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## Self-compacting concrete using pretreated rice husk ash

Самоуплотняющийся бетон с использованием  
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**Abstract.** Self-compacting concrete was obtained by partially replacing Portland cement with a previously prepared rice husk ash. Preliminary preparation included the thermal treatment of the ash under various conditions. The optimum technology of preparation, allowing to receive a homogeneous concrete mix is revealed. All concrete mixtures were designed in such a way as to have a slump flow of  $680 \pm 30$  mm in diameter, which was achieved by using different dosages of a superplasticizer based on polycarboxylate ether. All mixtures with the replacement of cement by ash to 25 % meet the requirements for rheological characteristics and resistance to segregation. The bulk density for the samples with 10 %, 15 %, 20 % and 25 % of the rice husk ash was reduced by 3.19 %, 5.18 %, 5.58 and 6.37 % respectively, compared to the samples without ash. An increase in the rice husk ash content led to a decrease in the early mechanical properties, while the final strength of self-compacting concrete containing ash was comparable to conventional samples. This was achieved due to the pozzolanic activity of the ash. Inclusion of rice husk ash reduced the amount of portlandite in the system by obtaining an additional C-S-H gel, which led to matrix compacting and blocking of networks with open porosity.

**Аннотация.** Самоуплотняющийся бетон получен путем частичной замены портландцемента предварительно подготовленной золой рисовой шелухи. Предварительная подготовка включала в себя термическую обработку золы при различных условиях. Выявлена оптимальная технология приготовления, позволяющая получить однородную бетонную смесь. Все бетонные смеси были спроектированы таким образом, чтобы иметь осадку конуса диаметром  $680 \pm 30$  мм, что было достигнуто за счет использования различных дозировок суперпластификатора на основе поликарбоксилатного эфира. Все смеси с заменой цемента золой до 25 % отвечают требованиям к реологическим характеристикам и сопротивлению расслоению. Объемная плотность для образцов

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с 10 %, 15 %, 20 % и 25 % золы рисовой шелухи была снижена на 3,19 %, 5,18 %, 5,58 и 6,37 % соответственно по сравнению с образцами без золы. Увеличение содержания золы рисовой шелухи привело к снижению ранних механических свойств, тогда как конечная прочность самоуплотняющегося бетона, содержащего золу, была сопоставима с обычными образцами. Это было достигнуто за счет пуццолановой активности золы. Включение золы рисовой шелухи уменьшило количество портландита в системе за счет получения дополнительного геля C-S-H, что привело к уплотнению матрицы и блокированию сетей с открытой пористостью.

## 1. Introduction

Self-compacting concrete (SCC) is characterized by the fact that under its own weight a dispersed-reinforced concrete mix completely fills the formwork without the need for an external seal. Delamination resistance and adaptability allow the SCC remain homogeneous and to maintain stable characteristics. SCC is mainly characterized by excellent workability of the concrete mix. Reducing labor and construction time, improving the quality of the finished surface make self-compacting concrete better than ordinary concrete. Nevertheless, the production of self-compacting concrete with high workability and the required strength requires more cement and the addition of expensive chemical impurities to reduce the amount of water-binding ratio, which leads to an increase in the cost of self-compacting concrete. Furthermore, the SCC production occurs more carbon dioxide emissions than ordinary concrete production. And also, a higher consumption of Portland cement in SCC mixture leads to an increase in hydration energy and high autogenous shrinkage [1].

Addition of pozzolanic materials leads to energy savings and materials costs, economic efficiency, durability, increased productivity of workplaces [2]. In addition, the best performance characteristics of concrete are achieved by reducing of energy of hydration and autogenous shrinkage [3]. In addition, in terms of environmental considerations, reducing cement consumption results in energy and resource savings, as well as a significant reduction in greenhouse gas emissions [4].

