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Application of Biomass Ashes as Supplementary Cementitious Materials in the Cement Mortar Production

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Abstract**—**The production of low cost and environmentally friendly products represents an important step for developing countries. Biomass is one of the largest renewable energy sources, and Serbia is among the top European countries in terms of the amount of available and unused biomass. Substituting cement with the ashes obtained by the combustion of biomass would reduce the negative impact of concrete industry on the environment and would provide a waste valorization by the reuse of this type of by-product in mortars and concretes manufacture. The study contains data on physical properties, chemical characteristics and pozzolanic properties of obtained biomass ashes: wheat straw ash and mixture of wheat and soya straw ash in Serbia, which were, later, used as supplementary cementitious materials in preparation of mortars. Experimental research of influence of biomass ashes on physical and mechanical properties of cement mortars was conducted. The results indicate that the biomass ashes can be successfully used in mortars as substitutes of cement without compromising their physical and mechanical performances.

Keywords**—**Biomass, ash, cementitious material, mortar.

I. Introduction

BIOMASS is one of the greatest, but poorly utilized, renewable energy sources and accounts for more than 4% of the total energy consumption in the European Union [1]. Various types of biomass, from agro-industrial processes, generate considerable quantities of ash through their combustion (as rice husk ash, cob corn ash, wheat straw ash (WSA), etc.) which under certain conditions (chemical configuration, level of fineness) can have a similar cementitious property to coal fly ash. Biomass ashes, which contain a large amount of silica in amorphous form, can potentially be used as pozzolanic materials. However, unlike coal, biomass meets the principles of sustainable development; it is CO_2 neutral fuel, as the amount of CO_2 absorbed by a plant during lifecycle and the amount released by the plant during its thermal decomposition are equal.

It is estimated that, every year in Serbia, a total amount of 12.5 million tons of biomass is produced, of which 9 million tons (72%) come from Vojvodina. Crop husbandry in Vojvodina generates more than 6 million tons of biomass waste every year, most of which is corn residue (stalk and cobs) with 54.8%, then wheat straw with 18.7% and sunflower residue (stalk, head and husk) with 13% [2]. Fig. 1 illustrates the availability of biomass in Serbia.

Harvested quantities of different types of crops in Vojvodina and Serbia, in 2016, in tons, are given in Table I.

TABLE I
HARVESTED QUANTITIES OF DIFFERENT TYPES OF CROPS IN SERBIA IN 2016 [4]

Type of crop	Harvested quantities in Serbia in 2016 (tons)
Wheat	2 884 537
Corn	7 987 479
Barley	395 501
Rye	14 200
Oats	81 344
Soya	576 446
Sunflower	621 127
Rape seed	39 404
Sugarcane	2 683 860
Clover	291 365

Biomass is partially used as an energy source, but the ashes, generated by the combustion of biomass, are rather disposed on the landfill then further utilized. In order to promote sustainable development, there is a need to find sustainable ways of proper management of those ashes. The main idea of the IPA Interreg project Agricultural Waste – Challenges and Business Opportunities (Eco Build) is to introduce biomass ash use in civil engineering through composite building materials. Within IPA project, a group of researchers from Novi Sad - Serbia, has investigated the availability and possible application of biomass ashes in Vojvodina. For that purpose, samples of two biomass ashes (WSA and mixture of wheat and soya straw ash) were collected, and their basic physical and pozzolanic properties were tested. Furthermore, these ashes were used as supplementary cementitious materials in cement mortars in order to determine their influence on mechanical properties of mortars.

II. Utilization of WSA as SCM Material: Literature Review

Wheat is one of the primary sources of food for 2.45 billion people [5]. In 2017, the annual global production of wheat was

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around 730 million tons, whereas the global estimate of wheat straw is at around 534 million tons [6]. The current use of wheat straw is associated with energy source, pulp and paper, nano-materials, bioethanol, fertilizer, as addition in mud for mud houses. In developing countries wheat straw is burnt in open field causing environmental and health problems.

