastic and asphalt mixture, which becomes more brittle, resulting in lower ITS values. Contrary to this, fly ash P is dominantly silicate (SiO_2 content is 42.83%) and has the lowest ΔR&B value (17.0°C). This is consistent with findings of Sharma et al. [14] who tested four fly ashes with high SiO_2 contents, similar to fly ash P, and found that for all four, the ITS was increasing with increasing fly ash content.

The ITSR indicates moisture susceptibility and should be higher than 70–80%, depending on the specification. Figure 4 plots the ITSR values versus fly ash percentage. Similar to Figure 3, the full line is the control mix value and the blue, red, and green dashed lines are linear regression relationships for P, G, and K mixtures, respectively. ITSR for all P and G mixtures is higher than for the control mixture, with an increasing trend with increasing fly ash content, indicating that the use of these fly ashes improves the resistance to moisture of asphalt mixtures. Asphalt mixtures with fly ash G showed superior ITSR due to the high CaO content in this fly ash, which helps establish a good bond between bitumen and aggregate, therefore reducing stripping damage. For fly ash K, the opposite trend is observed and ITSR was decreasing with increasing fly ash content.

3.3. Resistance to Permanent Deformation. Rut resistance is an important parameter for design as well as evaluation of performance of asphalt mixtures. To check the rutting resistance of mixtures, simulation tests were performed using the wheel-tracking test according EN 12967-22 [30].

Table 11 presents the rut resistance of mixtures expressed as the proportional rut depth (PRD, ratio of the rut depth and the slab thickness) and the slope of the wheel-tracking deformation curve, i.e., the speed of increasing deformation expressed as the wheel-tracking slope (WTS) from 10 000 to 20 000 cycles.

The PRD for all mixtures was below 7%, and for most of mixtures below 5%, which is considered acceptable and would result in rut resistant asphalt surface courses.

The trends of PRD depend on fly ash type and content. Figure 5 presents the ratios of PRD values of fly ash mixtures, relative to the PRD value for the control mixture. For fly ash P, there is increasing trend of permanent deformation with increasing fly ash content, and for contents below 50%, the PRD of asphalt mixtures is lower than for the control mixture. The opposite, decreasing trend which indicates improved performance, was observed with fly ash G. If fly ash content is below 50%, PRD is higher, while for higher percentages, it is similar as for the control mixture. The main reason for these trends is particle shape and surface texture. The spherical shape and smooth texture of fly ash P particles result in increased permanent deformation with increased fly ash content, while the presence of irregular particles and rougher surface texture of fly ash G result in a decrease of permanent deformation of asphalt mixtures. For fly ash K, mixed trends were observed, but the performance of all mixtures was superior compared to control mixture.

WTS results generally follow the trends for PRD. Figure 6 presents the ratios of WTS values of fly ash mixtures, relative to the WTS value for the control mixture. For P mixtures, the increase in PRD is accelerated with the increase of fly ash percentage, while for the G group of mixtures, there is a decreasing trend. For K mixtures, the slope of the permanent deformation curve decreases up to 50% of fly ash content, and then, similar to the mixtures in group P, increases with the addition of larger amounts of fly ash.

This finding for fly ash P is consistent with findings of Sharma et al. [14], although it should be noted that they used

Table 9: Volumetric properties, stability, and flow of asphalt mixtures.

Mix	Air voids (%)	Voids in mineral aggregate (%)	Voids filled with bitumen (%)	Bulk density (kg/m^3)	Apparent (maximum) density (kg/m^3)	Stability (kN)	Flow (mm)	Stability/flow ratio (kN/mm)
Control mix	4.7	17.9	73.7	2371	2488	10.2	4.2	2.4
P_{25}	5.5	18.6	70.1	2346	2484	11.3	3.3	3.4
P_{50}	6.7	19.6	65.7	2313	2480	10.0	3.4	2.9
P_{75}	7.1	19.9	64.3	2300	2476	11.8	3.3	3.6
P_{100}	7.7	20.4	62.1	2280	2471	11.2	2.5	4.5
G_{25}	4.6	17.8	74.3	2376	2490	11.5	3.8	3.0
G_{50}	4.4	17.6	75.0	2382	2492	10.7	3.7	2.9
G_{75}	4.6	17.8	74.4	2380	2494	11.0	3.5	3.1
G_{100}	4.4	17.7	75.0	2385	2495	11.3	3.7	3.1
K_{25}	5.2	18.3	71.7	2360	2489	11.1	3.5	3.2
K_{50}	5.0	18.2	72.2	2364	2490	10.4	3.6	2.9
K_{75}	5.7	18.8	69.5	2348	2490	11.0	3.2	3.4
K_{100}	6.0	19.0	68.3	2341	2491	9.8	3.3	3.0
Specification	3.0–6.5	N/A	65–77			N/A	N/A	N/A

Table 10: ITS and ITSR values obtained for the tested asphalt mixtures.

Groups of mixtures	Control mix	P group				G group				K group			
Mixture code	CM	P_{25}	P_{50}	P_{75}	P_{100}	G_{25}	G_{50}	G_{75}	G_{100}	K_{25}	K_{50}	K_{75}	K_{100}
ITS (MPa)	1.07	1.14	1.11	1.17	1.09	1.15	1.11	0.99	1.01	1.00	1.03	0.96	0.97
ITSR (%)	72.4	74.3	76.7	75.8	77.0	78.5	82.5	81.8	82.2	74.9	69.4	64.6	65.1

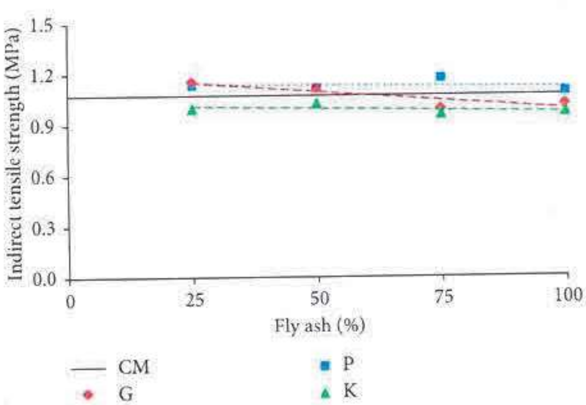

Figure 3: ITS results.

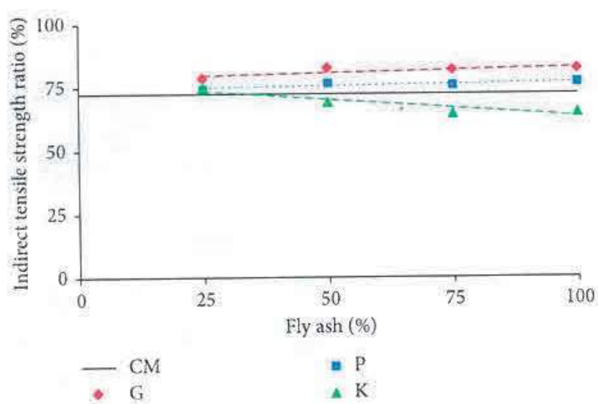

Figure 4: ITSR results.

a static creep test at 30°C and the wheel-tracking test at 60°C is considered more representative for determination of rutting resistance of asphalt mixtures.

4. Conclusions

This paper presents the results of testing the properties of different types of fly ash in order to determine their influence on properties and performance of asphalt mixtures. Also, the results of the volumetric composition tests are shown, as well as the results for water resistance and resistance to permanent deformation of 12 experimental asphalt mixtures in which the mineral filler was replaced with three types of fly ash of different origin (P, G, and K) in four different percentages (25%, 50%, 75%, and 100%).

Based on the research results, the following conclusions can be drawn regarding the properties of asphalt mixtures with fly ash:

(i) The apparent and bulk densities of asphalt mixtures with fly ash depend on the density of fly ash and also on the stiffening effect the fly ash has on bitumen mastics.