Nearly one hundred million tons of ordinary rice husks are produced every year, which is an excellent raw material – cheap, renewable, with a chemical composition that is constant for a given region and plant variety, suitable for obtaining about 15 million tons of pure amorphous silica. In particular, according to prof. L. Zemnukhova et al [5], from 1 ton of rice husks it is possible to obtain from 120 to 200 kg of silica with a SiO<sub>2</sub> content of 90 to 99.999 %. In addition, from the standpoint of environmental protection, waste utilization is one of priority tasks [6]. A successful implementation of this task can be rice husk ash (RHA) using as an alternative material in the production of concrete.

The use of crop waste for the production of building materials was investigated in a number of papers by the world leading experts. In these works, the possibility of using rice husk ash in cementitious materials based on Portland cement has been proved [7–10].

Therefore, the abundance of RHA, coupled with a high content of SiO<sub>2</sub> in them, opens the way for its use as a partial replacement of Portland cement and the development of concrete with high mechanical characteristics [10]. In particular, the additive RHA improves the strength of concrete, due to the increase in the amount of CSH gel during the hydration process over time. However, to our knowledge, the use of RHA as pozzolanic materials as partial replacements of Portland cement has not been widely investigated in the self-compacting concrete. Considering the agglomeration of particles and their inherent high requirements for water consumption for the addition of RHA, it is expected that its inclusion in Portland cement based self-compacting concrete will reduce the rigidity of the mixture and, therefore, will adversely affect the properties of the matrix of setting concrete.

Thus, the purpose of the research is to study the effect of partial replacement of RHA in self-compacting Portland cement based concrete by 10, 15, 20, 25 and 30 wt. % of a binder in a concrete mixture and as well as in a concrete when applying a compression load.

To achieve this purpose, accomplished tasks include the following:

- research of the physical properties and chemical composition, and the structure of the raw materials;
- pretreatment of rice husk ash for increase its activity;
- determination of fresh properties the self-compacting concrete;
- research of concrete strength characteristics

## 2. Materials and Methods

### 2.1. Materials

The Spassky Portland cement type CEM I 42.5 N was applied for concrete samples. Parts of the rice plants of the Khankaisky district (Primorsky Krai) were burned to get ash for the RHA production. This RHA contained large particles and impurities. In addition, since the RHA was stored in an open area after production with an unknown moisture condition, it was dried at 105 °C for 24 hours before use in the composite binder. Then the dried RHA was sieved through a 300 µm sieve to remove coarse particles and impurities. Then the ash was ground in a vario-planetary mill to increase the specific surface area (550 m<sup>2</sup>/kg) and, accordingly, the reactivity. Physical characteristics and chemical composition of the starting materials are presented in Table 1 and Table 2.

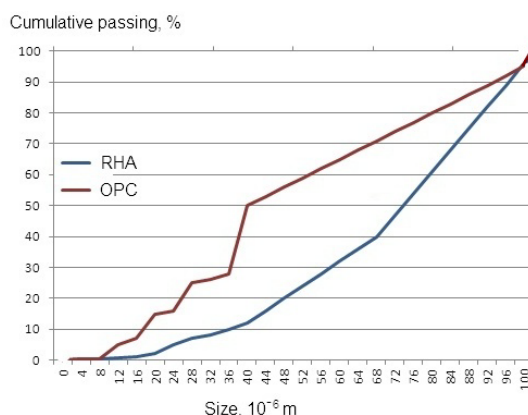
**Table 1. Physical characteristics of raw materials**

| Characteristic                             | Coarse aggregate | Fine aggregate | Portland cement | RHA  |
|--------------------------------------------|------------------|----------------|-----------------|------|
| Maximum size, mm                           | 12.4             | 4.65           |                 |      |
| Water absorption, %                        | 0.42             | 1.14           |                 |      |
| Size module                                | 6.2              | 2.93           |                 |      |
| Passage through a 45-µm sieve (No. 325), % |                  |                | 92              | 97   |
| Average particle size, µm                  |                  |                | 14.5            | 11   |
| Specific gravity, kg/m <sup>3</sup>        | 2.61             | 2.57           | 3.14            | 1.82 |

