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## Utilitization of agricultural residues of rice cultivation In manufacturing of light fired clay bricks.

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### ABSTRACT

The aim of the present work is to investigate the utilization of rice husk (RH) and rice straw (RS) as agricultural residues obtained from rice cultivation in Egypt in the manufacture of light fired clay bricks. RH and RS were substituted by weight percent (5%,10%,15%, 20%, 25% and 30%) to clay to form clay/RH and clay/RS bricks. Three sets of experiments were carried out. The first set was concerned with the characterization of the raw materials which covered screen analysis, free silica and organic matters, XRD, DTA and TGA analyses. The second and third sets were dedicated with the preparation and characterization of the mixed unfired and fired clay bricks. The tests of unfired bricks were the linear and volume shrinkage. The tests of fired bricks covered Lose On Ignition (LOI), firing shrinkage, cold and boiling point water absorption, saturation coefficient, apparent porosity, specific gravity, bulk density and compressive strength. The experimental results proved that the best composition of the clay mixture was 90% clay and 10% RH at 700 °C firing temperature, which produce lighter bricks suitable to be used in the construction. The physical, chemical and mechanical properties of the mixed clay/RH brick gave good results in agreement with the standard ASTM.

**Keywords:** Clay, rice husk, rice straw fired and unfired bricks, compressive strength.

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## INTRODUCTION

Brick is a common construction material which is used in dividing and carrying walls. The principal properties of bricks that make them superior building units are their strength, fire resistance, durability, beauty and satisfactory bond and performance with mortar. The quality of the brick depends on the composition of raw materials, production method, firing method and firing temperature [1-5]. Clays are composed mainly of silica, alumina, lime, iron, manganese, sulphur and phosphates. Clays used for brick making vary broadly in their composition and are dependent on the locality from which the soil originates [6,7]. Clay bricks are very durable, fire resistant, and require very little maintenance. Fired clay bricks are one of the known oldest construction materials. Normally, the physical nature of the raw materials controls the manufacturing methods [8-9]. A popular trend by researchers has been to incorporate wastes into fired clay bricks to assist in the production of normal and lightweight bricks. Friendly waste recycling is very important research fields for many decades. The utilization of these wastes reduces the negative effects of their disposal. Also, it would be more profitable to utilize a priceless waste while simultaneously minimizing pollution. Such a situation deals with the dual economical and environmental aspect of the solution. The accumulation of agricultural and industrial solid wastes represent a negative environmental impact in Egypt, if it is not reused or recycled. Rice Husk (RH) and Rice Straw (RS) are by-products of rice milling process. The disposal of large bulk of RH and RS have gain serious concerns due to the importance of preserve a clean environment in the present days. Cultivators in Egypt burn the straw and husks because they believe they have no value. Open field burning or uncontrolled combustion contributes to enormous environmental threats which lower the air quality in the area involved. Over one million acres of rice crops were grown in Egypt. One acre of rice produces 1.6 million tons of husks and two tones of straw after the harvest in October and November. Fore that the aim of the present work is to investigate the utilization of agricultural residues (RH and RS) wastes obtained from rice cultivation in Egypt in the manufacture of light fired clay bricks.

## MATERIALS AND METHODS

The raw materials used for preparation of the target clay bricks were desert clay obtained from Upper Egypt (Beni-Sweif), RH and RS obtained from rice cultivation in Egypt. Three sets of experiments were carried out. The first set was concerned with the characterization of the raw materials, the second and the third sets were dedicated with the preparation and characterization of the mixed unfired and fired clay bricks.

### Preparation and Characterization of the Raw Materials

The raw materials (clay, RH and RS) were subjected to: screen analysis, free silica and organic matters determination, mineralogical analysis (XRD) and thermal analysis (DTA and TGA).