(ii) By adding all of the three fly ashes, a satisfactory volumetric composition of asphalt mixtures can be achieved, whereby the density and voids in the mineral mixture (VMA) and air voids in asphalt mixtures (AV) generally depend on the fly ash type and content.

(iii) The G group mixtures have slightly lower AV and VMA and highest value of voids filled with

TABLE 11: PRD and WTS values obtained for the tested asphalt mixtures.

Groups of mixtures	Control mix	P group				G group				K group			
Mixture code	CM	P_{25}	P_{50}	P_{75}	P_{100}	G_{25}	G_{50}	G_{75}	G_{100}	K_{25}	K_{50}	K_{75}	K_{100}
PRD (%)	4.8	3.8	4.6	6.5	5.7	6.2	6.2	4.8	4.7	4.6	3.5	3.8	4.6
WTS (mm/10^3 cycles)	0.06	0.03	0.06	0.06	0.08	0.08	0.08	0.05	0.05	0.06	0.03	0.04	0.07

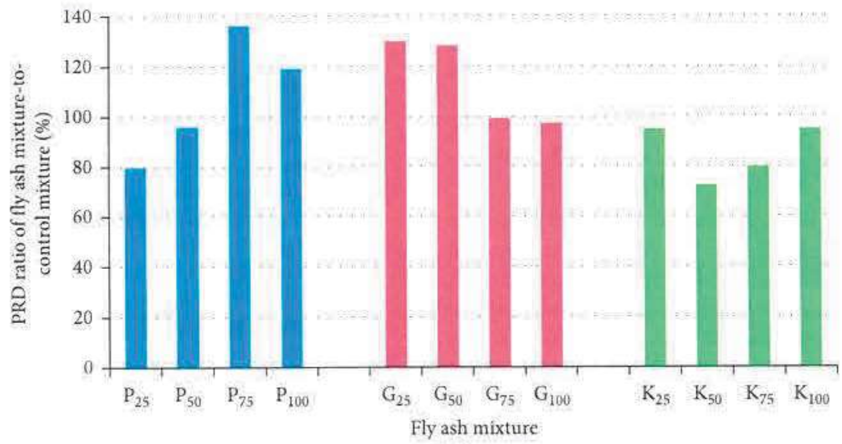

FIGURE 5: PRD results relative to CM.

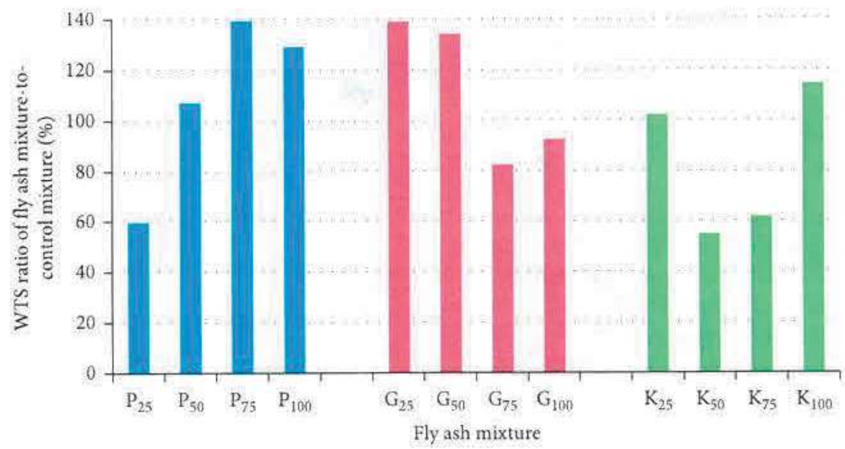

FIGURE 6: WTS results relative to CM.

bitumen indicating that fly ash G, which has finest particle size distribution, can be used as bitumen extender in asphalt mixtures.

(iv) For fly ashes P and K, who have coarser gradation, the AV and VMA are higher for control mixture which indicates that bitumen content should be increased.

(v) The addition of fly ash improves stability of asphalt mixtures for up to 16% and decreases flow for up to 40%. Higher stability and lower flow of almost all mixtures with fly ash are result of substantial stiffening effect of fly ash on asphalt mastics and mixture. The effect becomes more pronounced at higher temperatures, which recommends the use of fly ash as filler in asphalt mixtures in warmer climates.

(vi) The ITS of fly ash asphalt mixtures depends on fly ash type and content and ranges within ±10% of ITS of control mixture. The ITS of all P mixtures is consistently higher than the control mixture (for up to 9.3%), while for all K mixtures, the ITS is lower than the control mixture (up to 10.3%). For G mixtures, there is a decreasing trend with increasing fly ash content, and for contents below 50%, the ITS is higher than for the control mixture, whereas for higher percentages, it is lower. This significant difference in ITS results is related to the chemical composition of fly ashes and their stiffening effect on asphalt mixtures, which can be assessed based on ΔR&B values.

(vii) The ITSR for all P and G mixtures is higher than for the control mixture, with increasing trend with increase of fly ash content, indicating that use of these fly ashes improves resistance to moisture of asphalt mixtures. Asphalt mixtures with fly ash G showed superior ITSR due to high CaO content in this fly ash, which helps establish a strong bond between bitumen and aggregate and therefore reducing stripping damage. For fly ash K, the opposite trend is observed and ITSR was decreasing with increase of fly ash content.

(viii) The PRD for all mixtures was below 7%, and for most of mixtured below 5%, which is considered acceptable and would result in rut-resistant asphalt surface courses. The PRD and WTS mainly depend on particle shape and surface texture. K mixtures showed superior rut resistance, while for fly ashes P and G, rutting resistance depends on the fly ash content and is below 5% for asphalt mixtures with less than 50% of fly ash P and more than 50% of fly ash G.

The findings of this study confirm that it is possible to achieve satisfactory properties and performance of asphalt mixtures with partial replacement of mineral filler with fly ash. The optimal replacement percentage depends on type and chemical composition of fly ash, as well as its properties and affinity to bitumen. Fly ash can be used as a bitumen extender or to improve stability, resistance to moisture, and permanent deformation of asphalt mixture as economic solution instead of using polymer modified bitumen and at the same time reduce negative environmental impact.

Future research will include testing of stiffness and development of master curves for asphalt mixtures with fly ash, as well as testing of asphalt mixtures resistance to low temperatures.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

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ISPITIVANJE SVOJSTAVA ELEKTROFILTERSKOG PEPELA U CILJU PRIMJENE U ASFALTNIM MJEŠAVINAMA

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Rezime: *U radu su predstavljeni rezultati ispitivanja svojstava elektrofilterskog pepela iz termoelektrana TE „Gacko", TE „Pljevlja" i TE"Kosovo B" kako bi se utvrdila njihova podobnost za primjenu u asfaltnoj mješavini AC 11s kao djelimične ili potpune zamjene za kameno brašno – filer. U eksperimentalnom dijelu rada su ispitane fizičko-mehaničke i hemijske osobine elektrofilterskih pepela: hemijski sastav elektrofilterskog pepela (SEM metoda), mineraloški sastav elektrofilterskog pepela (XRD metoda), sadržaj teških metala, stepen radioaktivnosti (gama-spektrometrijska analiza), prisustvo organskih materija, fizičko-mehanička svojstva, kao i fizičke karakteristike pepela. Rezultati izvršenih ispitivanja su pokazali da je primjena ispitivanih elektrofilterskih pepela u svrhu zamjene kamenog brašna u asfalnim mješavinama moguća, bez rizika po ekološku bezbijednost.*

Ključne riječi: *elektrofilterski pepeo, asfaltna mješavina, fizičko-mehaničke i hemijske karakteristike*