**Table 2. The chemical composition of Portland cement and RHA**

| Content of elements in terms of oxides, % | RHA  | Portland cement |
|-------------------------------------------|------|-----------------|
| SiO <sub>2</sub>                          | 84.3 | 20.2-20.9       |
| Al <sub>2</sub> O <sub>3</sub>            | 1.1  | 6.0-6.7         |
| Fe <sub>2</sub> O <sub>3</sub>            | 0.3  | 3.5-4.0         |
| CaO                                       | 0.5  | 66.2-67         |
| MgO                                       | 0.9  | 1.4-2.0         |
| K <sub>2</sub> O                          | 3.7  | -               |
| Na <sub>2</sub> O                         | 1.0  | -               |
| SO <sub>3</sub>                           | 0.1  | -               |
| LOI                                       | 8.1  | 0.18            |

Both Portland cement and RHA are in the form of agglomerated particles, but RHA has a more porous structure than Portland cement. As shown in Figure 1, the particle size distribution of the materials after preparation of the rice husk ash becomes smaller than that of the Portland cement.



**Figure 1. Particle size distribution for ground ash and Ordinary Portland cement (OPC)**

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Various types of pretreatment of ash were used to increase the reactivity, as shown in Table 3.

**Table 3. Pretreatment of ash used in the study**

| Heat treatment methods                | Processing sequence  |            |                          |                       |
|---------------------------------------|----------------------|------------|--------------------------|-----------------------|
|                                       | Pre-drying at 105 °C | Moistening | Heat treatment at 800 °C | Cooling in cold water |
| 1. PD – Pre-drying (reference sample) | X                    |            | X                        |                       |
| 2. US – Unprepared sample             |                      |            | X                        |                       |
| 3. MD – Moistening / drying           |                      | X          | X                        |                       |
| 4. Moistening / drying / cooling      |                      | X          | X                        | X                     |

The reference sample of the ash is preliminarily held at 105 °C for 24 hours, then heated to 800 °C at a heating rate of 10 °C/min and cooled at room temperature.

The second set of samples, marked as unprepared, was heated directly to 800 °C using a heating rate of 10 °C/min and cooled at room temperature without preliminary and subsequent treatment.

The following samples, called MD, were preliminarily held under water for 24 hours to fill all the pores with water. The ash was then removed from the water and placed in a preheated oven at a temperature of 800 °C for 60 minutes and gradually cooled.

The last set of samples designated as MDC was treated similarly to the MD, but after heat treatment at 800 °C for 60 minutes, they were immediately cooled in cold water. It was found that shock cooling leads to the formation of cracks in the ash particles. The powder was then dried at 300 °C for 30 minutes.

Thus, the surface area of the RHA was increased using furnaces. This increase in particle size is caused by the formation of cracks, because the captured porous water can not be removed as quickly as it evaporates. This causes internal stress, which leads to the formation of cracks. When the RHA powder is heated to 800 °C, the sintering of the particles leads to deformation. In addition, as was revealed in [1], during the preheating reactivation of the RHA occurs. Both effects in combination result in a reduced setting time and an increase in compressive strength at the beginning.

As a fine aggregate, the local mountain sand was used with a modulus of fineness 2.93, specific gravity 2.57 kg/m<sup>3</sup> and water absorption 1.14 %. As a coarse aggregate, limestone crushed stone was used with a maximum size of 12.4 mm, specific gravity 2.61 kg/m<sup>3</sup> and water absorption 0.42 %. The test procedures and data obtained were in accordance with ASTM C33 [11], GOST 8736-2014 [12] and GOST 8269.0-97 [13] for compliance.

