



## **Effect of Calcination on the Chemical and Microstructural Properties of Rice Husk Ash**

**Khalil Ur Rehman <sup>a\*</sup>, Afaq Ahmad <sup>a</sup>, Fawad Ahmad <sup>a</sup>, Ezaz Ali Khan <sup>a</sup>  
and Muhammad Armaghan Siffat <sup>a</sup>**

<sup>a</sup> National Institute of Urban Infrastructure Planning (NIUIP), University of Engineering & Technology, Peshawar, Pakistan.

### **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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### **ABSTRACT**

This research study is aimed to evaluate the effects of different calcination temperatures on the properties of rice husk ash such as the chemical and microstructural properties. Rice husk ash is not utilized properly; it is not dumped with proper handling which is also causing environmental issues. Currently researchers are working on supplementary cementitious materials in concrete, in light of which, this research study is aimed to evaluate the effects of burning on Rice Husk Ash (RHA) structure and its pozzolanic reactivity for utilizing it in concrete. The rice husk is burnt at temperatures of 600-800°C for a duration of 8, 16 and 24 hours and for evaluating different chemical and structural properties through tests of X-ray Diffraction (XRD), X-Ray fluorescence (XRF) and Fourier Transform Infrared Spectroscopy (FTIR). It is concluded that burning of rice husk at 600-800°C for duration of 24 hours gives us more reactive and amorphous material and can be used as a cement substitute for sustainable concrete production.

**Keywords:** RHA; calcination; microstructural properties; supplementary cementitious material; controlled burning.

## ABBREVIATIONS AND ACRONYMS

WSA = *Wheat Straw Ash*  
 XRD = *X-Ray Diffraction Test*  
 XRF = *X-Ray Fluorescence Test*  
 RHA = *Rice Husk Ash*  
 FTIR = *Fourier Transform Infrared Spectroscopy*  
 C-H = *Carbon Hydrogen bond*  
 OH = *Hydroxide ion*  
 Si-O-Si = *Silica group*  
 N-O = *Nitric Oxide*  
 Si-O = *Silicon Monoxide*

## 1. INTRODUCTION

Utilization of the supplementary cementitious materials (i.e. Rice Husk Ash, Wheat Straw Ash, Silica Fume, Bagasse Ash etc.) is one of the major needs of this era, because the use of concrete is increasing day by day. A study shows the use of concrete per human being is approximately 1 ton/annum [1]. The production of concrete is backing the emission of Carbondioxide (CO<sub>2</sub>), from the cement industries; which is around 7% of the worldwide emission of CO<sub>2</sub> [2].

Rice husk ash is one of the major crops in the world. In the year 2016, the total production of the rice was around 745.5 million tons globally, similarly it is estimated that the production of rice

husk was 20% of rice paddy (149.1 million tons) and the production of ash from husk was around 18-20% of the rice husk (26.82 million tons). The detail for the production of rice, rice husk and rice husk ash for different countries are listed in Table 1 [3]. Rice is the most significant crop globally, in 2017 it is estimated that the production of rice was around 760 million tons (mt), the production of husk from rice was 150 mt yearly, around 83% of husk directly discarded to the environment, the discard of husk without proper handling can cause pollution of water and contamination of environment [4].

According to a report of the United State Department of Agriculture Foreign Agricultural Service in 2018, Pakistan has around 8% of the world's total rice exports. Pakistan is ranked 11th globally in the production of rice as contribution from Pakistan in exports of rice was around 9.9 million tons [3].

Rice husk is used as a fuel in different plants. It is the burning material for the production of electricity by generating steam in boilers [5]. Rice husk is also utilized in the kilns as fuel for burning of clay bricks [6,7], similarly the husk of rice is also used as fuel for rice processing industry [5,6]. In India the rice husk is utilized as fuel for the production of electricity and thermal energy instead of coal, it is found that CO<sub>2</sub> is reduced up to 14,744 tonnes [8].

**Table 1. Production of rice, husk of rice and ash of husk in year 2016 [3]**

Country/Region	Production of Rice (mt)	Husk of Rice (20% of the rice ) in (mt)	Ash (18-20% of the husk) in (mt)
China	211.20	42.20	7.60
India	158.40	31.70	5.70
Indonesia	71.90	14.30	2.60
Bangladesh	52.90	10.60	1.90
Vietnam	44.50	8.90	1.60
Thailand	30.30	6.10	1.10
Africa	29.10	5.80	1.10
Myanmar	28.00	5.60	1.01
South America	23.70	4.70	0.90
Philippines	18.70	3.70	0.70
Japan	10.60	2.10	0.40
North America (US)	10.40	2.10	0.40
Pakistan	9.90	1.98	0.40
Cambodia	9.40	1.90	0.30
Europe	4.20	0.80	0.20
Other Countries	32.30	6.50	1.20
Area (ha)	1650 Million hectares		
Total (Globally)	745.5 million tons		