TESTING OF FLY ASH PROPERTIES FOR THE APPLICATION IN ASPHALT MIXTURES

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Abstract: *This paper presents the results obtained after testing the characteristics of fly ash from thermal power plants TPP "Gacko", TPP "Pljevlja" and TPP "Kosovo B" in order to investigate whether it is suitable for use in the asphalt mixture AC11s as a partial or complete substitution for stone filler. In the experimental part of this study, the following features of fly ash were examined: chemical composition of fly ash (SEM method), mineral composition of fly ash (XRD method), content of heavy metals, radiation level (gamma-spectrometric analysis), presence of organic matter, physical and mechanical characteristics, and physical characteristics of ash. The results of the performed tests showed that the use of the tested fly ash as a substitute for stone filler in asphalt mixtures is possible and carries no risk in terms of environmental safety.*

Key words: *fly ash, asphalt mixture, physical, mechanical and chemical characteristics*

1. UVOD

Elektrofilterski pepeo nastaje kao nusproizvod pri proizvodnji električne energije u termoelektranama. Tipično se deponuje u okolini elektrana, čime se zauzima značajna površina zemljišta, koje može biti bolje iskorišćeno, a dolazi i do razvejavanja pepela i zagadjenja okolnih područja. Savremeni trendovi u gradjevinarstvu se zasnivaju na tome da se istraži upotreba otpadnih i alternativnih materijala u kompozitnim gradjevinskim materijalima sa ciljem da se dobiju materijali zadovoljavajućih karakteristika, da se smanje štetni efekti deponovanja otpadnih materijala, i da se u isto vrijeme sačuvaju prirodni resursi novih materijala.

Dosadašnja svjetska iskustva ukazuju na to da se ovaj materijal sa posebnim uspjehom može primijeniti u građevinarstvu, od betonskih proizvoda u visokogradnji do ugradnje u sve slojeve puteva u putogradnji. (American Coal Ash Association, 2003). Međutim, istraživanja mogućnosti primjene elektofilterskog pepela (EFP) u asfaltnim mješavinama su znatno manjeg obima u odnosu na istraživanja primjene EFP u ostalim segmentima građevinarstva.

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U studijama koje su objavljene iz ove oblasti rezultati variraju, prije svega u zavisnosti od osobina EFP koji se koristi u eksperimentu, kao i od količine u kojoj je pepeo zastupljen u mješavini.

Mistry i Roy (2016) su ispitivali efekte primjene EFP u gustoj makadamskoj mješavini tražeći optimalnu količinu bitumena (OKB, od 3,5 do 6,5%) pri zamjeni standardnog filera sa 2, 4, 6 i 8% EFP klase F. Za standardni filer izabran je hidratisani kreč. Laboratorijske analize sprovedene na Maršalovim uzorcima su pokazale da zamjenom standardnog filera od 2 do 6% EFP stabilnost sukcesivno raste do 21% u odnosu na kontrolnu mješavinu, dok sa udjelom od 8% EFP opada. OKB se smanjuje sukcesivno sa udjelom EFP od 2 do 6%, dok sa 8% raste, čak i u odnosu na KM. Odnos stabilnosti i tečenja mješavine sa udjelom od 4% EFP ima najmanju vrijednost, ali veću od standardne mješavine, dok mješavine sa 6 i 8% imaju značajan prirast ovog parametra (u prikazanom poretku) I prevazilaze dozvoljenu granicu od 5kN/mm. Generalno, mješavine sa EFP klase F se odlikuju većom čvrstoćom i otpornošću na deformacije.

Al-Suheibani i Tons (1991) su ispitivali uticaj veličine zrna EFP u asfalt betonskim mješavinama sa ciljem zamjene dijela asfaltnog cementa u asfaltnoj mješavini. EFP su klasifikovali u tri veličine: krupnozrni (>44 μm), srednjezrni (1-44 μm) i sitnozrni (<1μm). Sitnozrni pepeo je miješan sa silikatnim dimom (microsilica) krupnoće 0,5 μm u odnosu 50:50 težinski, krupnoće 1 μm, sa ciljem povećenja krutosti mješavine. Uočili su da je srednjezrni EFP najpogodniji kao dodatak bitumenu (krupnozrni daje veći udio šupljina, dok sitnozrni daje prekomjernu krutost mješavini). Oni su zaključili da je dodavanje do 40% pepela u odnosu na zapreminu bitumena, u suvim klimatskim uslovima mješavini dalo bolju čvrstoću na zatezanje, bolju otpornost na kolotrage i duži vijek trajanja. U vlažnim klimatskim uslovima ovaj procenat ne bi trebao da pređe 30%.

Kumar i sar. (2008) su istraživali primjenu EFP u asfalt betonskim mješavinama i utvrdili da se indirektna zatezna čvrstoća asfalt betona povećava sa povećanjem letećeg pepela kao punila. Takva mješavina pokazuje i veću otpornost na pojavu termičkih pukotina, kao i pukotina od zamora. Međutim, otpornost na pojavu kolotraga se smanjuje sa povećanjem učešća letećeg pepela.

Sun i sar. (2011) su upoređivali primjenu izrazito kalcijumskog pepela i mješavine različitih vrsta pepela. Rezultati eksperimenta su pokazali da asfaltne mješavine za noseće slojeve, spravljene sa visokokalcijumskim pepelima imaju veću krutost i veću otpornost na vlagu.

Ali i sar. (2012) su ispitivali asfaltne mješavine sa 50% i 100% zamjene kamenog brašna letećim pepelom klase F, što predstavlja 2%, odnosno 4% u odnosu na ukupnu masu agregata. Mehaničke karakteristike mješavina za habajući sloj su određivali na tri različite temperature (0, 20 i 40°C). Autori su zaključili da se leteći pepeo može koristiti kao punilo (filer) u količini od 2% od ukupne težine agregata kako bi se povećao modul krutosti i veza između zrna agregata i bitumena. Takođe su zaključili da dodavanjem EFP nije značajno povećana otpornost mešavine na kolotrage, kao ni indeks uslužnosti, dok su mješavine sa procentom letećeg pepela većim od 2% pokazale manju otpornost na pojavu površinskih pukotina.

Cmiljanić i sar. (2011) su dali detaljan prikaz istraživanja EFP u Srbiji i njihove podobnosti korištenja u putogtadnji. U radu su prikazani podaci o produkciji EFP u elektranama Srbije kao i njihov prosječni hemijski sastav. Takođe je dat prikaz produkcije pepela u nekim zemljama svijeta u odnosu na površinu država i broj stanovnika.

Mikoč. i Marković (2010) su ispitivali i dokazali podobnost hrvatskih pepela, šljake i amorfnog silicijumdioksida za upotrebu u asfaltnim mješavinama. Fizičko-mehanička svojstva asfalnih mješavina ispitana su na mješavini AC11s. Mješavine spravljene sa EFP u odnosu na kontrolnu mješavinu imale su veću stabinost i gustinu mješavine, kao i šupljine u asfaltnoj mješavini.

Androić i sar. (2013) su ispitivali podobnost dominantno silicijumskog EFP sa malim udjelom kacijum oksida (CaO) kao potpune zamjene za kameno brašno u mješavini AC8 za habajući sloj. Rezultati ispitivanja su pokazali da primjena EFP klase F nije dala poboljšanje u pogledu gustine i udjela šupljina pri optimalnoj količini bitumena.

Jovanović i sar. (2011) su prikazali podobnost korištenja EFP iz Bosne i Hercegovine u asfalnoj mješavini BNS22A. U eksperimentu je umjesto standardnog filera korišten izrazito kalcijumski EFP. Autori su zaključili da je oprimalni udio bitumena (4.45%) kod ove mješavine nešto manji nego kod kontrolne mješavine (4.54%), što se objašnjava činjenicom da su zrna EFP sferičnog oblika i glatke površine u odnosu na standardni filer.

2. MATERIJALI I METODE

U istraživanju su korišćeni uzorci pepela iz TE "Gacko", TE "Pljevlja" i TE "Kosovo B". Sljedi pregled izvršenih istraživanje pepela.

Fizička svojstva elektrofilterskog pepela određena su metodama prikazanim u tabeli 1.