Considerable amounts of WSA are generated in the process of biomass combustion. This ash represents a potential source for the production of mineral binders, which mainly depends on its chemical composition.

WSA is, among biomass ashes, from the aspect of application as a binder in cement-based composites, investigated in a relatively small extent. Several experimental studies have been conducted on pastes and mortars where WSA was used as a replacement of a part of cement or sand, and some of the basic properties of cement composites in fresh and hardened state were tested - Table II.

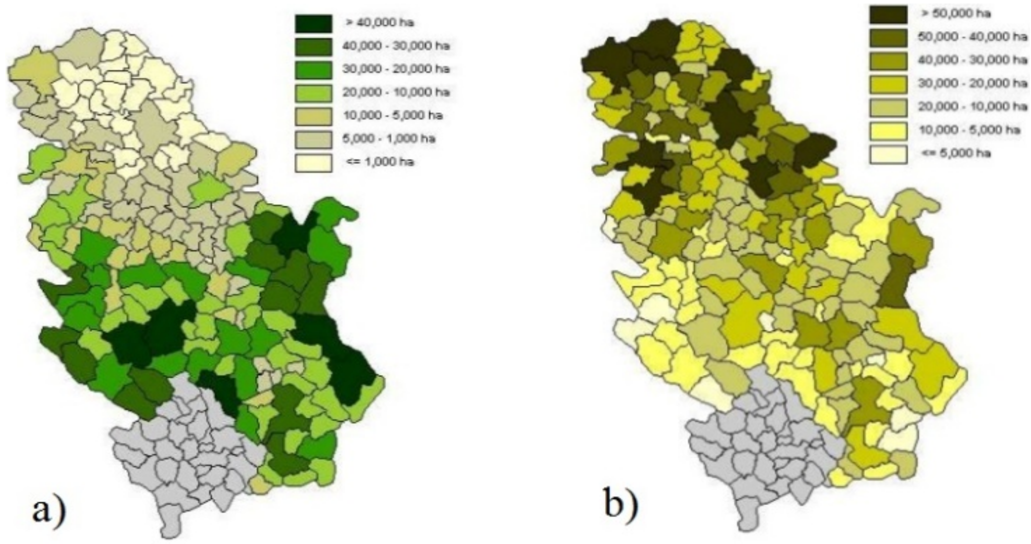

Fig. 1 Biomass in Serbia: a) areas under forests, b) agricultural land [3]

TABLE II
THE REVIEW OF THE INVESTIGATION OF CEMENT COMPOSITES WITH WSA AND TESTED PROPERTIES

Author	Composite	Tested properties	Cement replacement level (%)
Jankovský et al., 2017 [7]	paste	setting time compressive strength flexural strength	20
Khushnood et al., 2015 [8]	paste	modulus of elasticity compressive strength flexural strength water absorption acid resistance	10
Goyal et al., 2007 [9]	mortar	compressive strength flexural strength	15
Sani A. et al. [10]	mortar	compressive strength flexural strength	50
Dehane et al., 2015 [11]	mortar	compressive strength water absorption	37,5
Biricik et al., 2000 [12]	mortar	acid attack resistance	24
Mabil M.A. et al., 2002 [13]	mortar	compressive strength flexural strength tensile strength	10,9

According to the results of an experimental research, conducted by Radeka et al. [14], the chemical composition of WSA (obtained in Serbia) is characterized by high content of K_2O (18%), low content of Al_2O_3 (0.2%) and significant S_iO_2 content (52%). This chemical composition most likely leads to the formation of glassy phase on relatively low temperatures. Formation of the glassy phase and not so high temperature in the combustion boiler (760 °C) can result in a higher amount of amorphous phase, greater pozzolanic activity and possible application as a binding material in cement-based composites.

Jankovský et al. [7] evaluated the possibility of using WSA ash as a pozzolanic material. The chemical composition of ash is characterized by high alkali content (K_2O=11%), which is estimated to be very high, due to the possible alkali-silicate reaction in cementitious composites. In order to reduce the amount of alkalis in cement blends, water treatment of WSA was proposed as an effective tool to meet requirements on alkali content. Replacing cement with WSA improved the workability of the paste; the setting time was significantly prolonged (due to slower hydration and pozzolanic reaction), whereas the bulk density and the total open porosity of the paste with WSA decreased in relation to the reference cement paste. The use of ash in percentages of 10 and 15% proved to be favorable from the aspect of all the tested mechanical properties.