## 2.2. The proportions of the mixtures

The ground RHA was used as a partial replacement for Type I Portland cement in the proportions of 10 %, 15 %, 20 %, 25 % and 30 % by weight of the binder content. The composition of the mixtures of self-compacting concrete included 475 kg/m<sup>3</sup> of binder and a water-binder ratio of 0.34 for all samples, as shown in Table 4. Superplasticizer "HIDETAL-GP-9" alpha "A" (SKT-Standard, Russia) was used in SCC to reduce the water-cement ratio. "HYDETAL-GP-9" alpha "A" promotes maximum water reduction, which meets the requirements of superplasticizers in accordance with ASTM C494 [14] and GOST 24211-2008 [15]. Due to the agglomerated shape of the RHA particles, a higher superplasticizer was used to obtain a similar workability of the samples.

Table 4. Design of sample compositions

| Mixture number | Water-binder ratio | OPC, kg/m³ | RHA, kg/m³ | Fine aggregate, kg/m³ | Coarse aggregate, kg/m³ | Water, kg/m³ | Superplasticizer, % of binder |  |
|----------------|--------------------|------------|------------|-----------------------|-------------------------|--------------|-------------------------------|--|
| 0-PD*          | 0.34               | 475        | 0          | 930                   | 749                     | 151.5        | 1.1                           |  |
| 0-US**         |                    |            |            |                       |                         |              |                               |  |
| 0-MD***        |                    |            |            |                       |                         |              |                               |  |
| 0-DC****       |                    |            |            |                       |                         |              |                               |  |
| 10-PD          |                    | 427.5      | 47.5       |                       |                         |              | 1.2                           |  |
| 10-US          |                    |            |            |                       |                         |              |                               |  |
| 10-MD          |                    |            |            |                       |                         |              |                               |  |
| 10-MDC         |                    |            |            |                       |                         |              |                               |  |
| 15-PD          |                    | 403.75     | 71.25      |                       |                         |              | 1.3                           |  |
| 15-US          |                    |            | 71.25      |                       |                         |              |                               |  |
| 15-MD          |                    |            | 71.25      |                       |                         |              |                               |  |
| 15-MDC         |                    |            | 71.25      |                       |                         |              |                               |  |
| 20-PD          |                    | 380        | 95         |                       |                         |              | 1.4                           |  |
| 20-US          |                    |            | 95         |                       |                         |              |                               |  |
| 20-MD          |                    |            | 95         |                       |                         |              |                               |  |
| 20-MDC         |                    |            | 95         |                       |                         |              |                               |  |
| 25-PD          |                    | 356.25     | 118.75     |                       |                         |              | 1.5                           |  |
| 25-US          |                    |            | 118.75     |                       |                         |              |                               |  |
| 25-MD          |                    |            | 118.75     |                       |                         |              |                               |  |
| 25-MDC         |                    |            | 118,75     |                       |                         |              |                               |  |
| 30-PD          |                    | 332.5      | 142.5      |                       |                         |              | 1.6                           |  |
| 30-US          |                    |            | 142.5      |                       |                         |              |                               |  |
| 30-MD          |                    |            | 142.5      |                       |                         |              |                               |  |
| 30-MDC         |                    |            | 142.5      |                       |                         |              |                               |  |

\* – PD – Pre-drying

\*\* – US – Unprepared sample

\*\*\* – MD – Moistening / drying

\*\*\*\* – MDC – Moistening / drying / cooling

### 2.3. Methods for preparing and testing samples

#### 2.3.1. Fresh properties

Coarse and fine aggregates were mixed first. Then 10 % water was added. Then, cement and RHA were added to the mixture, followed by the addition of 50 % water. The remaining proportion of water was added to the mixture with the superplasticizer, so that homogeneous mixtures could be obtained. Usually the process of mixing self-compacting concrete mixes requires more time than ordinary concrete mixes. It should be noted that the inclusion of RHA leads to a further level of complexity in meeting the test requirements for SCC, so the maximum RHA content selected in this study was limited to 30 % due to the higher water demand for ash. The properties of the concrete mix have been tested in accordance with [16–18] for workability and resistance to delamination after mixing. Initially, the mixtures were examined for the slump flow and slump flow for 50 seconds.