Kang et al. [4] found that at optimal temperature of 500-800°C control burning of the Rice Husk can give us the ash by approximately 20% of the total rice husk weight. The obtained ash contains amorphous silica around 85% to 90% by weight [4]. It is concluded by Nair et al. [9] that more effective and reactive ash of rice husk is extracted, containing more amorphous silica content at control burning temperature of 500°C for 12 hours [9]. From research study review of by Thomas [3] it is found that a controlled burning temperature of 500°C yields more effective, reactive and amorphous silica content in the ash. It is evaluated that by Bie et al. [10] at controlled temperature of burning up to 600°C, the extracted ash from rice husk due to the presence of the amorphous and micro silicon dioxide was more effective and reactive. It is established by Liu et al. [11] that at controlled burning temperature of 600°C for the duration of 4 hours, the attained RHA can be more reactive and contain amorphous silica.

From the above literature it is evident that by changing controlled burning temperature, the structure and reactivity of silica content in the ash of rice husk can alter. The current study is aimed for evaluating the different controlled burning temperature effects on RHA chemical and microstructural properties. The objective of

current research work is to study the effects of the controlled burning temperature on Rice Husk Ash (RHA) in regards to the chemical and microstructural properties of ash of rice husk. For carrying out this research work the testing of X-Ray Fluorescence (XRF) testing, X-Ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) is used for the effects of burning temperature.

## 2. MATERIALS AND METHODS

### 2.1 Collection and Burning of Rice Husk

The sample of rice husk is obtained and burned at the specified temperatures in controlled conditions and for attaining sizing through grinding of ash in the ball mill.

The burning or calcination process of the rice husk is carried out in a laboratory. It is done in a burning drum in controlled conditions which is shown in Fig. 1. Rice husk calcined at controlled burning temperature of 600°C-800°C for different duration of burning of 8, 16 and 24 hour. The extracted ash is obtained and cooled down at room temperature after burning and then grinded in ball mill for more amorphous and reactive structure of silica. The pictures before and after grinding of ash are shown in the Fig. 2.



Fig. 1. Burning of Rice Husk



Fig. 2. Rice Husk Ash before and after grinding in ball mill

### 3. RESULTS AND DISCUSSION

#### 3.1 X-Ray Fluorescence (XRF)

The Rice Husk Ash samples are tested for XRF in a laboratory. The main purpose of this test is to evaluate the oxides percentages by weight in the ash, for checking the pozzolanic properties of the ash for different burning temperatures. The values of oxides percentages by weight in the

ash for different burning temperatures are listed in Table 2. According to the test performed per ASTM C618, all the ashes of different burning temperatures attained the requirements of the pozzolanic material because the composition of the  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  is greater than 70% for all ashes. For the burning temperature of 600-800°C for the duration of 24 hours in the burning drum the oxides composition of  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  is 95.122%.

**Table 2. Weight of oxides in percentages in RHA for different burning temperatures**

Compound / Item	Weight of Oxides in Percentages in Rice Husk Ash Burning Temperature for Calcination (°C)		
	600-800°C for 8hrs	600-800°C for 16hrs	600-800°C for 24hrs
SiO <sub>2</sub> (Silicon dioxide)	95.062	92.708	95.122
Al <sub>2</sub> O <sub>3</sub> (Aluminium oxide)	0.00	0.00	0.00
Fe <sub>2</sub> O <sub>3</sub> (Ferric Oxide)	0.725	1.761	0.398
SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub> Composition	<b>95.78</b>	<b>94.469</b>	<b>95.52</b>
K <sub>2</sub> O (Potassium oxide)	1.960	2.210	2.228
CaO (Calcium oxide)	1.711	2.274	1.121
SO <sub>3</sub> (Sulfur trioxide)	0.229	0.579	0.447
P <sub>2</sub> O <sub>5</sub> (Phosphorus pentoxide)	0	0	0.504
TiO <sub>2</sub> (Titanium dioxide)	0.033	0.058	0.038

### 3.2 X-Ray Diffraction (XRD)

The X-ray Diffraction test was performed for the evaluation of the crystalline and amorphous structure ashes, the results are shown in Fig. 3.

It is concluded that for the ashes having more amorphous structure than crystalline structure, the peaks are exhibited less as shown in Fig. 3. It is observed from the XRD testing results that peaks are located at the 2-theta of 22 and 27 degrees.