### Preparation, Formation and Testing of Unfired Brick Samples

The wastes were used to replace part of clay as the basic mixture in the unfired clay brick manufacturing, in percentage of weight starting from 0% (reference sample) till 35%, increasing by 5%. Therefore eight mixtures were prepared. These mixtures were mixed on dry basis for 10 minutes for each sample. Cubic brick specimens of approximate dimensions  $50 \times 50 \times 50 \text{ mm}^3$ , were molded by dry pressing using laboratory hydraulic press of 1 ton force with 20% water addition as a binder. Samples were dried on three steps : at 50°C for 24 hours, then at 80°C for three hours, followed by 110°C for another three hours. The brick sample consists of three specimens for each mixture. The linear and volume shrinkage were used for testing the unfired samples.

### Preparation and testing of firing brick samples

Brick specimens were fired using the laboratory furnace with heating rate 10°C/min., at three different firing temperatures (700°C, 750°C, and 800°C), for one hour soaking time, and three hours for each firing temperature. Heating rates were chosen to be as close as possible to industrial conditions. At first, the temperature increases from room temperature to 400°C for 1 hour, then the temperature is raised to 600°C for another 1 hour, in order to provide slowly escape for combined water and prevent crack formation, finally raising the temperature to firing temperatures for another 1 hour. The dimensions and the mass of the formed bricks were measured before and after firing. The tests used for fired samples were Loss On Ignition

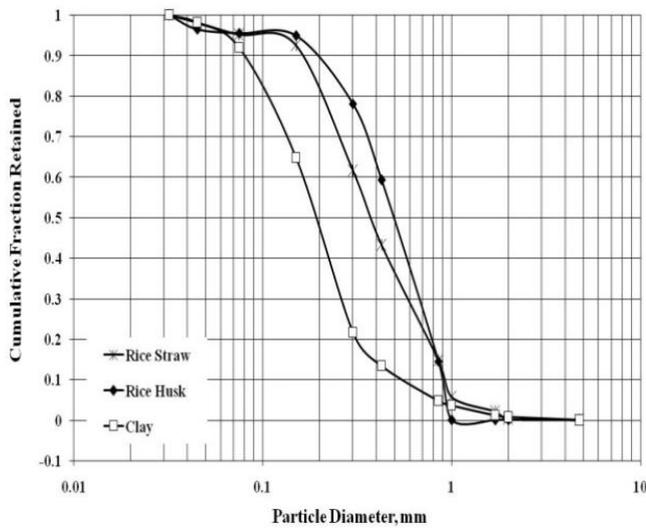
[LOI][10,11]linear and volume firing shrinkage[12], cold and boiling water absorption, saturation coefficient and compressive strength [13],apparent porosity, bulk density, and specific gravity [14],X-Ray Difraction (XRD) and thermal analyses (TGA and DGA).