#### 2.3.2. Research of concrete strength characteristics

Fresh properties of the concrete mixture were determined immediately after mixing. Then, cubes with an edge of 100 mm and prisms 100 x 100 x 500 mm in two layers of 5 cm each were molded from the concrete mix. The concrete mixture filled the molds under its own weight, without additional compaction. Then, the upper surface of the samples was smoothed and aligned by hand. After pouring,

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all samples were held for 24 hours under ambient conditions. The samples were then taken out of the mold and kept in water at  $25 \pm 3$  °C until the day of testing.

The bulk density of the final samples was measured using the Archimedes method. To observe the effect of RHA content on the microstructure of self-compacting concrete, electron microscopy images (FESEM MIRA 3 TESCAN) were obtained. The compressive strength of the samples was obtained on 100 mm cubes at the age of 7 and 28 days. All mechanical tests were completed using a Servo-hydraulic Fatigue and Endurance Tester Shimadzu Servopulser U-type with capacity of 200 kN according to BS EN 12390-3:2002.

The error in the results of the experiments is no more than 5 %, so the results can be considered adequate.

### 3. Results and Discussions

#### 3.1. Fresh properties of concrete mixes

The test results for fresh SCC properties were determined by performing filling tests (slump flow,  $T_{50\text{cm}}$  slump flow and V-funnel spread time), passing tests (U-box and L-box flow) and the segregation resistance test of various SCC mixtures. The results of studies of the properties of the concrete mixes are given in Figure 2.