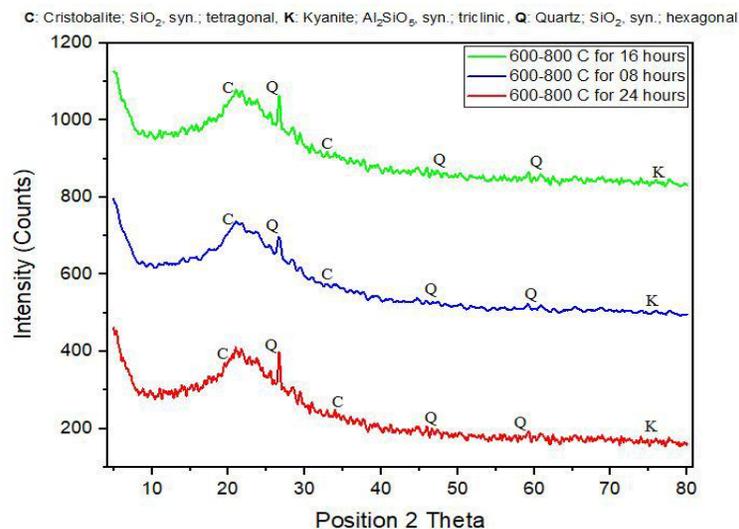
XRD testing was performed on the ash of Rice Husk for the identification of the material amorphous or crystalline structure. The ash samples are tested for the range of 5-80° for 2θ at the rate of 1.5 degree per minute, the

radiations used at the voltage of 30kV and used the monochromatic Cu-K α1 for the scanning.

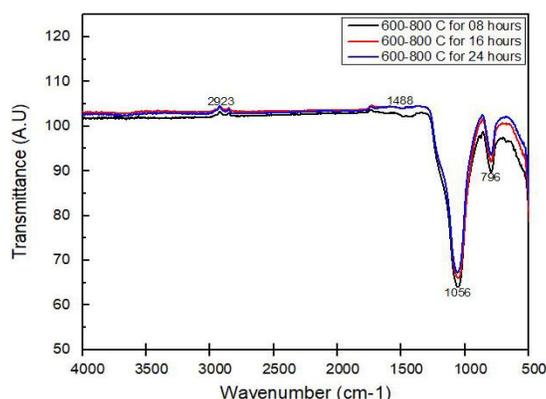
### 3.3 Fourier Transform Infra-Red Spectroscopy (FTIR) Analysis

Testing of FTIR on ashes of rice husk was conducted at Laboratory. The test is conducted on ash of rice husk because XRD testing cannot provides details of short range molecule for the crystalline and amorphous structure of ashes. For attaining short range molecular details, FTIR testing is performed on the ashes of rice husk. The results of FTIR are shown in Fig. 4.

These results can be understood from data in Table 3, in which the details of Wave number for different peaks are listed. Against the minerals in ashes.



**Fig. 3. XRD Testing Results**



**Fig. 4. FTIR testing results**

**Table 3. Band assignment of different peaks shown in Fig. 4 of RHA**

Mineral in the Ashes	Band/Peak Assignment	Reference of Paper	Wavelengths Reading (cm <sup>-1</sup> )
Organic Carbon	(C-H) Stretching vibration	[13]	2923
Montmorillonite	(OH) group adsorbed water dust-stretching	[12]	3454
Feldspar	Si-O-Si band (Amorphous silica)	[20]	563
Nitrate Species	(N-O) Stretching	[16]	1417
Quartz	(Si-O-Si) asymmetric stretching vibration	[17]	1016
Quartz	(Si-O) symmetric	[18,19]	794
Organic matter	C=O carboxylate stretching vibrations group of organic matter	[14,15]	1630

#### 4. CONCLUSIONS

It is concluded that from the testing of rice husk at different calcination temperatures that the burning of rice husk at 600-800°C for a duration of 24 hours gives us more amorphous and pozzolanic reactive material as compared to others. The conclusion is based on the microstructural testing of XRD, XRF and FTIR on the Rice Husk Ash as per the ASTM C618 requirements for pozzolanic material. It is therefore recommended that Rice Husk Ash obtained in this research can be used as a substituted with cement in concrete for minimizing the greenhouse gasses emissions from cement industry as well as for achieving high strength concrete.

#### 5. RECOMMENDATION

The study conducted yields good results of calcination at 600-800°C for 24 hours of controlled burning. It is therefore recommended

to use Rice Husk Ash (RHA) in concrete production. The microstructural and the chemical properties should be evaluated through testing to give an idea of the increase in strength and structure of concrete. The generation of greenhouse gasses and its decrease by using RHA should also be evaluated for environmental friendly concrete production.

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#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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