**RESULTS AND DISCUSSIONS**

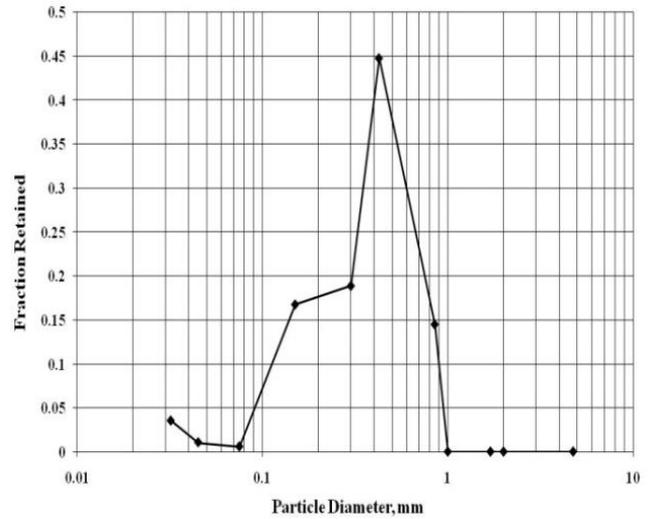
**Characterization of raw materials**

**Screen Analysis**

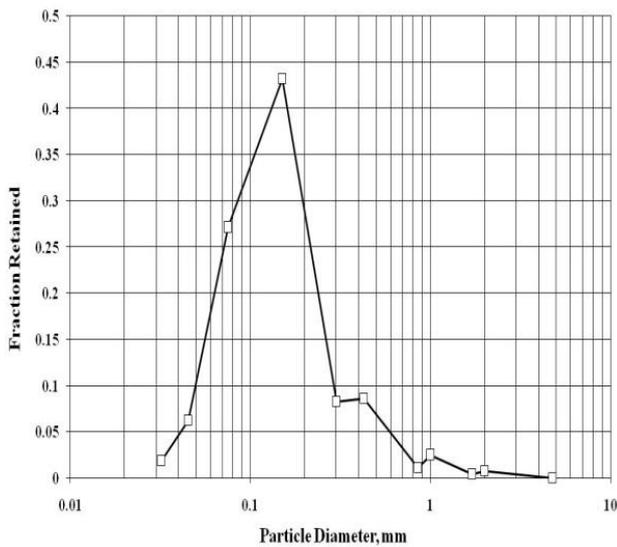
In order to determine the grain size distribution of both clay and wastes, the standard sieving procedure was used. The sieves used are in compliance with the standardized ASTM specifications [15]. The Cumulative screen analysis of raw materials and the differential screen analyses are presented in Figures(1-4).



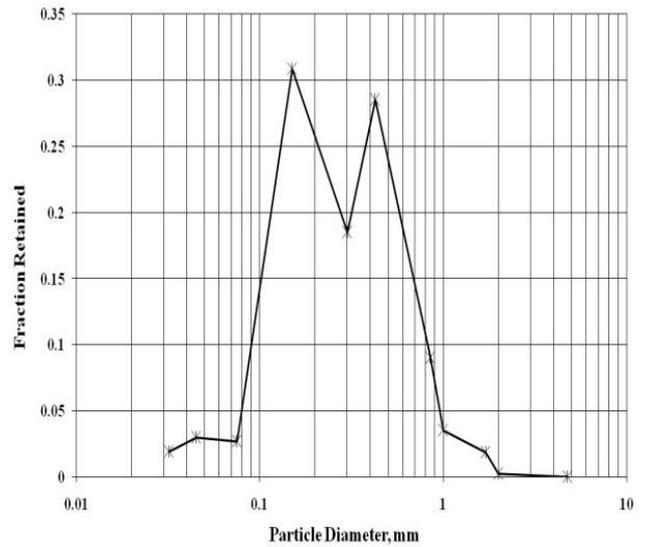
**Figure 1: Cumulative Screen Analysis of Raw Materials**



**Figure 3: Differential Screen Analysis of Rice Husk**



**Figure 2: Differential Screen Analysis of Clay**



**Figure 4: Differential Screen Analysis of Rice husk**

**Chemical Analysis**

Table (1) presents the chemical composition for clay, RH and RS by using the X-Ray Diffraction (XRD) which indicates that clay has the highest percentage of silica oxide (43%), followed by RH (13.28%) then RS (7.57%). The silica in clay presents in different forms as a free silica ( $\text{SiO}_2$ ) and in the form of compounds when mixed with other elements such as aluminum oxide  $\text{Al}_2\text{O}_3$  to form kaolinite in the feldspar group.

