# Preparation & Characterization of Vitrified Tiles using Rice Husk Ash & Glass Cullet

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***Abstract*— In this experimental work, Effect of replacing feldspar & quartz with glass cullet & Rice husk ash respectively has been studied. Here, RHA acts as a source of silica and glass cullets as a strong flux. Specimens were prepared by using semi dry process prior to Hydraulic pressing. Firing were done at different temperatures like 1050 ˚C, 1100 ˚C and 1150 ˚C, with a soaking period of 2 hours. The samples were characterized and it was found that the use of small amounts of glass cullet powder in addition with feldspar has shown good results in terms of physical & mechanical properties even when fired at certain low temperature in comparison with the standard temperature. It was found that samples with 5% glass replacing feldspar and 6% RHA replacing Quartz has better properties than the standard one. It is to be noted that this can lead to solve the waste disposal problem and has benefits over reducing maturing temperature also.**

***Index Terms*—Waste utilization, Liquid phase sintering, Flux, Amorphous silica, Energy conservation.**

## I. INTRODUCTION

Waste utilization strategies are major concern in many countries, particularly for non-toxic wastes that are able to replace the raw materials for many industries. Rice Husk Ash and Glass cullets, both can be considered in those non-toxic waste materials. Rice Husk is produced as residue of rice paddy during refining process. This is either burnt or dumped as a waste. After burning, residual ash is called Rice husk ash. Globally, approximately 650 million ton of Rice paddies are produced per year [1]. For every 1000 Kg of Rice paddies, about 230 kg (23%) of husk is produced & when this husk is burnt in Rice mills or Boilers as a fuel, about 58 Kg (25%) of RHA is generated, containing about 80%-95% amorphous silica (SiO2), which is considered more reactive than the crystalline one. RHA creates disposal problem due to less commercial profit and interest. It is a great environmental threat causing damage to land where it is dumped. Therefore, commercial use of RHA is the alternative solution to the disposal problem [2, 3].

Glass cullet, produced during demolition of glasses are widely available as waste. However, they are used in glass production itself but when this waste is incorporated into tile batch, it has a good potential to be used as a new fluxing agent. Therefore, glass may replace the expensive fluxes for example feldspar, talc, nepheline etc. and it would have a strong influence on the final cost of production [4-6].

Vitrified tile composition contains both plastic and non-plastic materials. Clay is the plastic material whereas quartz & feldspar are the non-plastic materials. Quartz, a source of silica is commonly used as filler for microstructure development and strength enhancement whereas Feldspar, is used to provide vitrification & reduce the firing temperature by acting as a flux [7].

The aim of this experimental work is to study the possibility of the use of waste glass powder and Rice Husk Ash for manufacturing of vitrified tiles, by partially replacing feldspar and quartz respectively, to reduce the firing temperature and to overcome the disposal problem of RHA [8]. Reduction in the final firing temperature may also lead to the reduction in environmental pollution and extending refractory and furnace life cycle [9]. This would be beneficial in terms of energy saving, waste utilization, economically and environment friendly production & overcoming the waste disposal problems.

## II. Materials and Methods

Rice Husk Ash is a valuable green resource with great potential. It doesn't has any specific formula but the chemical analysis [10] of RHA determined using X-Ray fluorescence spectrometer are tabulated in **Table 1**. It contains more than 85% of $SiO_2$. So, it may be used as a raw- material for the replacement of quartz. Physical properties of RHA are reported in **Table 2**. The production quality of RHA from Rice Husk also depends upon the variety of rice paddy, climate & fertilizers as well as upon the burning conditions.

In this experimental study, Soda-lime-silicate glass powder has been used for the feldspar replacement as it is most widely available in cullets form. Since glass is a strong flux as compared to feldspar because of high amount of alkali oxides present in it. It has been concluded that 5% or less amount of glass cullet is sufficient for complete vitrification even at certain low firing temperature in comparison with the standard one [6]. Chemical analysis results [10] using X-Ray fluorescence spectrometer for glass cullet are tabulated in **Table 3.**