Tabela 1. *Izvršena ispitivanja fizičkih svojstava EFP*

Ispitivanje	Metoda
Granulometrijski sastav	SRPS EN 933-10:2009
Boja, oblik,veličina čestice i površinska tekstura	Skenirajući elktronski mikroskop (SEM), model: JEOL JSM-6610LV.

Mehanička svojstva elektofilterskog pepela određena su metodama prikazanim u tabeli 2.

Tabela 2. *Izvršena ispitivanja mehaničkih svojstava EFP*

Ispitivanje	Metoda
Stvarna zapreminska masa	SRPS EN 1097-7:2008
Šupljine u suvo sabijenom kamenom brašnu	SRPS EN 1097-4:2008
Povećanje tačke razmekšavanja po metodi PK	SRPS EN 13179-1:2008
Bitumenski broj	SRPS EN 13179-2:2008

Prosječan hemijski sastav elektofilterskog pepela određen je metodom skenirajućeg elektronskog mikroskopa, metoda SEM, model JSM-6610LV.

Sadržaj organskih materija u elektrofilterskim pepelima je određen netodom EN 12879:2000.

Mineraloška karakterizacija elektrofilterskog pepela utvrđena je metodom rentgenske difraktometrije XRD na difraktometru za prah: PHILIPS PW 171 pod sledećim uslovima: zračenje sa antikatode bakra: CuKα = 1,54178 Å I grafitni monohromator, napon na cijevi: U = 40 kV, jačina struje: I = 30 mA. Svi uzorci su podvrgnuti istom opsegu ispitivanja 3 – 60° 2θ sa korakom: 0,02°, vremensko zadržavanje po koraku: 2,0 s.

Ekološka podobnost elektofilterskog pepela je sagledana kroz prisustvo teških metala i aktivnost radionukleida. Prisustvo teških metala određeno je određivanjem ukupnog sadržaja elementa I analiziranjem dobijene izlučevine.

Ukupni sadržaj elemenata u uzorcima elektrofilterskig pepela određen je metodom propisanom u standardu MEST EN 13656:2013, osim sadržaja žive koji je određen tehnikom atomske apsorpione spektrofotometrije na čvrstoj fazi. Uzorci pepela ispitivani su na ukupan sadržaj sljedećih elemenata: Sb, As, Ba, Cu, Zn, Cr, Sn, Co, Cd, Ni, Pb, Se, Ag, Hg i Mo.

Izlučevina dobijena metodom opisanom u standradu MEST EN 12457-4:2013 (odnos L/S=10 l/kg) analizirana je na sadržaj istih elemenata za koje je određen ukupan sadržaj, primjenom metoda opisanih u standardima BS EN 12506:2003, BS EN 13370:2003 i EPA 200.7.

Određivanje aktivnosti radionukleida izvršeno je na uzorcima pepela gama-spektrometrijskom analizom. Analiza je izvršena na sistemu sa poluprovodničkim HPGe detektorom firme ORTEC, relativne efikasnosti 41%. Obrada snimljenih spektara obavljena je softverom Gamma Vision 32, Nuclide Navigator. Energetska kalibracija i kalibracija efikasnosti HPGe detektora urađena je korišćenjem tačkastih izvora i multi standarda proizvođača Czech Metrological Institute. Priprema uzorka i analiza je urađena u skladu sa normativima IAEA i EML, prema metodi Measurement of Radionuclides in Food and the Enviroment; Technical Reports series No 295*. Uzorak je doveden do visokog stepena homogenosti i upakovan u standardne Marineli posude od 1 litra, u kojima je i obavljeno snimanje.

3. PREGLED REZULTATA ISTRAŽIVANJA

3.1 Fizička katakterizacija

Rezultati ispitivanja fizičkih osobina su predstavljeni u tabeli 3.

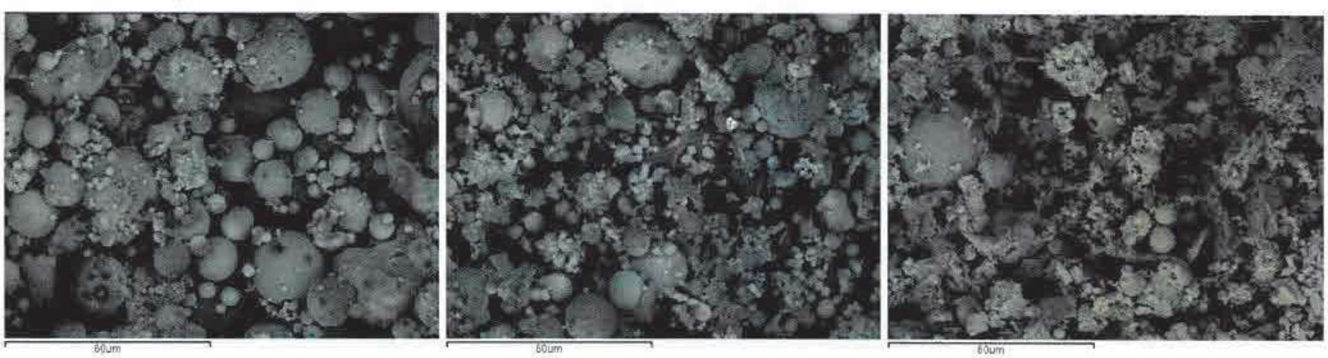
Tabela 3. *Fizičke karakteristike uzoraka elektrofilterskog pepela*

Osobina pepela	Porijeklo pepela		
	TE„Pljevlja"	TE„Gacko"	TE „Kosovo B"
Boja	siva	blijeda mrkožuta	tamna mrkožuta
Oblik čestice	uglavnom sferičan	kombinovano sferičan i nepravilan	kombinovano sferičan i nepravilan
Veličina čestice (µm)	2-30	1-20	1-20
Površinska tekstura	uglavnom glatka	uglavnom hrapava	uglavnom hrapava
Stvarna zapreminska masa (Mg/m³)	2,272	2,966	2,821

Na slici 1. prikazan je izgled uzoraka elektrofilterskog pepela pod elektronskim mikroskopom.

Čestice pepela TE„Pljevlja" su većinom pravilnog sferičnog oblika, veličine 2-30 µm sa manjim udjelom nepravilnih oblika sa malo izraženom tendencijom aglomeracije. Sferične čestice se odlikuju glatkom površinskom teksturom, ali i izvjesnom mjerom poroznosti. Sferične forme su produkti sagorijevanja i to je zapravo amorfna i često šuplja materija, dok su pojedine kristalne faze zastupljene u nepravilnim formama ispitivanog materijala. Sipka, zrnasta struktura i siva boja su najizrazitije vizuelne karakteristike ovog pepela.

TE "Pljevlja" TE "Gacko" TE "Kosovo B"



Slika 1. *Izgled uzoraka elektrofilterskog pepela pod elektronskim mikroskopom*

Oblik čestica pepela TE„Gacko" može se opisati kao kombinacija sferičanih i nepravilnih oblika, veličine 1-20 µm. Površinska tekstura čestica je uglavnom hrapava, dok je pojava aglomeracije vrlo izražena. Stvarna zapreminska masa ovog pepela naveća od svih uzoraka i iznosi 2,966 Kg/m³. Vizuelni opis ovog pepela može se izraziti brašnastom strukturom i blijedom mrkožutom bojom. Oblik čestica pepela TE „Kosovo B" može se takođe opisati kao kombinacija sferičanih i nepravilnih oblika, veličine 1-20 µm, uglavnom hrapave površinske teksture i izražene sklonosti ka aglomeraciji, karakteriše ga tamna mrkožuta boja.

Rezultati ispitivanja granulometrijskog sastava pepela, zajedno sa zahtjevima odgovarajućih standarda, prikazani su u tabeli 4.

Pepeo iz TE "Gacko" u potpunosti zadovoljava zahtjeve u pogledu granulometrijskog sastava prema SRPS EN 13043, dok se pepeo iz TE "Kosovo B" nalazi na granici u pogledu zadovoljenja uslova za prolaz na situ 0.125 mm. Pepeo iz TE "Pljevlja" je krupnozrniji i ne zadovoljava zahtjeve standarda SRPS EN 13043.