**Table 1: Chemical Analysis of Clay, Rice Husk and Rice Straw (as Oxides)**

Constituents, Wt. (%)	Clay	Rice Husk	Rice Straw
$\text{SiO}_2$	43.08	13.28	7.57
$\text{Al}_2\text{O}_3$	16.54	0.22	0.13
$\text{Fe}_2\text{O}_3^{\text{tot.}}$	8.08	0.57	0.6
$\text{TiO}_2$	0.91	0.08	0.07
MgO	1.38	0.13	0.19
CaO	9.00	0.77	1.16
$\text{Na}_2\text{O}$	3.40	0.06	0.24
$\text{K}_2\text{O}$	1.29	2.22	3.90
$\text{P}_2\text{O}_5$	0.18	0.26	0.12
$\text{SO}_3$	1.91	0.33	0.36
SrO	0.16	0.02	0.02
$\text{Cr}_2\text{O}_3$	0.02	—	—
MnO	0.03	0.06	0.11
$\text{ZrO}_2$	0.07	—	0.004
$\text{Co}_3\text{O}_4$	0.02	—	—
ZnO	0.01	—	—
$\text{CeO}_3$	0.04	—	—
NiO	0.01	0.02	0.01
CuO	0.01	0.01	0.01
$\text{Nb}_2\text{O}_3$	0.01	—	—
$\text{Rb}_2\text{O}$	0.01	—	—
Cl	0.59	0.41	1.43
Br	—	0.01	0.01
L.O.I	13.26	81.75	84.07
<b>Total</b>	<b>100</b>	<b>100</b>	<b>100</b>

**Determination of Free Silica and Organic Matters**

The weight percent of organic matters and the free silica for raw materials were determined and presented in Table (2). It is remarkable that clay has the highest percentage of free silica content (22.82%), followed by RH (17%), then RS (11.80%). In addition it is noticeable that the organic matters release from RH and RS are nearly the same but they are of high values when compared with the clay.

**Table 2: Free Silica and Organic Matters in Raw Materials**

Raw Material Component	Clay	Rice Husk	Rice Straw
Organic Matter, %	5.07	68.7	70.56
Free Silica, %	22.82	17.00	11.80

**Mineralogical Analysis**

The XRD pattern of clay is shown in Figure (5). It indicates that the clay composition consists of quartz, calcite, kaolinite, montmorillonite and albite. Figures (6&7) represent the XRD pattern of RH and RS, which proves that the silica exists in the amorphous form, but it is reported that it will not remain amorphous, when incinerated for a prolonged period at a temperature above 700°C [16]. The XRD results also indicates that the RH and RS presents in their composition amorphous silica which is similar to that commonly occurring in clay as reported by other authors[17].