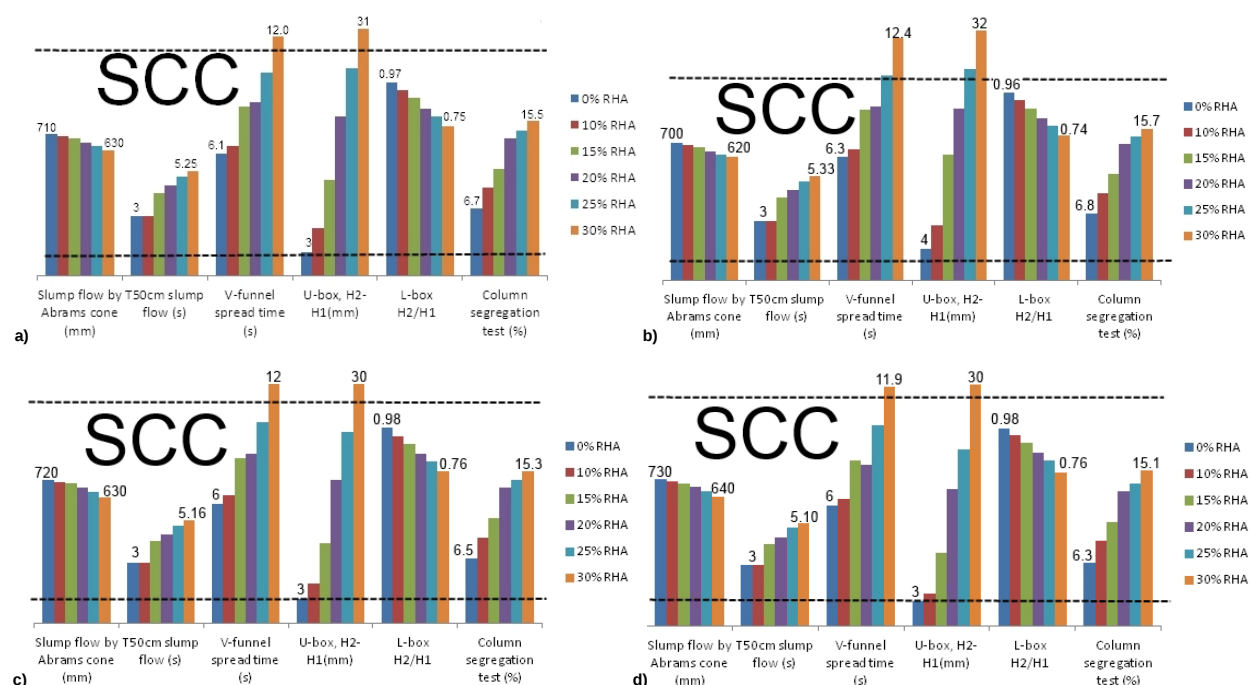


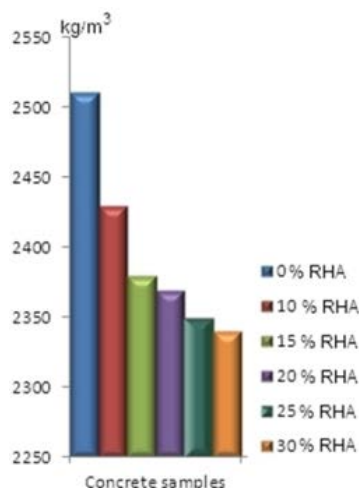
Figure 2. Fresh properties of concrete mixes: a) Pre-drying; b) Unprepared sample; c) Moistening / drying; d) Moistening / drying / cooling

For the study of unconfined flowability, all concrete mixtures were designed in such a way as to have a slump flow of an average diameter of  $680 \pm 30$  mm, which was achieved by using different superplasticizer values. However, for a number of samples this was not achieved. In particular, this is observed for an unprepared sample with 25 % ash and for all samples with the replacement of 30 % ash. Thus, an increase in the ash content of the mixtures led to a reduction in workability due to a higher specific surface area of the ash particles, which led to a greater consumption of water to facilitate movement and rolling of particles over each other. Nevertheless, in the RHA there are still some unmilled or insufficiently ground particles. This can be found in previous studies [1, 21–25], from which it can be concluded that the particles of the grounded and unmilled RHA are extremely porous and agglomerated, while the Portland cement particles were denser even than the RHA with a reduced form. As the water content increases, the porosity increases too, this can lead to unfavorable effects on the properties of the concrete mix. The results of V-funnel and  $T_{50\text{cm}}$  flow times for RHA mixtures were close to the control mixture and ranged between 6.0–12.4 s and 3–5.33 s, respectively, as shown in Figure 2. In addition, the data obtained showed that all mixtures with ash replacement by up to 25 % of ash meet the requirements of segregation resistance according to [18]. A higher content of RHA showed an increase in the viscosity

of the concrete, which led to a decrease in slump flow, U-box and L-box, while it increased the flow time of the  $T_{50}$  cm, V-funnel and the segregation index. Summarizing, it can be concluded that up to 25 % of cement can be replaced by rice husk ash without adversely affecting the properties of the concrete mix. The results of the study of the properties of concrete mixtures are in good agreement with previous studies published by [1, 19, 20, 27].

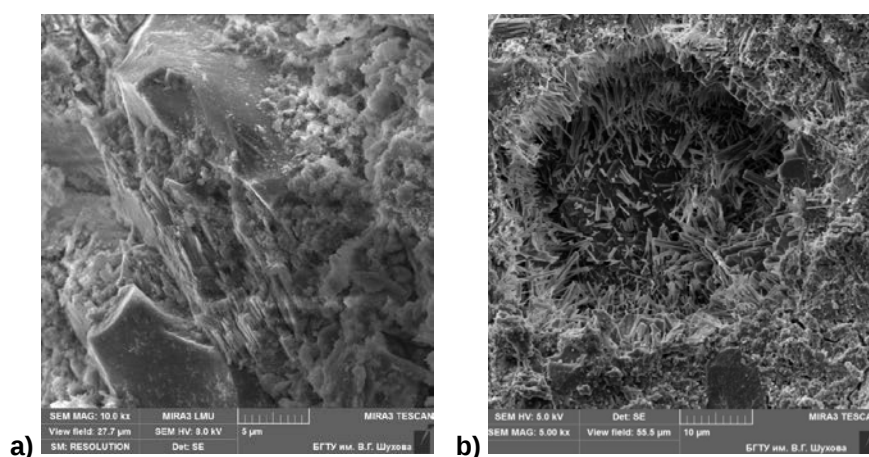
### 3.2. Mechanical properties of concrete