EFP iz TE "Gacko" i "Kosovo B" zadovoljavaju u pogledu prolaza na situ 0.063 mm za kvalitet I prema standardu SRPS B.B3.045. Prolaz na situ 0.125 mm je dosta blizak zahtijevanim prolazima na situ 0.09 mm prema standardu, tako da se može smatrati da ova dva pepela generalno zadovoljavaju u pogledu granulometrijskog sastava za kvalitet i prema navedenom standardu. EFP iz TE "Pljevlja" ne zadovoljava u pogledu granulometrijskog sastava ni za kvalitet II.

Tabela 4. *Granulometrijski sastav uzoraka elektrofilterskog pepela i zahtjevi odgovarajućih standarda*

Sito (mm)	Prolaz kroz sito %					
	Porijeklo pepela			Zahtjevi standarda		
	TE "Pljevlja"	TE "Gacko"	TE "Kosovo B"	SRPS EN 13043	SRPS B.B3.045	
					Kvalitet I	Kvalitet II
0,063	42	77	75	70 - 100	60 - 85	50 - 85
0.090					80 – 95	65 - 95
0,125	70	89	84	85 - 100		
0.250					95 – 100	95 – 100
0.710					100	100
2,0	100	100	100	100		

3.2 Mehanička karakterizacija

Kako je ispitivanje pepela sprovedeno prvenstveno sa ciljem određivanja podobnosti za upotrebu u asfaltnim mješavinama, ispitivanjem su bili obuhvaćeni određivanje bitumenskog broja koji predstavlja prividnu viskoznost mješavine kamenog brašna/EFP i vode, kao i povećanje tačke razmekšavanja koja definiše stepen ukrućivanja mješavine kamenog brašna/EFP i bitumena. U tabeli 5 su prikazani rezultati ispitivanja mehaničkih osobina uzoraka elektrofilterskog pepela.

Tabela 5. *Mehaničke osobone uzoraka elektrofilterskog pepela*

Ispitivanje	Metoda	Jedinica mjere	Porijeklo pepela		
			TE "Pljevlja"	TE "Gacko"	TE "Kosovo B"
Šupljine u suvo sabijenom kamenom brašnu/pepelu	SRPS EN 1097-4	%	46	59	55
Povećanje tačke razmekšavanja po mertodi prstena i kuglice	SRPS EN 13179-1	°C	16,95	28,8	38,6
Bitumenski broj	SRPS EN 13179-2	ml	45	27	46

Mineralni fileri imaju šupljine u suvosabijenom stanju (šupljine po Rigdenu) najčešće između 28% i 45%, dok se za krečnjačka kamena brašna one kreću najčešće između 30% i 34% (Grabowski i sar., 2009). Pepeo iz TE "Pljevlja", sa 46% šupljina se odlikuje najmanjim procentom šupljina u zbijenom uzorku i nešto je veći od uobičajenog sadržaja šupljina za krečnjačko kameno brašno, dok pepeli iz TE "Kosovo B" i TE "Gacko" imaju veći sadržaj šupljina (55% i 59%, respektivno), što znači da se nalaze u zoni gornje granice šupljina, odnosno nešto iznad, koje se srijeću kod različitih filera, što nije neuobičajeno za leteće pepele (Voskuilen i Molenar, 1996). Povećani sadržaj šupljina po Rigdenu može imati značajnog uticaja na zahtijevanu količinu bitumena u asfaltnoj mešavini.

Ispitivanje povećanja tačke razmekšavanja po metodi prstena i kuglice ukazuje na povećanje krutosti bitumena 70/100 u koji se doda 37.5 % (v/v) određenog filera. Mineralni fileri imaju povećanje tačke razmekšavanja između 8°C i 25°C, pri čemu je tipična vrijednost 15°C.

Povećanje tačke razmekšavanja po metodi prstena i kuglice je najmanje za pepeo iz TE "Pljevlja" i nalazi se u zahtijevanom rasponu, što znači da mastiks sa tim pepelom iskazuje najmanje povećanje krutosti bitumena, u poređenju sa druga dva pepela, što je povoljno sa aspekta dugotrajnosti asfaltnih mješavina i njihove otpornosti na pucanje. Ova veličina za pepeo iz TE "Gacko" je nesto iznad gornje granične vrijednosti, dok je za pepeo iz TE "Kosovo B" značajno van zahtijevanog raspona.

Bitumenski broj ukazuje na količinu vode potrebnu da se doda pepelu kako bi se postigao prividni viskozitet adekvatan za proizvodnju asfaltnih mješavina. Ovo ispitivanje se posebno primjenjuje u Holandiji (gdje je poznato i kao određivanje Van der Baan-ovog broja) i rezultati su donekle analogni šupljinama po Rigdenu. Za mineralna kamena brašna, bitumenski broj se po pravilu kreće između 40 i 50. Pepeo iz TE "Gacko" ima najmanji bitumenski broj koji je niži u poređenju sa očekivanim vrijednostima za mineralna kamena brašna, dok druga dva pepela imaju bitumenski broj u očekivanim granicama.

3.3 Hemijska karakterizacija

U tabeli 6 prikazan je prosječni hemijski sastav pepela dobijen SEM metodom.

Tabela 6. *Prosječni hemijski sastav elektrofilterskih pepela dobijen metodom SEM*

Ispitivani parametar	Jed. mjere	Ispitivani pepeo		
		TE "Pljevlja"	TE "Gacko"	TE "Kosovo B"
Na_2O	%	0.53	0	0.53
MgO	%	1.18	0.78	3.93
Al_2O_3	%	19.62	3.77	3.51
SiO_2	%	42.83	6.04	16.91
SO_3	%	3.94	10.71	23.2
K_2O	%	2.9	0.22	0.22
CaO	%	21.08	74.44	45.98
TiO_2	%	0.6	0	0
Fe_2O_3	%	7.33	4.04	5.72
Gd_2O_3	%	0	0	0
LOI – gubitak žarenjem na 800°C	%	0.39	0.51	2.55
$R=(SiO_2+Al_2O_3)/(CaO+MgO+Fe_2O_3)$		2.11	0.123	0.37

U pogledu hemijskih karakteristika pepela, u svijetu postoji više različitih klasifikacija.

U ovom radu klasifikacija EFP izvršena je prema tri klasifikacije: ASTM C 618-05, klasifikaciji Rudarskog instituta – Beograd (Grbović i sar., 1986.), kao i prema Ph klasifikaciji koja pepele dijeli na "kisele" i "bazične", (Brzaković i Stamenković, 1971., Mihajlović, 2015.).

Pepele nastale sagorijevanjem uglja u termoelektranama američki standardi klasifikuju kao pepele klase F i pepele klase C. Za ovu klasifikaciju mjerodavan je zbirni sadržaj silicijum dioksida, aluminijum oksida i gvožđevog oksida (SiO_2+ Al_2O_3+ Fe_2O_3), gdje pepeli sa zbirnim sadržajem ovih jedinjenja od min 50% spadaju u klasu C, a sa min 70% spadaju u klasu F. Elektrofilterski pepeo TE "Pljevlja" se može klasifikovati kao pepeo klase F. Prema istom parametru, pepeli iz TE "Gacko" i TE "Kosovo B", iako imaju visok sadržaj CaO - 74.44% i 45.98%, respektivno, ne mogu se svrstati u pepele klase C, jer je njihov zbirni udio SiO_2+ Al_2O_3+ Fe_2O_3 značajno manji od minimalnog zahtjeva od 50%.

Klasifikacija Rudarskog instituta, koja je nastala kao rezultat prilagođavanja raznih klasifikacija uslovima regiona, prepoznaje tri tipa elektrofilterskog pepela: silikatni, kalcujumski i silikatno-kalcijumski.

