1. Introduction

Sustainable development of the cement and concrete industry requires the utilization of industrial and agricultural waste components. At present, for a variety of reasons, the concrete construction industry is not sustainable. Firstly, it consumes huge quantities of virgin materials which can remain for next generations. Secondly, the principal binder in concrete is Portland cement, the production of which is a major contributor to greenhouse gas emissions that are implicated in global warming and climate change. Thirdly, many concrete structures suffer from lack of durability which may waste the natural resources. So, finding a solution to substitute a practical recycled product for part of the cement seems to be desirable for sustainable development. [1-7]

Recycling of waste components contribute to energy savings in cement production, to conservation of natural resources, and to protection of the environment. Furthermore, the use of certain components with potentially pozzolanic reactivity can significantly improve the properties of concrete. [8-14] One of the most suitable sources of pozzolan material among agricultural waste components is rice husk, as it is available in large quantities and contains a relatively large amount of silica. When rice husk is burnt, about 20% by weight of the husk is recovered as ash in which more than 75% by weight is silica. Unlike natural pozzolan, the ash is an annually renewable source of silica. It is worth to mention that the use of RHA in concrete may lead to the improved workability, the reduced heat evolution, the reduced permeability, and the increased strength at longer ages. [15-21]

In Iran, rice production has increased during these years, becoming the most important crop. Rice husks are residue produced in significant quantities. While in some regions, they are utilized as a fuel in the rice paddy milling process, in our county they are treated as waste, causing pollution of environment and disposal problems. Due to increasing environmental...
concern, and the need to preserve energy and resources, efforts have been made to burn the husks under controlled conditions and to utilize the resultant ash as a building material. In addition, rice husks are able to be an ideal fuel for electricity generation [11-14].

The use of Rice Husk Ash (RHA) in concrete was patented in the year 1924 [14]. Up to 1978, all the researches were concentrated to utilize ash derived from uncontrolled combustion. Mehta published several papers dealing with rice husk ash utilization during this period. He established that burning rice husk under controlled temperature-time conditions produces ash containing silica in amorphous form [22-26].

Depending on produce method, the utilization of rice husk ash as a pozzolanic material in cement and concrete provides several advantages, such as improved strength and durability properties. Rodrý´guez de Sensale [16] reported that mortars and concrete containing RHA have compressive strength values inferior or superior to that of OPC concrete. In addition, in most of the cases [18,19,26,27], mortars and concrete containing RHA improves durability of concrete at various ages.

Generally, there are two types of RHA in concrete. The type of RHA which is suitable for pozzolanic activity is amorphous rather than crystalline. Therefore, substantial researches have been carried out to produce amorphous silica. The results have shown that RHA quality depends on temperature and burning time.

Apparently, for an incinerator temperature up to 700 °C, the silica is in amorphous form and silica crystals grew with time of incineration. The combustion environment also affects specific surface area, so that time, temperature and environment also must be considered in the processing of rice husks to produce ash of maximum reactivity [5,7].

2. Production & optimization of rice husk ash quality

2.1. Rice husk ash (RHA) production

The quality of RHA as an additive for cement and concrete depends on its reactivity. The reactivity of RHA contributes to the strength of RHA-based materials by pozzolanic reactions between the silica and the calcium hydroxide liberated during the cement hydration process. These reactions produce additional amounts of calcium silicate hydrate that makes the microstructure of the RHA concrete denser as compared to that of concrete without RHA. The reactivity of RHA depends on amorphous silica content available and on the porous structure of...
the ash. On the other hand, the ashes produced by burning rice husks under uncontrolled conditions may contain various amounts of un-burnt carbon. In order to supply typical RHA, a special furnace was designed and constructed in the Amirkabir University of Technology. A schematic view of the furnace is shown in Figure 1.

This furnace was manufactured in the pilot size, capable of controlling the conditions of combustion. The furnace was designed and manufactured in order to control the temperature and the rate of burning. Therefore, the furnace can be used to produce rice husk ashes with various un-burnt carbon contents. At the beginning of combustion, the firing natural gas is burnt directly in the furnace. During this time, the husks are dried and combustion is started. Then, the gas supply as a fuel is discontinued and combustion procedure is continued automatically.

Moreover, there are two ways for supplying required air for combustion. Primary, the hot air is blown into the main storage of ash through a porous plate. Simultaneously, the air is injected from another blower through pipes of air transfer. The air blowers can directly control the combustion procedure.