Dehane et al. [11] studied the effect of replacing cement with WSA on compressive strength and water absorption of cement mortars. Ash content was varied in replacement percentages: 12.5, 25, and 37.5% by weight of cement. At the age of 28 days, the strength of the mortar W12.5 exceeded the strength of the reference mortar, and this difference, due to the pozzolanic reaction, increased over time. The mortars W12.5 and W25 were characterized by a smaller water absorption compared to the absorption of the reference mortar at the age of 28 days (due to the filler effect of small WSA particles). In another study, conducted by Goyal et al. [9], it was reported that the use of 7.5% WSA can be the optimum proportion to enhance the strength of cement mortar.

Biricik et al. [12] investigated magnesium and sodium sulfate resistance of mortars containing WSA. It was reported that WSA replacement affected beneficially compressive strength of mortars in sodium sulfate solutions. The strengths of mortars generally increased in magnesium sulfate solutions during the tests. The only exception is decreasing strength, after 56 days in solution at a concentration of 40,000 mg/l at the 24% replacement level. Similar research was conducted by Khushnood et al. [8]. Acid attack resistance of self-compacting cement paste containing WSA was evaluated. WSA formulation was found to be effective in consuming free lime and showed significant decrease in porosity with time, which in turn improved the resistance against water absorption and acid attack.

Results of the listed research indicate that WSA can be effective in improving the ultimate strength of mortar, due to the pozzolanic reaction and filler effect and, used as an alternative binder, it can offer durable and environmental friendly option to construction industry. Substituting cement in mortar, with the biomass ashes, as CO_2 neutral fuel, would reduce the emission of CO_2 during the manufacturing of cement and the consumption of non-renewable resources as raw materials and would give a new use-value and the possibility of economic valuation of ashes as a new product. This research was carried out to evaluate the feasibility of WSA ash in cement mortar formulations and define optimal cement replacement level, from the aspect of their mechanical properties.

III. Materials and Experimental Methods

Biomass ashes, used in this experimental study, were obtained from two manufacturers of biomass ash in Serbia. In the first phase of the experiment, the following properties of ashes were tested:

- Basic physical properties (density, specific surface area),
- Chemical composition,
- Pozzolanic activity,
- Activity index,
- Initial and final setting time,
- Soundness.

After determining of physical, chemical and pozzolanic properties of biomass ashes, they were used as supplementary cementitious materials for preparation of mortars. Replacement of cement with biomass ashes was varied up to 50% (10, 20, 30 and 50%). Mortar compressive strength at different ages (28, 56, and 91 days) was tested.

A. Materials

For experimental investigation of biomass ashes as supplementary cementitious materials, the following materials were used:

- Portland cement CEM I 42,5R (Lafarge-BFC Serbia),
- WSA, “Mitrosrem”, Sremska Mitrovica, Serbia,
- Mixture of wheat and soya straw ash (WSSA), “Soya Protein”, Bečej, Serbia,
- CEN sand,
- Deionized water.

B. Methods

Specific surface of biomass ashes is determined according to Blaine air permeability method given in standard SRPS EN 196-6 [15].

The chemical composition of biomass ashes is determined using EN 196-2 [16] and ISO 29581-2 [17].

The pozzolanic activity is studied on specimens prepared according to the procedure given in SRPS B.C1.018:2015 [18]. The pozzolanic activity is determined based on 7-day compressive strength.

Activity index, initial and final setting time and soundness of biomass ashes are tested in accordance with the procedure given in SRPS EN 450-1:2014 [19].

Compressive strength of all types of mortars is tested according to SRPS EN 196-1:2008 [20]. Samples for testing of compressive strength were cured in tap water to the anticipated age.