Pepeo TE "Gacko", sa izrazito nadmoćnom količinom kalcijum oksida (CaO) od 74,44% u odnosu na silicijumov oksid (SiO_2) od 6,04% je tipičan predstavnik kalcijumskih pepela.

Pepeo TE "Kosovo B", iako se odlikuje većim sadržajem CaO (45,98%) u odnosu na SiO_2 (16,91%), može se svrstati u silikatno–kalcijumske pepele sa dominacijom kalcijum oksida.

Pepeo TE "Pljevlja" se odlikuje većim sadržajem SiO_2 (42,83%) u odnosu na CaO (21,08%), ali se takođe može svrstati u silikatno–kalcijumske pepele sa dominacijom silicijum dioksida.

Treba napomenuti da je kalcijum oksid svakako povoljniji, a silicijum dioksid nepovoljniji u pogledu interakcije sa bitumenom u asfaltnim mješavinama, tako da se i na bazi hemijskog sastava može zaključiti da je pepeo iz TE "Gacko" povoljniji za primjenu u asfaltnim mješavinama u odnosu na druga dva pepela.

Za ispitane pepele Ph klasifikacija (Brzaković i Stamenković, 1971., Milosavljević, 1974.), koja pepele dijeli na "kisele" i "bazične", daje najizrazitiju podjelu. "Kiseli" pepeli nastaju sagorijevanjem kamenih ugljeva i u njima preovladavaju SiO_2 (30-50%) i Al_2O_3(15-35%), dok "bazični" pepeli nastaju sagorijevanjem mrkih ugljeva i lignita kod kojih je osnovna komponenta CaO (20-50%). Razlikuju se četiri tipa pepela u zavisnosti od sadržaja SiO_2, Al_2O_3, Fe_2O_3, CaO I SO_3 i to:

- tip 1 - kiseli pepeli sa velikom pucolanskom aktivnošću (SiO_2 > 50%)
- tip 2 - alumosilikatni pepeli sa nižim učešćem SiO_2 (40-50%), Al_2O_3 (17-25%), Fe_2O_3 (18-12%) i CaO (9-22%) koji su pucolanski aktivni
- tip 3 - bazični pepeli sa visokim učešćem CaO (40-46%), Al_2O_3+Fe_2O_3 (6-8%), SiO_2 (2-5%)
- tip 4 - visoko sulfatni i visoko bazični pepeli sa učešćem SO_3 (>26%) I CaO (>33%) koji nemaju pucolanska svojstva.

Tabela 7. *Ph klasifikacija (na "kisele" i "bazične" pepele) ispitanih uzoraka*

	Uslov klasifikacije	TE „Pljevlja"	TE „Gacko"	TE „Kosovo B"
Tip 1	SiO_2 (>50%)	*	*	*
Tip 2	SiO_2 (40-50%)	42,83	*	*
	Al_2O_3 (17-25%)	19,62	*	*
	Fe_2O_3 (18-12%)	7,33	*	*
	CaO (9-22%)	21,28	*	*
Tip 3	CaO (40-46%)	*	74,44	*
	Al_2O_3+Fe2O3 (6-8%)	*	7,81	*
	SiO_2 (2-5%)	*	6.04	*
Tip 4	SO_3 (>26%)	*	*	23,2
	CaO (>33%)	*	*	45,98

* - ne pripada zahtijevanom intervalu

Iz tabele 7 uočljivo je da je pepeo TE "Pljevlja" tipičan predstavnik tipa 2 - alumosilikatni pepeli, pucolanski aktivan, s tim što je sadržaj Fe_2O_3 manji u odnosu na raspon vrednosti za ovaj tip. Pepeo TE "Gacko" spada u pepele tipa 3 – bazične pepele, ali ima značajno veći sadržaj CaO, kao i veoma malo odstupanje parametra SiO_2, , dok pepeo TE „Kosovo B", uz veoma malo odstupanje parametra SO_3, spada u pepele tipa 4 - visoko sulfatne i visoko bazične pepele i nema pucolanskih svojstava.

tabeli 8 dat je prikaz opisanih klasifikacija ispitanih pepela.

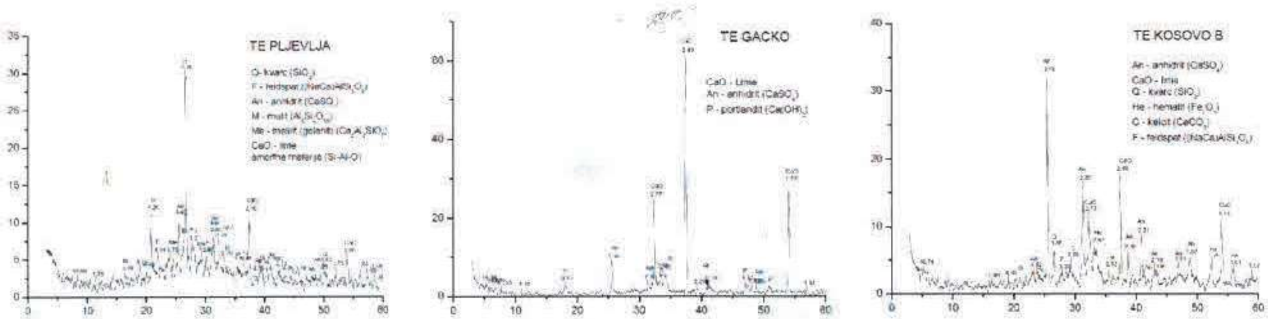
Elektrofilterski pepeo	**SiO_2**	**CaO**	**SiO_2+Al_2O_3+Fe_2O_3**	**Klasifikacija**		
				ASTM C 618	Rudarski Institut Bg	Ph klasifikacija
TE "Pljevlja"	42,83	21,08	69,78 ~ 70	F	silikatno-kalcijumski	alumosilikatni, pucolanski aktivan
TE "Gacko"	6,04	74,44	13,85 << 50	/	kalcijumski	bazični
TE "Kosovo B"	16,91	45,98	26,15 << 50	/	silikatno-kalcijumski	visoko sulfatni i visoko bazični, nema pucolanskih svojstva

3.4 Gubitak pri žarenju (Loss on ignition-LOI)

Značajno hemijsko svojstvo EFP je i gubitak pri žarenju (Loss on ignition-LOI) koji predstavlja mjeru količine ostatka ugljenika koji se zadržao u EFP nakon izlaganja visokoj temperaturi (800°C). Iako ne postoji standard za sadržaj ugljenika ili gubitka pri žarenju (LOI) za pepele klase F i klase C koji se koriste kao mineralno vezivo u asfaltnim mješavinama, smatraju se pogodnim standardi za upotrebu EFP u cementnim mješavinama. Naime, preporučljivo je koristiti pepele sa relativno niskim LOI, manjim od 5% (AASHTO M 295), odnosno manje od 6% (ASTM C 618-05) kako bi se moguća absorbcija bitumena od strane čestica ugljenika svela na minimum. Uočljivo je da je kod sva tri ispitana pepela LOI znatno ispod preporučenih granica, što ove pepele preporučuje za korištenje u eksperimentu. Veličina Loss on ignition (LOI) prikazana je u tabeli 6.

3.5 Mineraloška karakterizacija

Rezultati mineraloških analiza prikazani su na slici 2. Dobijeni podaci položaja difrakcionih maksimuma 2Θ (°), vrijednosti međupljosnih rastojanja d (Å), kao i odgovarajući intenziteti, dati su grafički. Na osnovu dobijenih vrijednosti intenziteta I/I_{max} i međupljosnih rastojanja d i upoređivanjem sa podacima iz literature i JCPDS standardima (Joint Committee on Powder Diffraction Standards) identifikovane su prisutne kristalne faze.