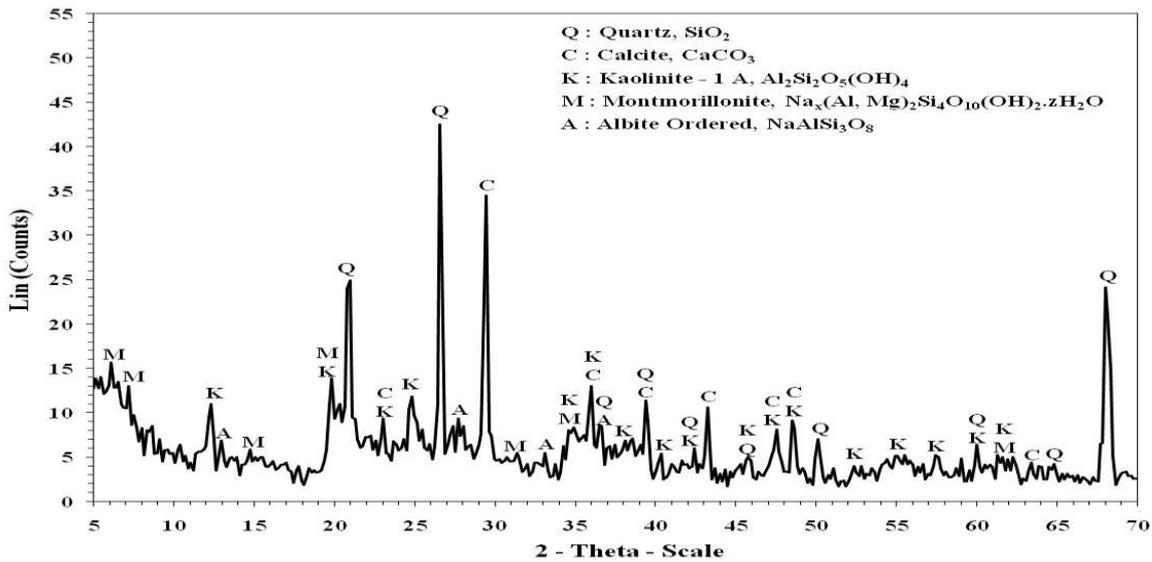


Figure 5: XRD of Clay

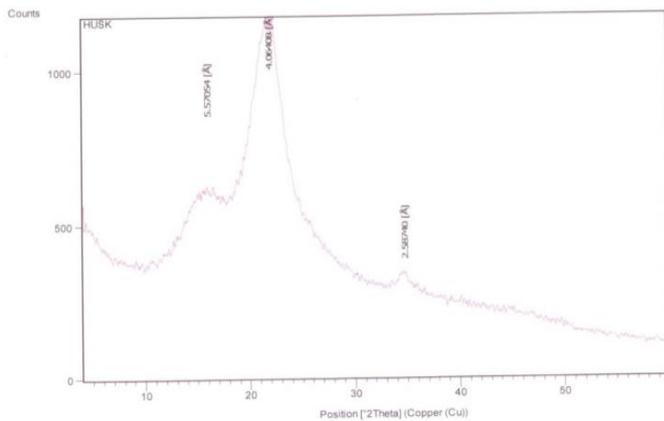


Figure 6: XRD Pattern for Rice Husk

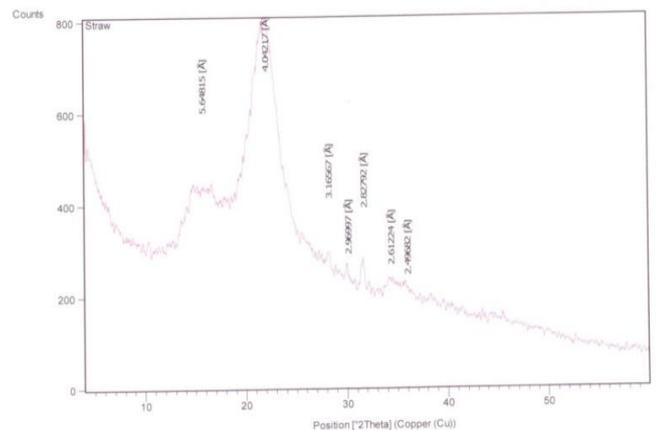


Figure 7: XRD Pattern for Rice Straw

**Thermal Analysis**

The effect of heat on the clay was studied using TG-DTA as shown in Figure (8). The TG-DTA analysis curves present the changes in the clay when heated. From the thermo gravimetric curve, the sample exhibits a decomposition loss of about 19 % of weight, it means that about 81% was left undecomposed at 1000°C. The clay started losing water when heated from 54°C to 127°C. The big changes can be seen between 400°C to 470°C where the dehydroxylation of clay minerals occurred. The differential thermal analysis curve shows three sharp exothermic peaks appeared at 54°C, 471°C and 665°C corresponding to the loss of surface water and to dehydroxylation of the clay material [6].

Figures (9&10) represent the TG-DTA curves of RH and RS. It is observed that the total weight loss was about 80% at 1000°C. The first 9% decreased in the mass occurred between 50-180°C due to the evaporation of physical water [18]. The second mass loss was observed in the range of 200°C-400°C which may be due to burn-out of volatile organic components (weight loss around 59%). After 400°C, a slow rate of

weight loss occurred until 700°C. It is reported that the rice husk mainly consists of three basic constituents of biomass; hemicelluloses decomposed at 220-350°C, cellulose decomposed at 325-375°C and lignin decomposed at 200-700°C [19]. In addition, it is reported that the weight loss during thermal treatment of RH in air may be attributed to three stages such as drying at 40°C –150°C for removal of physically bonded water, burn-out of volatile organic component at 215°C –350°C, followed by degradation of carbonaceous phases cellulose and hemi-cellulose (>350 °C)[20].