**Table-1:** Chemical analysis of RHA

| S. no. | Particulars | Properties |
|---|---|---|
| **1.** | $SiO_2$ | 85.1 |
| **2.** | $P_2O_5$ | 4.14 |
| **3.** | $K_2O$ | 2.52 |
| **4.** | CaO | 1.65 |
| **5.** | MgO | 0.96 |
| **6.** | $Fe_2O_3$ | 0.918 |
| **7.** | MnO | 0.324 |
| **8.** | $TiO_2$ | 0.22 |

**Table-2:** Physical properties of RHA

| S. no. | Characteristics | Results |
|---|---|---|
| **1.** | Color | Grey |
| **2.** | Mineralogy | Non crystalline |
| **3.** | Particle Size | < 45 microns |
| **4.** | Odor | Odorless |
| **5.** | Specific gravity | 2.3 |
| **6.** | Appearance | Very fine powder |

**Table-3:** Chemical analysis of soda-lime-silicate glass

| S. no. | Particulars | Properties |
|---|---|---|
| **1.** | $SiO_2$ | 73 |
| **2.** | $Na_2O$ | 14 |
| **3.** | CaO | 9 |
| **4.** | MgO | 3.4 |
| **5.** | $Al_2O_3$ | 0.15 |
| **6.** | $Fe_2O_3$ | 0.1 |
| **7.** | $K_2O$ | 0.03 |

For the preparation of samples, granulating semi dry pressing method was used. All the raw materials were grinded using ball mill separately and then dry batch were prepared. Water was added to prepare plastic mass which were firstly granulated prior to pressing using a hydraulic press into rectangular shape using metallic mould. Samples were dried at 110 ˚C in a hot air oven for 6 hours. After drying, they were set to fire at three different temperatures of 1050 ˚C, 1100 ˚C and 1150 ˚C. Heating rate was kept 5 ˚C/min upto maximum temperature and 2 hours of soaking period was allowed. Batch compositions of prepared samples are tabulated in **Table 4.**

**Table-4:** Compositions for Sample preparation

| Composition | Clay (wt %) | Quartz (wt %) | Feldspar (wt %) | Glass (wt %) | RHA (wt %) |
|---|---|---|---|---|---|
| Standard | 55 | 15 | 30 | - | - |
| 5G4R | 55 | 11 | 25 | 05 | 04 |
| 5G6R | 55 | 09 | 25 | 05 | 06 |
| 5G8R | 55 | 07 | 25 | 05 | 08 |
| 5G10R | 55 | 05 | 25 | 05 | 10 |

## III. RESULTS & DISCUSSION

### Water absorption

Water absorption is an important physical property of ceramic tiles. The pore percentage and water absorption depends upon each other significantly. However, closed pores doesn't participate in this water absorption. It was determined according to ASTM C20 [11] using the following formulae-

$$\boldsymbol{Water\ absorption}\ (\%) = \frac{\boldsymbol{Saturation\ weight}\ (\boldsymbol{W}) - \boldsymbol{Dry\ weight}\ (\boldsymbol{D})}{\boldsymbol{Dry\ weight}\ (\boldsymbol{D})}$$

The results are shown in **figure 3.1** where it can be observed that absorption decreases upto a certain limit and again start to increase. These results comes from two factors- First, the presence of glass causes vitrification at low temperature and Second, the presence of RHA in a limited range. Water absorption is decreased due to the formation of glassy phase having low viscosity, which accelerated the densification. This glassy phase fills the pores hence water absorption decreases.