Temperatures can be measured by thermocouples at the fire zone, in air inlet and outlet zones. The measured temperature in the fire zone shows directly the temperature of the ash. This temperature is checked by an electrical controller. The temperature controller is installed on electric control box and can switch on or off the blowers. No control is required during burning process.

2.2. Rice husk ash (RHA) optimization

From literature review it can be concluded that burning rice husks at temperature below 700°C produces rice husk ashes with high pozzolanic activity [3,4,11]. At first, ashes used for investigating properties of RHA concrete were produced by burning rice husks in the furnace. The temperature in the furnace was maintained below 750°C. This temperature was recorded in the fire zone where the rice hush was burnt. The measured temperatures were 550, 600, 650, 700 and 750°C. Furthermore, time of burning was another variable parameter that was investigated at 30, 60 and 90 minutes. In addition, an ash sample was produced at temperature of 1100°C after 5 minutes burning. The temperatures can be varied by regulating the blowing air. Therefore ashes with various amount of unburnt carbon were obtained.

To identify and quantify the major and minor oxide elements present in the samples of obtained rice husk ash, X-Ray Fluorescence (XRF) analysis was carried out. The results are given in Table 1. The chemical compositions of the RHA indicate that the material is mainly composed of SiO₂. The table implies that the ashes produced in the specially designed furnace at low temperature or during 30 minutes of burning are not appropriate due to their high loss on ignition. So, they were not suitable for RHA concretes. Based on results of Table 1, Figure 2 can be drawn out.

<table>
<thead>
<tr>
<th>Temperature and time duration</th>
<th>550°C</th>
<th>600°C</th>
<th>650°C</th>
<th>700°C</th>
<th>750°C</th>
<th>1100°C</th>
<th>Cement type I</th>
</tr>
</thead>
<tbody>
<tr>
<td>30min</td>
<td>76.21</td>
<td>80.76</td>
<td>76.21</td>
<td>89.61</td>
<td>92.19</td>
<td>95.31</td>
<td>21.50</td>
</tr>
<tr>
<td>60min</td>
<td>75.22</td>
<td>80.55</td>
<td>85.60</td>
<td>89.61</td>
<td>90.21</td>
<td>93.11</td>
<td>21.50</td>
</tr>
<tr>
<td>90min</td>
<td>80.76</td>
<td>85.55</td>
<td>89.60</td>
<td>92.19</td>
<td>90.21</td>
<td>93.11</td>
<td>21.50</td>
</tr>
</tbody>
</table>

Table 1 Results of XRF on rice husk ash samples and cement
Figure 2-(a) can be used to obtain silicon dioxide content from temperature and time duration of burning ashes and Figure 2-(b) shows variation of LOI value vs. temperature and time. Figure 3 shows a photo of rice husk, high carbon RHA, optimum RHA and RHA with crystalline silica. Then in order to determine the crystalline compounds present in the various rice husk ash specimens, X-ray Diffraction (XRD) test was carried out. Fig. 4 shows XRD patterns of the produced ashes. The ash patterns were denoted as RHA-Temp.-Min. respectively. The figure shows that silica in the rice husk initially exists in the amorphous form, but will not remain porous and amorphous, when combusted for a prolonged period at a temperature above 650°C, or during less than a few minutes at 1100°C, under oxidizing conditions. It means that the reactivity of rice husk ash is generally decreased by the increase of burning temperature and the heating duration. Burning rice husks at temperature below 650°C produces amorphous crystals of rice husk ashes. Combination of 650°C temperature and 60 minutes burning time seems to present the optimized solution resulting in non-crystallize RHA.

3. Experimental program

3.1. Materials used

The following materials were used in the preparation of the concrete specimens. Local natural sand according to ASTM Standard with maximum aggregate size of 4.75 mm; Crushed granite according to ASTM Standard with...
maximum aggregate size of 19 mm; Tehran potable water, Type I Portland cement and homogeneous rice husk ash produced by the special designed furnace at 650°C and 60 minutes burning time. Table 2 shows the physical and chemical characteristics of RHA (RHA-650-60) and cement.

3.2. Specimens preparation

A total of 4 concrete mixtures were made; one corresponding to a control concrete (CTL) and three others with 7%, 10% and 15% RHA replaced with cement by weight. Table 3 lists the mix proportions of concrete. Slumps were kept constant at 70 ± 10 mm. Superplasticizer was used at very low percentages according to the results obtained for the slumps. Concrete test specimens were compacted by external vibration and kept protected after casting to avoid water evaporation. After 24 hr. they were demolded and cured in lime-saturated water at 23 ± 2°C to prevent possible leaching of Ca(OH)2 from these specimens.