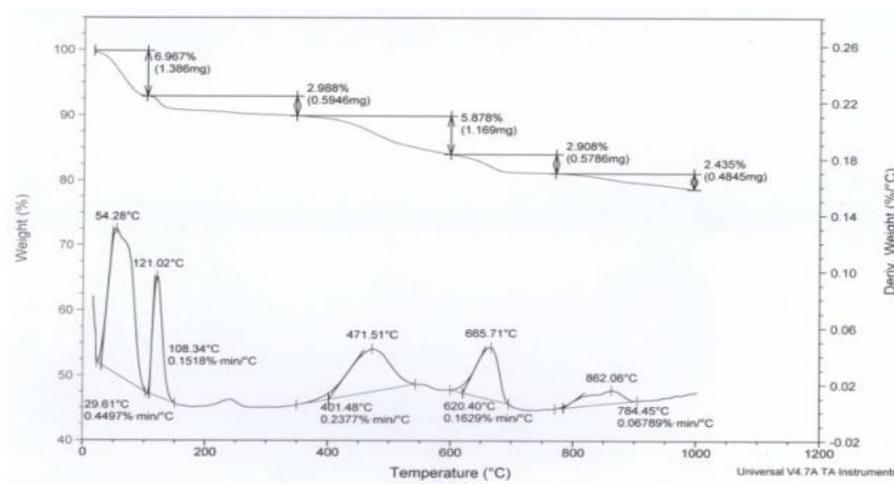
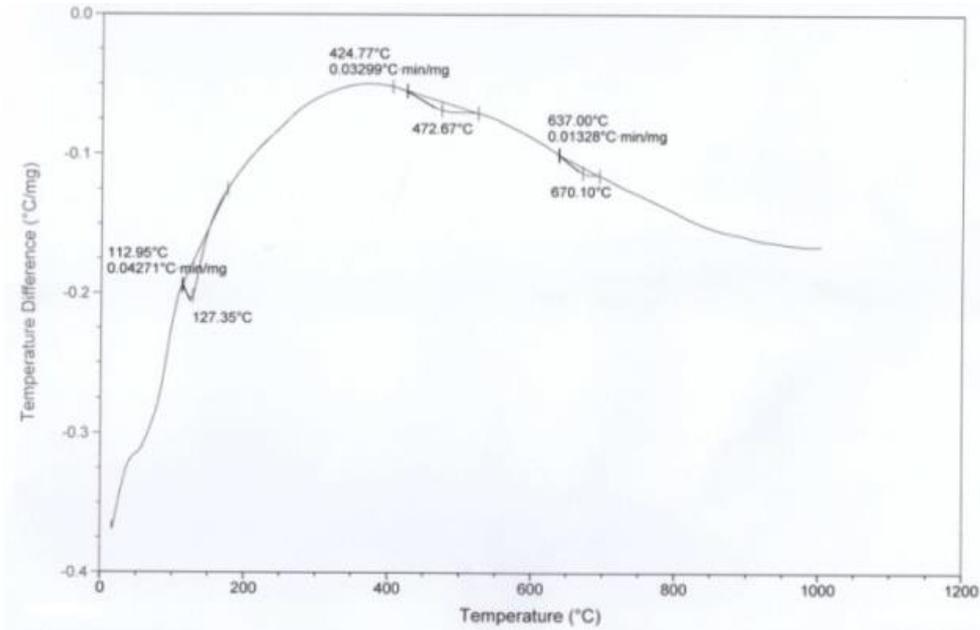