Slika 2. *Difraktogrami uzoraka elektrofilterskih pepela*

Pregled skraćenica prisutnih minerala na difrktogtamima:

Q – kvarc: SiO_2;	An – anhidrit: $CaSO_4$
F – feldpast: (NaCa) $AlSi_3O_8$;	M – mulit: $Al_6Si_4O_{13}$
Me – melilit (gelenit): $Ca_2Al_2SiO_7$;	Kreč - CaO
C – kalcit: $CaCO_3$	Portland $Ca(OH)_2$
Hematit – Fe_2O_3;	Amorfna materija – Si-Al-O

Ispitivanja su pokazala da je u uzorku TE "Pljevlja" prisutna znatna količina amorfne materije. Od kristalnih faza javljaju se silikati: kvarc, feldspatski mineral iz grupe plagioklasa, melilitski mineral tipa gelenita i mulit. Takodje, prisutna je i mala količina anhidrita i slobodnog CaO, dok je u uzorku TE "Gacko" daleko najzastupljenija faza u uzorku slobodni CaO. Prate ga manje količine anhidrita i portlandita. U uzorku TE "Kosovo B" najzastupljenija faza je anhidrit. Prisutna je i nešto manja količina slobodnog CaO. Javlja se i malo hematita, kvarca, kalcita i vrlo malo feldspatskog minerala tipa plagioklasa. Pregled sadržaja amorfne i kristalne faze u elektrofilterskim pepelima prilazan je u tabeli 9.

Tabela 9. *Sasržaj amorfne I kristalne faze u elektrofilterskim pepelima*

Elektrofilterski pepeo	Sadržaj amorfne faze	Sadržaj kristalne faze
TE "Pljevlja"	Značajan	kvarc, feldspatski mineral iz grupe plagioklasa, melilitski mineral tipa gelenita i mulit, mala količina anhidrita i slobodnog CaO
TE "Gacko"	Beznačajan	najzastupljeniji anhidtid, manje količine anhidrita i portlandita
TE "KosovoB"	Beznačajan	najzastupljenija faza je anhidrit, malo hematita, kvarca, kalcita i vrlo malo feldspatskog minerala tipa plagioklasa

3.6 Ekološka karakterizacija

U okviru ekološke karakterizacije ispitani su sadržaj teških metala i stepen radioaktivnosti uzoraka pepela.

Sadržaj teških metala – metoda ukupnog sadržaja elemenata

U tabeli 10 prikazani su rezultati analize totalnog sadržaja metala u uzorcima elektrofilterskih pepela koji su ovom analizom tretirani kao otpadni materijal.

Tabela 10. *Sadržaj teških metala u uzorcima elektrofilterskog pepela*

Parametar	Jed. mjere	Ispitivani pepeo			Oznaka metode
		EFP TE "Pljevlja"	EFP TE "Gacko"	EFP TE "Kosovo B"	
Ukupni sadržaj*					
Antimon (Sb)	mg/kg	<4,0	<4,0	<4,0	MEST EN 13656:2013
Arsen (As)	mg/kg	18,7	10,1	21,5	MEST EN 13656:2013
Barijum (Ba)	mg/kg	2,9	2,4	3,4	MEST EN 13656:2013
Bakar (Cu)	mg/kg	55,3±6,2	20,6±2,3	28,9±3,2	MEST EN 13656:2013
Cink (Zn)	mg/kg	88,9±9,2	38,5±4,0	25,8±2,74	MEST EN 13656:2013
Ukupni hrom (Cr)	mg/kg	124,5	34,5	74,0	MEST EN 13656:2013
Kalaj (Sn)	mg/kg	2,4	1,7	1,5	MEST EN 13656:2013
Kobalt (Co)	mg/kg	18,2±3,2	7,5±1,3	11,1±1,9	MEST EN 13656:2013
Kadmijum (Cd)	mg/kg	1,2±0,2	0,85±0,11	0,13±0,02	MEST EN 13656:2013
Nikal (Ni)	mg/kg	80,8±8,8	48,3±5,3	115,0±12,6	MEST EN 13656:2013
Olovo (Pb)	mg/kg	18,5±2,0	3,5±0,4	7,0±0,7	MEST EN 13656:2013
Selen (Se)	mg/kg	<0,8	<0,8	<0,8	MEST EN 13656:2013
Srebro (Ag)	mg/kg	<0,2	<0,2	<0,2	MEST EN 13656:2013
Živa (Hg)	mg/kg	0,26	0,041	0,112	AMA-112
Molibden (Mo)	mg/kg	<0,8	1,35	0,94	MEST EN 13656:2013
Žareni ostatak na 800° C	%	99,61	99,49	97,45	EN 12879
Silicijum kao SiO_2	%	44,04	5,22	39,63	MEST EN 13656:2013
Kalcijum kao CaO	%	13,47	29,34	26,59	MEST EN 13656:2013

* - preračunato na suvu masu

Dobijeni rezultati za ukupan sadržaj elemenata, u skladu sa Pravilnikom o bližim karakteristikama lokacije, ne pokazuju visoke vrijednosti sadržaja bilo kojeg od njih (manje su od 0,1%), iz čega se zaključuje da analizirani uzroci pepela nemaju karakteristiku opasnosti.

Sadržaj teških metala – metoda sadržaj parametara izlučevine L/S

U tabeli 11 prikazani su rezultati analize sadržaja parametara izlučevine u uzorcima elektrofilterskih pepela koji su ovom analizom tretirani kao otpadni material, kao i dozvoljene količine za različite načine odlaganja.

Sva tri ispitana pepela, sadržaj teških metala u analiziranim izlučevinama, u skladu sa Pravilnikom o bližim karakteristikama lokacije, ispunjavaju uslove za odlaganje otpada kao bezopasnog otpada, što ove pepele preporučuje za upotrebu u asfaltnoj mješavini.

Stepen radioaktivnosti

U tabeli 12 prikazan je sadržaj radionukleida u uzorcima elektrofilterskih pepela.

Za date uslove snimanja spektra, zbog veoma niske koncentracije nekih radionukleida, oni nisu mogli biti detektovani, pa je data njihova minimalna detektabilna vrednost. Izvršena analiza je pokazala da je aktivnost svih analiziranih radionuklida (^{226}Ra, ^{232}Th, ^{40}K, ^{137}Cs, ^{235}U, ^{238}U) u uzorcima pepela ispod maksimalno dozvoljenih vrijednosti, a u skladu sa odredbama datim u Pravilniku o granicama sadržaja radionukleida. Da bi materijal koji sadrži radionukleide bio primijenjen u putogradnji mora da zadovolji uslov da je njegov gama indeks manji od jedan (Kisić (2012), Pravilnik o granicama sadržaja radionukleida).

Tabela 11. *Sadržaj parametara izlučevine u uzorcima elektrofilterskog pepela*

Sadržaj parametara izlučevine L/S=10 l/kg - MEST EN 12457-4	Jed. mjere	EFP TE "Pljevlja"	EFP TE "Gacko"	EFP TE "Kosovo B"	Jed. mjere	Oznaka metode
Antimon (Sb)	mg/kg	<0,05	<0,05	<0,05	(5)[1] (0,7)[2,4,5] (0,06)[6]	EPA 200.7
Arsen (As)	mg/kg	<0,05	<0,05	<0,05	(25)[1] (2)[2,4,5] (0,5)[6]	BS EN 12506:2003
Barijum (Ba)	mg/kg	0,72	0,43	0,30	(300)[1] (100)[2,4,5] (20)[6]	BS EN 12506:2003
Bakar (Cu)	mg/kg	0,06	0,06	0,07	(100)[1] (50)[2,4,5] (2)[6]	BS EN 12506:2003
Cink (Zn)	mg/kg	0,04	0,05	0,05	(200)[1] (50)[2,4,5] (4)[6]	EPA 200.7
Ukupni hrom (Cr)	mg/kg	3,1	0,33	3,5	(70)[1] (10)[2,4,5] (0,5)[6]	EPA 200.7
Kalaj (Sn)	mg/kg	<0,05	<0,05	<0,05		EPA 200.7
Kobalt (Co)	mg/kg	<0,01	<0,01	<0,01		EPA 200.7
Kadmijum (Cd)	mg/kg	<0,005	<0,005	<0,005	(5)[1] (1)[2,4] (3)[5] (0,04)[6]	BS EN 12506:2003
Nikal (Ni)	mg/kg	<0,01	<0,01	<0,01	(40)[1] (10)[2,4,5] (0,4)[6]	BS EN 12506:2003
Olovo (Pb)	mg/kg	<0,05	<0,05	<0,05	(50)[1] (10)[2,4,5] (0,5)[6]	BS EN 12506:2003
Selen (Se)	mg/kg	<0,1	<0,05	<0,05	(7)[1] (0,5)[2,4,5] (0,1)[6]	EPA 200.7
Srebro (Ag)	mg/kg	<0,01	<0,01	<0,01		EPA 200.7
Živa (Hg)	mg/kg	0,001	0,002	0,001	(2)[1] (0,2)[2,4,5] (0,01)[6]	ENV 13370
Molibden (Mo)	mg/kg	<0,5	0,11	0,09	(30)[1] (10)[2,4,5] (0,5)[6]	BS EN 12506:2003