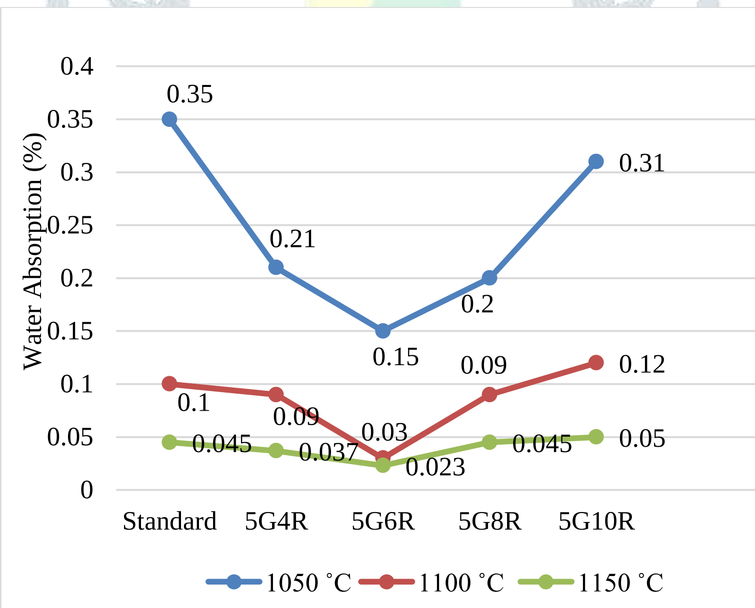


**Fig 1:** Water absorption for samples fired at different temperatures

### Apparent Porosity & Bulk Density

Porosity plays an important role in ceramic tiles, which affects several other properties also. As the porosity increases, the water absorption also increases. Bending strength may decrease due to less compaction. However, such relationship between these characteristics aren't true always. In the present study, as the temperature varied in ascending order, bending strength wasn't increased all time because viscosity of glassy phase decreases at such high temperatures and pass away its range. Apparent porosity and bulk density were measured according to ASTM C20 [11] using the following formulae utilizing Archimedes principles. The results of apparent porosity and bulk density are shown in **Fig 2** & **Fig 3** respectively.

$$\boldsymbol{Apparent\ porosity}\ (\%) = \frac{\boldsymbol{Saturation\ weight\ (W) - Dry\ weight\ (D)}}{\boldsymbol{Saturated\ weight\ (W) - Suspended\ weight\ (S)}}$$

$$\boldsymbol{Bulk\ density\ (gm/cc)} = \frac{\boldsymbol{Dry\ weight\ (D)}}{\boldsymbol{Saturated\ weight\ (W) - Suspended\ weight\ (S)}}$$