3.3. Test method

Concrete cubes of 100×100×100 mm dimension were cast for compressive strength and water penetration tests. The results obtained are reported as an average of three tests. Splitting tensile strength test was conducted on RHA blended concrete cylinders as per IS 5816-1999 after 7, 28 and 90 days of moisture curing. The results obtained are reported as an average of two tests. While two 150×300 mm cylindrical concrete specimens were prepared for the static modulus of elasticity tests. Samples of rapid chloride permeability tests (RCPT), according to ASTM C 1202, were prepared by cutting and discarding 25mm slices from the top and bottom of 100×200 mm cylinders, and the remaining section cut into three 50mm thick slices. The water permeability test was conducted using a high-pressure permeability cell. The specimens used were cubes of 150×150×150 mm dimension. In addition, 50×50×50 mm mortar samples were prepared for the pozzolanic activity test. All specimens were moist cured until the time of testing.

3.4. Test results

The results of pozzolanic activity test are shown in Table 4. Results demonstrate high pozzolanic activity index of RHA over that of the

### Table 2: Physical and chemical characteristics of cement and RHA

<table>
<thead>
<tr>
<th></th>
<th>Physical Tests</th>
<th>Chemical Analyses, (%)</th>
<th>Bogue Composition, (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Specific Gravity</td>
<td>Blaine, (cm²/gram)</td>
<td>SiO₂</td>
</tr>
<tr>
<td>RHA</td>
<td>2.15</td>
<td>3600</td>
<td>89.61</td>
</tr>
<tr>
<td>cement</td>
<td>3.21</td>
<td>3200</td>
<td>21.50</td>
</tr>
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</table>

![Fig. 5](https://via.placeholder.com/150)

**Fig. 5** Compressive strength (MPa) at various ages for control (CTL) & RHA mixtures

![Fig. 6](https://via.placeholder.com/150)

**Fig. 6** Tensile strength (MPa) at various ages for control (CTL) & RHA mixtures
control in accordance with ASTM C-311/ASTM C-618 test method. On the other hand, produced rice husk ash is a high reactive pozzolanic material, and entirely satisfies other requirements.

Results of the compressive strengths of concretes are given in Figure 5. In general, the RHA concrete had higher compressive strengths at various ages and up to 90 days when compared with the control concrete. The results show that it was possible to obtain a compressive strength of as high as 46.9 MPa after 28 days. In addition, strengths up to 63.2 MPa were obtained at 90 days.

Figure 6 shows that concrete containing RHA has a greater splitting tensile strength than that the control concrete at all ages. It is clear that, as the amount of RHA increases, the tensile strength increases up to 20%. For instance, at 90 days the 15% RHA concrete had a compressive strength of 5.62 MPa compared with 4.58 MPa for the control concrete.

Figure 7 shows that the static modulus of elasticity in compression of concrete mixed with different proportions of RHA at 28 and 90 days. After 90 days, mixture containing 15% of RHA showed 7% increase in static modulus of elasticity in compression as compared to the control concrete. On the other hand, concrete containing RHA depicts a higher static modulus of elasticity when compared to the control concrete.

Results of the rapid determination of chloride permeability of concrete test (Figure 8) show that using RHA drastically enhances resistance to chloride penetration compared to control concrete on average, around 4–5 times higher for the 15% RHA. At 7 days, the control concrete showed the highest value of 6189 coulombs while the charge passed through the 15% RHA concrete was 1749 coulombs.

With a continuous moist-curing of up to 91 days, the charge passed through all concretes; was reduced. The charge for the 15% RHA concrete was reduced to 576 coulombs, which was well below that of the control concrete (2563 coulombs). According to ASTM C 1202, when the charge passed through concrete is below 1000 coulombs, it is categorized as a very high resistance concrete to chloride ion penetration.

The chloride permeability of the concrete specimens incorporating 15% RHA was “very

<table>
<thead>
<tr>
<th>Table 3</th>
<th>Mix proportions of concrete</th>
</tr>
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<tr>
<td></td>
<td>RHA (kg/m³)</td>
</tr>
<tr>
<td>CTL</td>
<td>0</td>
</tr>
<tr>
<td>7%RHA</td>
<td>29.4</td>
</tr>
<tr>
<td>10%RHA</td>
<td>42</td>
</tr>
<tr>
<td>15%RHA</td>
<td>63</td>
</tr>
</tbody>
</table>

Fig. 7 Modulus of elasticity (GPa) at various ages for control (CTL) & RHA mixtures

Fig. 8 Resistance to chloride ion penetration (coulomb) at various ages for control (CTL) & RHA mixtures
In addition to RCPT, investigations of water permeability were carried out. In this test, water was forced into the concrete samples from one side for three days and under constant pressure of 0.5 MPa. Then, the samples were split in a plane parallel to the direction of water penetration, and the greatest depth of water penetration into the concrete sample was measured. The depth of water penetration of concrete incorporating RHA specimens is shown in Figure 9. As expected, depth of water penetration of concrete specimens decreased significantly with an increasing in RHA content and curing period.