The bulk density of the samples was reduced by increasing the percentage of RHA in SCC, as shown in Figure 3.



**Figure 3. Bulk density of the concrete samples**

The greatest decrease in bulk density was 6.77 % due to the inclusion of 30 % RHA in SCC. The bulk density for samples with 10 %, 15 %, 20 % and 25 % RHA was reduced by 3.19 %, 5.18 %, 5.58 and 6.37 % respectively, compared to samples without RHA. The decrease in the density of concrete samples can be explained by several reasons. The main reason may be that the RHA has a lower specific gravity than cement. In Figure 4 microphotographs of cement stone without additives and cement stone with replacing 25 % of cement with rice husk ash are presented. It can be concluded from Figure 4 that the sample without RHA content is denser than the sample with 25 % RHA.

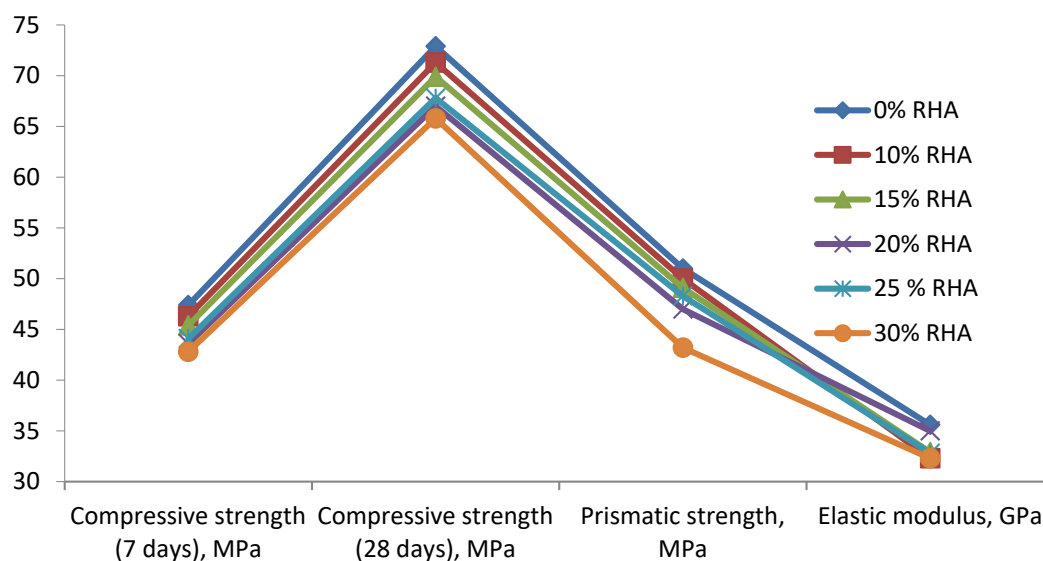


**Figure 4. Microphotographs of cement stone without additives (a) and cement stone with replacing 25 % of cement by rice husk ash (b)**

The initial study showed that preheat treatment of RHA not only affects the particle size distribution, but also causes dehydroxylation [1]. It is expected that both effects will be useful for the mechanical properties of concrete.