[1] – Uslovi odnosno granične vrijednosti koje mora ispunjavati opasan otpad za odlaganje na deponiji za opasan otpad
[2] – Uslovi odnosno granične vrijednosti za stabilni i nerektivni otpad koji se odlaže na deponiji za neopasan otpad
[3] – Uslovi odnosno granične vrijednosti koje treba da ispunjava komunalni otpad za odlaganje na deponiji za neopasan otpad
[4] – Uslovi odnosno granične vrijednosti koje treba da ispunjava neopasni otpad za odlaganje na deponiji za neopasan otpad
[5] – Uslovi odnosno granične vrijednosti koje treba da ispunjava neopasni otpad sa visokim sadržajem biološki razgradivih materija za odlaganje na deponiji za neopasan otpad
[6] – Uslovi odnosno granične vrijednosti koje treba da ispunjava inertni otpad za odlaganje na deponiji za inertni otpad

Tabela 12. *Aktivnost radionukleida u uzorcima elektrofiltersk*g *pepela*

uzorak / jedinica (Bq/kg)	^{226}Ra	^{232}Th	^{40}K	^{137}Cs	^{235}U	^{238}U
TE "Pljevlja"	107±7	68.1±2.4	678±22	≤ 0.32	7.7±1.8	130±28
TE "Gacko"	486±20	27.8±1.5	66.2±3.8	≤ 0.49	31.5±2.2	548±41
TE "Kosovo B"	48.9±6.7	26.9±1.3	148±6	≤ 0.36	≤ 2.43	67.1±7.0

$$I = \frac{CRa}{700} + \frac{CTh}{500} + \frac{CK}{8000} \quad \text{[1]}$$

Vrijednosti 700, 500 i 8000 označavaju granične, maksimalne vrijednosti radionuklida Ra, Th i K (respektivno) za materijal koji se primjenjuje u niskogradnji (putevi i igrališta), dok su C_{Ra}, C_{Th} i C_K detektovane vrijednosti radijuma, torijuma i kalijuma u uzorku. Kada se dobijene vrijednosti radionukleida uvrste u jednačinu (1), dobija se vrijednost 0,37; 0,75 i 0,142 za pepele Pljevlja, Gacko i Kosovo B , respektivno, što zadovoljava traženi uslov da je manje od jedan. Može se zaključiti da su sva tri pepela bezbjedna sa stanovišta radioaktivnosti i da se mogu koristi u niskogradnji.

4. Diskusija i zaključci

Iako su pogodniji za umješavanje zbog svog sferičnog oblika i glatke površinske teksture, primjena silikatnih filera, usled svoje hidrofilnosti i slabog afiniteta prema bitumenu, zna da rezultira slabijom otpornošću prema ljuštenju i treba biti oprezan u pogledu njihove primjene zbog relativno slabije adhezije čestica i bitumena. Sa druge strane, fileri sa visokim udjelom poroznog krečnjaka, zbog svoje osobine da jako upijaju uljne komponente bitumena, mogu negativno uticati na svojstva bitumena i bitumenskih mješavina (Bedaković 1964). Pepeli sa umjerenim sadržajem kreča (CaO) mogu se koristitii kao dodatak za poboljšanje adhezije agregata i bitumenskog veziva u asfaltnim mješavinama. Takođe pozitivno utiču i na stabilnost asfaltne mješavine (AASHTO M 17-07 (2007). Pepeli koji se ne mogu jasno specifikovati poznatim klasifikacijama mogu naći svoje mjesto u primjeni za stabilizaciju gornjeg i donjeg nevezanog nosećeg sloja i posteljice (Edil 2006). Može se reći da elektrofilterski pepeo TE "Pljevlja" spada u grupu pepela F klase, koji se zbog svoje pucolanske aktivnosti uglavom preporučuju za upotrebu u cementnoj industriji. Takođe, prisustvo amorfne faze u pepelu TE "Pljevlja" treba da obezbujedi veću reaktivnost u odnosu na prisutne kristalne veze, što ovaj pepeo preporučuje kao zamjenu za prirodne materijale u mješavinama za sintezu portland-cementa. Međutim ovaj pepeo sadrži i značajan procenat kalcijum oksida (CaO – 21,08 %), što kod pepela F klase uglavnom iznosi 1-12% (Knežević, 2014). Prisustvo kalcijum oksida u ovoj mjeri čini ga podobnim za upotrebu u asfaltnim mješavinama. Pepeo TE "Pljevlja" je pucolanski aktivan alumosilikatni pepeo.

Visok udio kreča (CaO) u pepelu TE "Gacko" (74.44%) i nešto manji u pepelu TE"Kosovo B" (45.98%) treba da bude preporuka za korištenje ovih pepela u asfaltnim mješavinama zbog svoje osobine poboljšavanja adhezije agregata i bitumenskog veziva. Ipak, pepeli TE "Gacko" i TE "Kosovo B" ne zadovoljavaju ASTM standard po pitanju minimalnog udjela $SiO_2+Al_2O_3+Fe_2O_3$ od 50%, pa se ne mogu svrstati u C klasu pepela. Pepeo TE "Gacko" je bazični pepeo sa visokim učešćem CaO, dok je pepeo TE "Kosovo B" visoko sulfatni i visoko bazični pepeo bez pucolanskih svojstava.

Veće povećanje krutosti, na koje indikuje značajno veća tačka razmekšavanja kod pepela iz TE "Kosovo B" može biti povoljna samo u inicijalnim fazama eksplatacije kolovoza, u pogledu povećanja otpornosti asfaltne mješavine na trajnu deformaciju, ali dugoročno može dovesti do veće krutosti i osjetljivosti na pucanje asfaltnih mješavina.

Uzimajući u obzir procenat zastupljenosti elektrofilterskog pepela u asfaltnim mješavinama (max 4%), i oštre zahtjeve Pravinika prema kojem je vršeno ispitivanje, može se zaključiti da rezultati analize pokazuju da svi ispitani EFP, planirani za upotrebu u asfaltnim mješavinama u svrhu djelimične ili potpune zamjene za kameno brašno, ispunjavaju sve uslove u pogledu sadržaja teških metala i predstavljaju bezbjedan materijal za primjenu u asfaltnim mješavinama.

Kako je gama indeks svih ispitanih pepela manji od jedan, može se zaključiti da su sva tri pepela bezbjedna za ugradnju u asfaltnu mješavinu, sa stanovišta radioaktivnosti. Fizičko-mehaničke osobine sva tri ispitana pepela su u zadovoljavajućem opsegu, kao i prisustvo organskih materija iskazano kroz veličinu gubitka pri žarenju.

Na osnovu osobina elektrofilterskih pepela koje su bile predmet ispitivanja predstavljenih u ovom radu, može se zaključiti da su ispitani pepeli pogodni za upotrebu u asfaltnim mješavinama kao djelimična ili potpuna zamjena za kameno brašno, bez rizika po ekološku bezbijednost.

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