5. Conclusions

Based on the results of the present experiments, the following conclusions can be drawn out:

1) The duration and temperature of furnace are important parameters, influencing the reactivity of RHA pozzolans. Silica in the rice husk initially exists in the amorphous form, but may become crystalline when rice husk is burnt at high temperature. In addition, silica in rice husk ash will not remain porous and amorphous, when combusted for a prolonged period at a temperature above 650°C, or during less than a few minutes at 1100°C, under oxidizing conditions. The results of XRD analysis show that quartz crystal is present in both types of ashes. So, investigation on the influence of combustion conditions on the amorphous silica suggests that the RHA-650-60 can be considered to be non-crystalline RHA and to save the RHA aggregates fill the spaces between coarse aggregates in concrete. Moreover, small particles of additions generate a large number of nucleation sites for the precipitation of the hydration products. This will accelerate the reactions and form smaller C-H crystals. RHA reduces the number of large pores and increases the probability of transforming the continuous pores into discontinuous ones. Therefore, all these mechanisms make the microstructure of the paste more homogeneous and denser.

4. Discussion

Improvements in mechanical and durability properties of the concretes containing RHA can be explained by the chemical and physical effects of RHA. Chemical effect is mainly due to the pozzolanic reactions between the amorphous silica of RHA and calcium hydroxide (C-H) produced by the cement hydration to form calcium-silicate-hydrates (C-S-H). The physical effect which can also be considered as filler effect is that RHA particles increase the packing of the solid materials by filling the spaces between the cement grains in much the same way as cement fills the spaces between fine aggregates, and fine aggregates fill the spaces between coarse aggregates in concrete. Moreover, small particles of additions generate a large number of nucleation sites for the precipitation of the hydration products. This will accelerate the reactions and form smaller C-H crystals. RHA reduces the number of large pores and increases the probability of transforming the continuous pores into discontinuous ones. Therefore, all these mechanisms make the microstructure of the paste more homogeneous and denser.

Table 4 Comparison in chemical and physical specifications of produced RHA with ASTM standard C618-03

<table>
<thead>
<tr>
<th>Chemical Requirements</th>
<th>ASTM</th>
<th>RHA results</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{SiO}_2 + \text{Al}_2 \text{O}_3 + \text{Fe}_2 \text{O}_3 ), min., %</td>
<td>70</td>
<td>89.9</td>
</tr>
<tr>
<td>( \text{SO}_3 ), max., %</td>
<td>4</td>
<td>0.15</td>
</tr>
<tr>
<td>Moisture Content, max., %</td>
<td>3</td>
<td>0.23</td>
</tr>
<tr>
<td>Loss On Ignition (LOI), max., %</td>
<td>6</td>
<td>5.9</td>
</tr>
</tbody>
</table>

| Physical Requirements | | |
|------------------------|------------------|
| Fineness: Amount retained when wet-sieved on 45 µm sieve, max., % | 34 | 8 |
| Strength Activity Index (20% RHA) at 3-day, min. % control | --- | 102 |
| Strength Activity Index (20% RHA) at 7-day, min. % control | 75 | 106 |
| Strength Activity Index (20% RHA) at 28-day, min. % control | 75 | 110 |
production time.

2) The results of pozzolanic activity demonstrate high pozzolanic activity index of produced rice husk ash concrete over that of the control. In other words’ the produced rice husk ashes containing up to 90 percents amorphous silica entirely satisfy other requirements of ASTM standard C618-03. This shows the high quality of produced rice husk ashes. In addition, The RHA concrete showed higher compressive strength at various ages in comparison with that of the concrete without RHA. In addition, the RHA concrete had higher splitting tensile strength and modulus of elasticity in comparison with that of the concrete without RHA. It is concluded that produced RHA provides a positive effect on the compressive strength of concretes.

3) The performance of concrete with cement replacement by RHA is outstanding considering resistance to water and chloride ion penetrations which are in many cases the most important characteristic concerning durability and corrosion prevention.

References