IV. Preparation and Testing of Pozzolanic Properties of Biomass Ashes

A. Preparation

Preparation of biomass ashes included sieving and grinding phase. Both types of ashes were roughly sieved (through a 4-mm sieve) in order to separate unburnt straw and other large impurities. Then, ashes have been grinded in ball mill for 6 hours in order to obtain material with high specific surface area. Appearances of the ashes before and after grinding phase are shown in Figs. 2-3.

Fig. 2 View of WSA biomass ash before sieving and after grinding

Fig. 3 View of WSSA biomass ash before sieving and after grinding

B. Physical Properties and Chemical Composition

Both types of tested biomass ashes have similar densities, cca 2.400 kg/m^3. After grinding phase, specific surface (Blaine) of biomass ashes exceeded 5.500 cm^2/g. Table III shows physical properties of tested biomass ashes.

Content of SiO_2, Al_2O_3, Fe_2O_3 oxides has the greatest importance for potential pozzolanic materials. The chemical composition of tested biomass ashes is given in Table IV. Both types of ashes have relatively high values of the total amount of those oxides, exceeding 50%.

TABLE III
PHYSICAL PROPERITES OF TESTED BIOMASS ASHES

Ash	Density (kg/m^3)	Specific surface (Blaine) (cm^2/g)
WSA	2.380	5.800
WSSA	2.370	5.500

TABLE IV
THE CHEMICAL COMPOSITION OF TESTED BIOMASS ASHES

Ash (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
WSA	53.21	4.00	2.69	13.45	1.90	0.41	12.05	10.19
WSSA	51.93	0.19	1.39	14.28	2.07	0.43	18.43	9.27

C. Testing of Pozzolanic Properties

The pozzolanic activity was studied on specimens prepared according to the procedure given in SRPS B.C1.017-2001. Standard mortar prisms were prepared with biomass ash, slaked lime and quartz sand (Figs. 4 and 5). The pozzolanic activity was determined based on 7-day compressive strength.

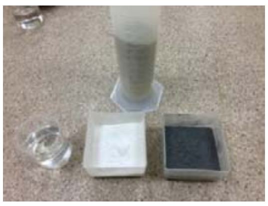

Fig. 4 Component materials for testing of pozzolanic activity

Fig. 5 Standard mortar prisms for testing of pozzolanic activity

Testing of pozzolanic properties showed that WSA has pozzolanic activity of Class 10, while WSSA has pozzolanic activity of Class 5. Results are given in Table V.

TABLE V
POZZOLANIC ACTIVITY OF TESTED BIOMASS ASHES

	$f_{zs,sr}$ [MPa]	$f_{zs,min}$ [MPa]	$f_{p,sr}$ [MPa]	$f_{p,min}$ [MPa]	**CLASS**
WSSA	3.6	3.3	9.3	8.75	**5**
WSA	3.4	3.3	11.0	10.3	**10**

D. Testing of Activity Index

Activity index was studied on specimens prepared according to the standard EN 450-1. Activity index is calculated as a ratio (in percent) of the compressive strength of standard mortar bars, prepared with 75% test cement plus 25% fly ash by mass, to the compressive strength of standard mortar bars prepared with 100% test cement, when tested at the same age. Preparation of standard mortar bars and determination of the compressive strength was carried out in accordance with EN 196-1.

According to the criteria, given in standard EN 450-1, the activity index at 28 days and at 90 days shall not be less than 75% and 85%, respectively. Both types of biomass ashes fulfilled criteria. Results are given in Table VI.

TABLE VI
ACTIVITY INDEX OF TESTED BIOMASS ASHES

	$f_{p,sr,28d}$ [MPa]	INDEX, 28d (%)	$f_{p,sr,90d}$ [MPa]	INDEX, 90d (%)
C	50.26	-	54.53	-
WSSA	40.73	81.04	46.87	86.00
WSA	52.39	104.24	58.96	108.12

E. Initial and Final Setting Time

The initial setting time was determined on a 25% fly ash plus 75% test cement paste (both by mass) in accordance with EN 196-3 and EN 197-1. According to these standards, the initial setting time shall not be more than twice as long as the initial setting time of a 100% (by mass) test cement paste (criterion 1). The initial setting time, as specified in EN 197-1, shouldn't be shorter than 60 minutes - criterion 2 (for cement type CEM I 42,5R). Both types of biomass ashes fulfilled criteria. Results are given in Table VII.