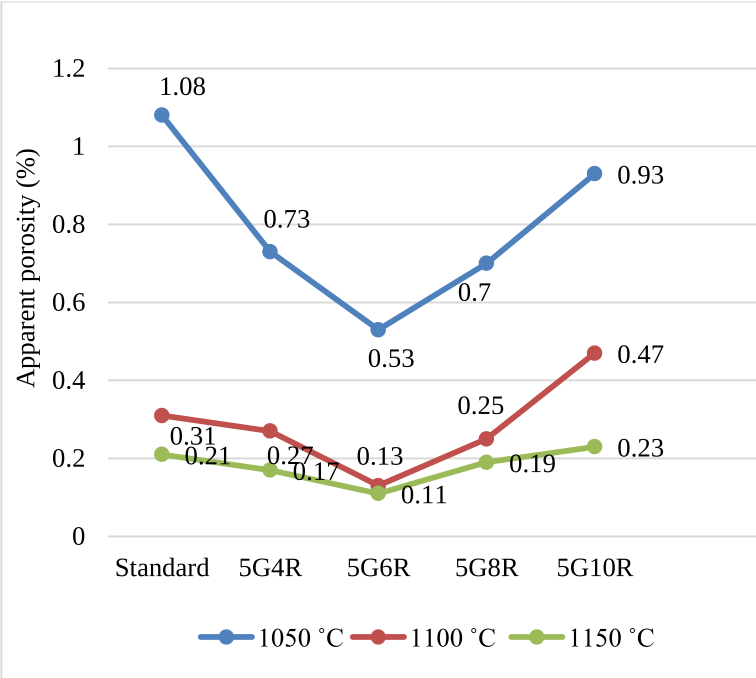


**Fig 2:** Apparent porosity for samples fired at different temperatures

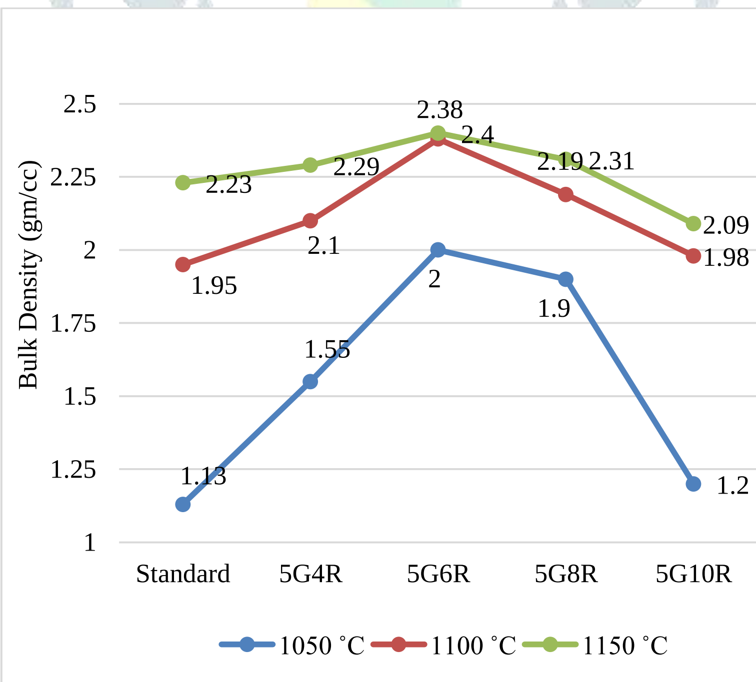


**Fig 3:** Bulk density for samples fired at different temperatures

**Modulus of Rupture**

Modulus of Rupture also known as bending or flexural strength, is a type of characterization which is used to measure the transverse breaking strength of material. It was determined using the following formulae in a three-point bending strength test according to ASTM C-133/97 [12] and results are shown in **Fig 4.** It was found that sample 5G6R has maximum bending strength among all the firing temperature range (1050 ˚C, 1100 ˚C and 1150 ˚C). As the temperature increases, samples has gained the strength however it may obtain a saturation after a limit. Sample 5G6R has highest flux present, which responds in liquid phase formation and this liquid phase is responsible for better densification prior to high strength.

$$\boldsymbol{Modulus\ of\ Rupture} = \frac{3[\boldsymbol{Axial\ load}\ (\boldsymbol{F}) \times \boldsymbol{Length\ of\ support\ span}\ (\boldsymbol{L})]}{2[\boldsymbol{Width}\ (\boldsymbol{b}) \times \boldsymbol{Thickness}\ (\boldsymbol{d})^2]}$$

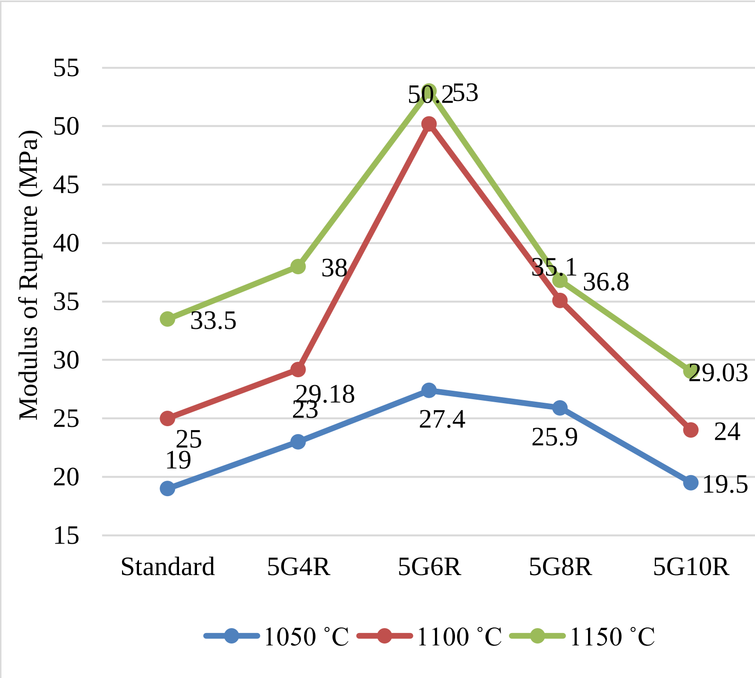


**Fig 4:** Modulus of rupture for samples fired at different temperatures

**X-Ray Diffraction**

XRD analysis was done for the most optimum sample 5G6R, because it has shown better properties even at low firing temperatures. Diffractometer was operated in a range of 10º to 70º of Bragg's angle and result are shown in **Fig 5.** The results were studied using JCPDS file system to identify the crystalline phases developed after sintering. Quartz, Anorthite and Mullite crystalline phases were found to be in majority. Quartz has come up as a residual mineral from the original raw material quartz sand as well as from the crystallization of RHA, beginning from 600 ˚C and reaching completion above 900 ˚C. Mullite has formed during firing but due to such low temperatures, the amount of formation was limited. Anorthite is formed as a consequence of incorporating glass cullets which contains a considerable amount of calcium oxide. Some unreacted amorphous silica coming from RHA were neglected. The low intensities shown by these phases ensure that the sintering behavior of the sample was driven by liquid phase sintering giving rise to more vitrification and densification. The high amount of liquid formation reduces the porosity to some extent but extensively, it can also reduce the mechanical strength after a certain limit due to more glass formation.