The tests in this paper were focused on the activity of the optimized composite binder, and, accordingly, the strength characteristics of self-compacting concrete. It was found that after 28 days, the maximum compressive strength and prismatic strength measure for the MDC samples, then the MD, then

the unprepared samples, and the lowest strength for the reference samples when heated to 800 °C. The mechanical properties of self-compacting concrete, in which the introduced RHA are preliminarily prepared, are shown in Figure 5.



**Figure 5. Strength characteristics of self-compacting concrete**

According to the spread of the elastic modulus of the samples, no dependence is observed either on the amount of the introduced RHA, or on its method of preliminary preparation. It is obvious that elastic modulus of self-compacting concrete primarily depends on the amount of coarse aggregate what was revealed earlier [21, 22].

If we monitor the dependence of the 28-day strength on the amount of ash introduced, the following is noted. Samples containing RHA have slightly lower compressive strengths than conventional SCCs without RHA.

As a rule, the process of hydration of a composite binder can be divided into two main stages. The first stage is mainly related to the reaction of cement and water; the second stage is associated with the pozzolanic activity of ash with portlandite neoplasms from cement hydration. As a result of the reaction of alite and water, a C-S-H gel and portlandite are formed. Since hardening of the C-S-H gel is the main factor in enhancing the strength of concrete, a decrease in the content of Portland cement has led to a decrease in the strength of concrete. Meanwhile, the content of silicon dioxide in the RHA is able to react with portlandite and generates a secondary C-S-H gel. Therefore, the hardening of concrete occurs at a later stage in comparison with cement concrete. On the other hand, the pozzolanic reaction mainly contributes to the increase in compressive strength of concrete at a later age by improving the interfacial bond between the cement paste and the aggregate. In addition, the smallest particles of ash improve the strength of concrete, filling the gaps between the cement particles.

### 3.3. Place of rice husk ash among other mineral additives

Several composites with various mineral additives were compared. In particular, fine-grained concretes developed by the authors [21–22], as well as self-compacting concretes with the use of palm oil fuel ash [1] were considered.

The first concrete shows higher mechanical characteristics, in particular a compressive strength of about 80 MPa. This is achieved through the mechanochemical activation of the binder. However, the developed concretes with the use of fly ash do not provide the necessary rheological characteristics of self-compacting concrete. In addition, the fly ash of thermal power plants is potentially radioactive material, therefore, spectroscopic studies of the radioactive background of the raw materials are mandatory. On the contrary, the ash of the rice husks does not show a radioactive background.

The second composite, with the inclusion of palm oil fuel ash, is able to meet the requirements for self-compacting concrete mixtures (slump flow – 660–690 mm, depending on the percentage of the introduced ash,  $T_{50cm}$  spread time 3–4.57 s). The compressive strength at the age of 28 days is 50–55 MPa. However, these additives are not available for Russia, on the contrary, there are many ashes of rice husk in Russia, so it can be used as a mineral additive in concrete.

## 4. Conclusion

Thus, in the course of studying the mechanical properties of self-compacting concrete with partial replacement of Portland cement with rice husk ash, the following was revealed. As a rule, RHA as a secondary material has a great potential for use as a replacement for Portland cement in self-compacting concrete, which preserves the fresh and mechanical characteristics of the concrete mix and ready-mix concrete in an acceptable range. The surface area of the RHA was increased using furnaces. The particle size can even be increased by soaking the RHA in water and after quenching.

The inclusion of RHA led to a decrease in the workability of concrete, but with the help of an additional superplasticizer, these SCC properties for all samples were almost identical. An increase in the content of the RHA resulted in a decrease in the early mechanical properties, whereas the final strength of the SCC containing the RHA was comparable to that of the conventional samples. This was achieved due to the pozzolanic activity of the RHA. The inclusion of the RHA reduced the amount of portlandite in the system by obtaining an additional gel C-S-H, which led to matrix tightening and blocking of networks with open porosity. These results confirm the well-known patterns (for example, [3, 4, 27], which can be extended to pozzolanic additives of other species.

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