Both initial and final setting times were prolonged in relation to the ordinary Portland cement. This is due to the low rate of hydration in the paste containing biomass ashes. WSSA ash had considerably increased initial setting time, which may be result of remained organic substances – straw remains.

TABLE VII
INITIAL AND FINAL SETTING TIME OF BIOMASS ASHES

	The initial setting time (minutes)	The final setting time (minutes)	Criterion 1	Criterion 2
C	230	275	fulfilled	fulfilled
WSA	210	320	fulfilled	fulfilled
WSSA	350	420	fulfilled	fulfilled

F. Soundness

The soundness was determined on 30% fly ash plus 70% test cement (both by mass) in accordance with EN 196-3 and EN 197-1. According to these standards, the soundness shall not be greater than 10 mm. As both types of biomass ashes had negligible expansion, up to 1mm, criteria are fulfilled. Results are given in Table VIII.

TABLE VIII
SOUNDNESS OF BIOMASS ASHES

	Expansion (mm)	Criterion
C	0	fulfilled
WSA	0.5	fulfilled
WSSA	1	fulfilled

V. PREPARATION AND TESTING OF CEMENT MORTARS WITH BIOMASS ASHES

A. Mix Proportion of Cement Mortars

Both types of biomass ashes were used as supplementary cementitious materials in preparation of mortars. Compressive strength of all types of mortars was tested according to EN 196-1 at different ages (28, 60, and 90 days).

The experimental study was carried out on nine mortar mixtures. Referent mortar (C) was prepared with Portland cement as binder, standard sand and water. In four mortar mixtures (WSA10, WSA20, WSA30, and WSA50), the part of cement was replaced with 10%, 20%, 30% and 50% of WSA, by mass. In remaining four mixtures (WSSA10, WSSA20, WSSA30, and WSSA50), the part of cement was replaced with mixture of wheat and soya straw ash, likewise. The mix proportions are given in Table IX.

From each mortar mixture, three standard prisms were made.

B. Results and Discussion

Compressive strength is considered as one of the most important properties of cement-based composites and a major indicator of general quality. The factors affecting the compressive strength of mortars with WSA are water to binder ratio, curing duration and WSA replacement level. In this study, the water to binder ratio was kept constant at 0.5 for all mixtures. The average compressive strength results are given in Table X.

TABLE IX
LABELS AND COMPOSITIONS OF MORTAR MIXTURES

Mortar type	C	WSA 10	WSA 20	WSA 30	WSA 50	WSSA 10	WSSA 20	WSSA 30	WSSA50
Cement, g	450	405	360	315	225	405	360	315	225
WSA, g	-	45	90	135	225	-	-	-	-
WSSA, g	-	-	-	-	-	45	90	135	225
Water, g	225	225	225	225	225	225	225	225	225
CEN sand, g	1350	1350	1350	1350	1350	1350	1350	1350	1350

TABLE X
COMPRESSIVE STRENGTH OF TESTED MORTARS, IN MPA

days	C	WSA10	WSA20	WSA30	WSA50	WSSA10	WSSA20	WSSA30	WSSA50
28	54.84	56.30	55.38	50.96	40.09	40.89	39.22	38.49	26.46
56	56.84	60.57	57.81	58.07	42.05	42.08	41.88	39.06	32.91
91	57.65	62.08	60.68	64.25	45.78	44.64	43.02	40.57	40.00