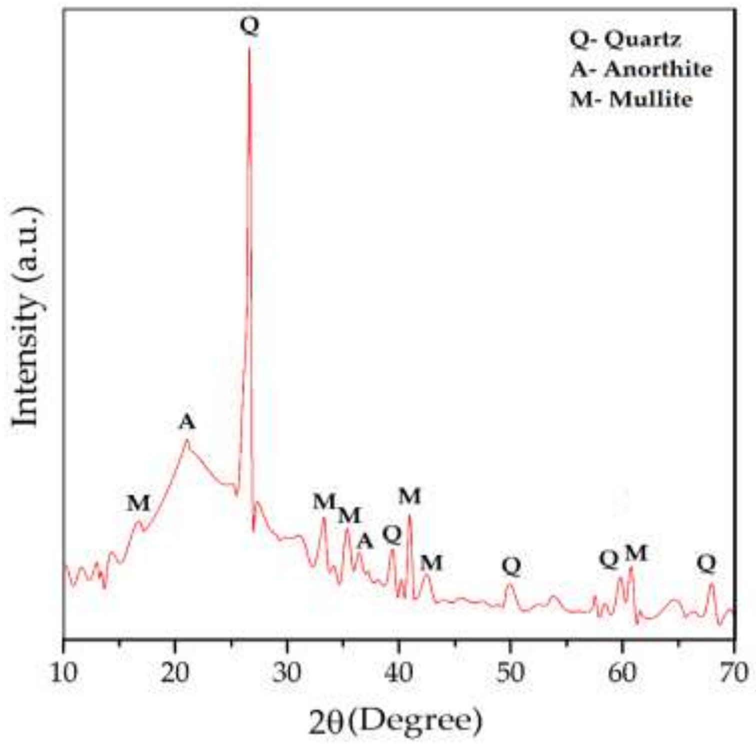


**Fig 5:** X-Ray Diffraction pattern for sample 5G6R

## IV. CONCLUSION

In this experimental study, the optimum sample was 5G6R since it helped in vitrification at lower temperature with giving good mechanical strength. In this way if the temperature is increased (after a limit) for optimum sample, flexural strength starts to decrease due to high amount of glass or amorphous phase formation during liquid phase sintering. It can be concluded that there are two reasons behind this accelerated liquid phase formation. First, the glass cullet which has a high amount of alkali oxide present in it which works as a strong flux. Second, the RHA replacing quartz which has a low melting temperature of 1440 ˚C comparing to 1723 ˚C for quartz. So, it has also found that complete replacement of quartz by RHA emphatically reduces both firing temperature as well as the strength of ceramic tiles which limits its uses upto 5-7 %.

Liquid phase sintering is widely used for consolidation of ceramics into final shapes. Main advantages of this method is to reduce sintering temperature, better densification corresponding to high final densities and resulting microstructures providing mechanical or physical material properties superior to that of solid state sintered materials. Densification achieved through liquid phase sintering is based upon rearrangement and shape change mechanism of solid particles. However, besides rise in densification, liquid phase sintering also affects the grain growth and particle size development adversely comparing to solid state reactions.

Incorporating RHA and glass cullets in ceramic tile production is highly beneficial as using these waste may solve their disposal problem as well as cost reduction in production will also occur because of negligible cost of RHA comparing with quartz. Reduction in the firing schedule either temperature or residence time, also leads to a significant amount of energy saving in the firing stages prior to fuel saving and economically beneficiation. It will also help in reducing greenhouse gases emission and environmental pollution. Reduction in maximum operating temperature of kiln will support in extending life cycle of the refractory lining upto 1-2 years additionally.

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