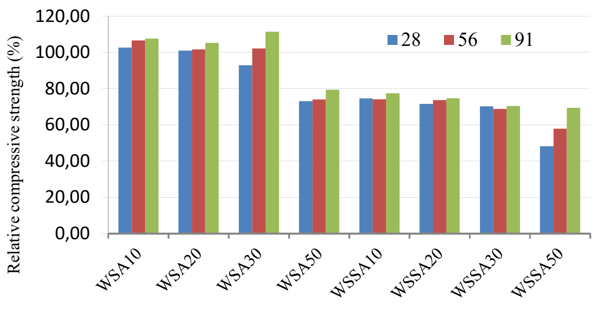

Fig. 6 Relative compressive strength of mortars with biomass ashes at the different ages

The partial replacement of Portland cement with WSA at levels of 10% and 20% resulted in slightly higher compressive strength than that of the reference mortar at the age of 28 days. The increases are 2.6% and 1%, respectively. Possible cause of strength increase is filler (packing) effect of WSA, which has smaller particle size than cement. However, lower compressive strengths have been observed for mortars containing WSA at replacement levels 30% and 50%. This 28-day reduction in compressive strength can be attributed to delayed pozzolanic activities in WSA mortars. Relative compressive strength of mortar mixtures with biomass ashes at the different ages is shown in Fig. 6.

After 28 days, due to pozzolanic reaction of biomass ashes, compressive strength of mortars containing WSA has been gradually increased. It is noteworthy that the compressive strength of the mortars with WSA ash at replacement levels up to 30% was higher than that of the reference mortar, particularly at the age of 91 days. Interestingly, the 91 day-compressive strength of the mortar with 30% WSA reached 64 MPa, which is higher than strength of the reference mortar and mortars containing 10% and 20% WSA. The compressive strengths at 91 days of WSA10, WSA20, WSA30 and WSA50 mortars were 108%, 105%, 111% and 80% of the reference mortars, respectively.

Regarding WSSA ash influence on the mechanical properties of cement mortars, it can be observed that the compressive strength generally declined at all ages with increasing its content. At the age of 28 days, compressive strengths of WSSA10, WSSA20, WSSA30 and WSSA50 mortars were 75%, 72%, 70% and 48% of the reference mortars, respectively. Up to the age of 91 days, WSSA mortars gained their compressive strength 9%, 10%, 5% and 51% in relation to their 28-day compressive strength, confirming their pozzolanic properties. This increase is higher than increase of compressive strength of reference mortar (5%). The mortar mixture WSSA50 showed the highest compressive strength increase, which may be the result of filler (particles packing) effect, in addition to the pozzolanic reaction of biomass ash. Further investigation is needed regarding these causes.

VI. Conclusion

The following conclusions are drawn based on the test results of this study:

- Relatively high silica content in WSA makes it suitable pozzolanic material for long term strength development in cement mortars,
- Both types of ashes: WSA and WSSA fulfill criteria for pozzolanic materials, given in EN 450-1, including: activity index, setting time and soundness.
- WSA prolongs the setting time of the mortar and, therefore, it is a retarder, which may be beneficial for massive concrete structures, as well as concreting in summer at higher temperatures. This behavior may be due to the low rate of hydration in the paste containing WSA.
- It can be concluded that inclusion of WSA enhances compressive strength of cement mortars up to 90 days due to pozzolanic activity and micro-filling ability of the biomass ash. The compressive strength of mortars with WSSA declines at all ages with increasing its content, which is the result of lower pozzolanic activity of soya straw ash in WSSA.
- Based on the obtained results, WSA can be used to replace Portland cement up to 30% replacement level without any adverse effect on the mechanical properties of mortar.
- WSSA mortars achieved compressive strength around 40MPa (reduction of cca 25% in relation to reference mortar compressive strength), at the cement replacement level up to 30%, thus showing satisfactory mechanical properties and confirming the possibility of their application in some cement-based composites.

The results prove that it is possible to obtain WSA-based ECO mortar with comparable or better mechanical properties than those of the reference mortar with a lower consumption of cement, thus reducing the CO_2 emissions during the production of cement and meeting the principles of sustainable development.

Utilization of biomass ashes would provide a new use-value and the possibility of economic valuation of ashes as a new product. All these effects would constitute a strong impetus for the creation of conditions for the integrated management of large quantities of ash from biomass originating from agriculture in Serbia

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