Figure 8: TG-TDA Pattern for Clay

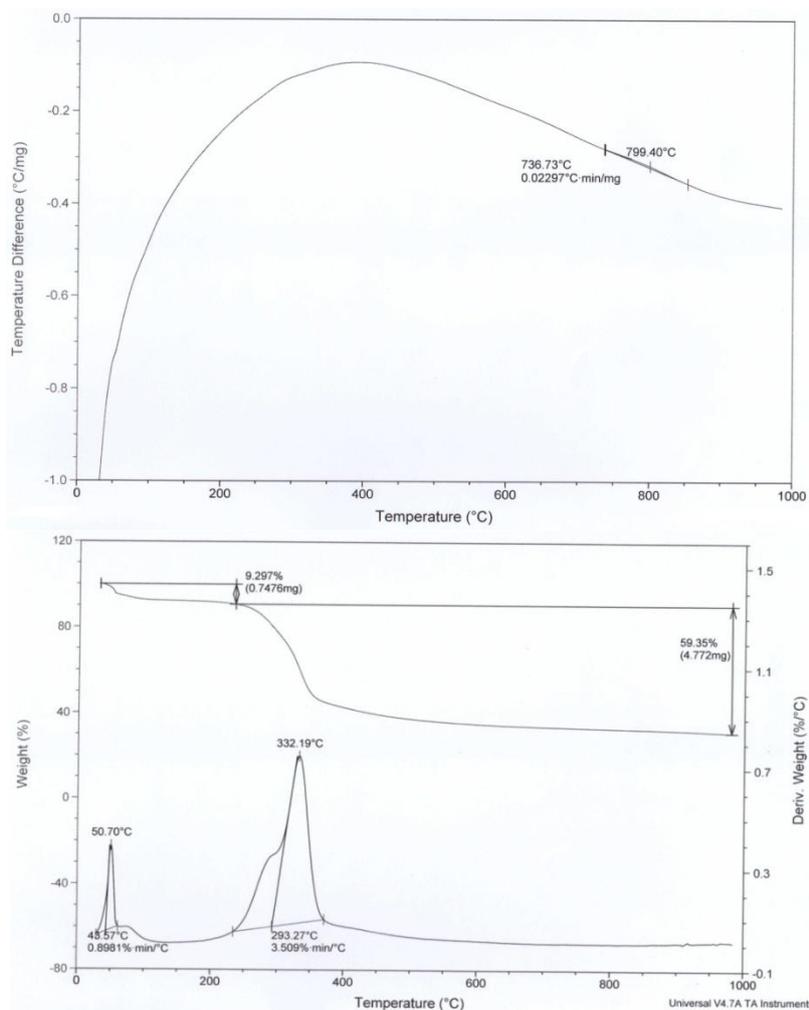
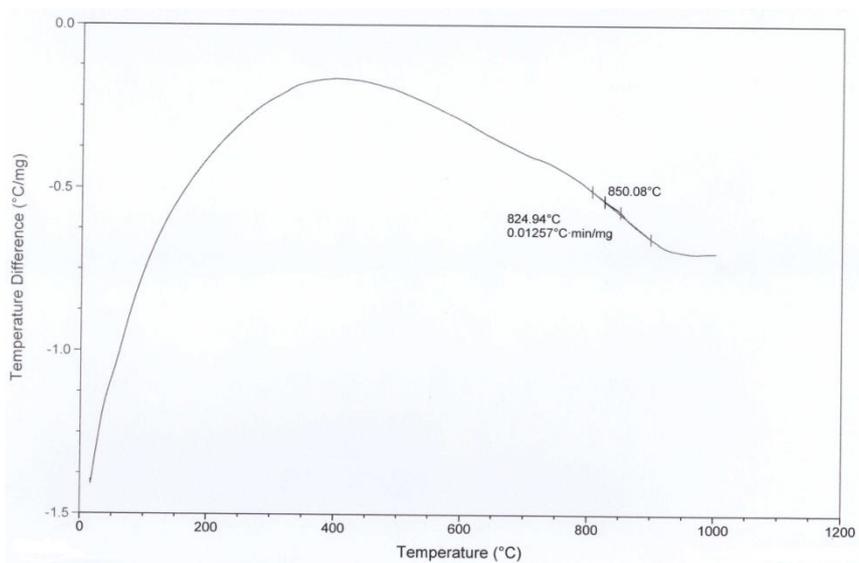


Figure 9: TG-DTA Pattern for Rice Husk



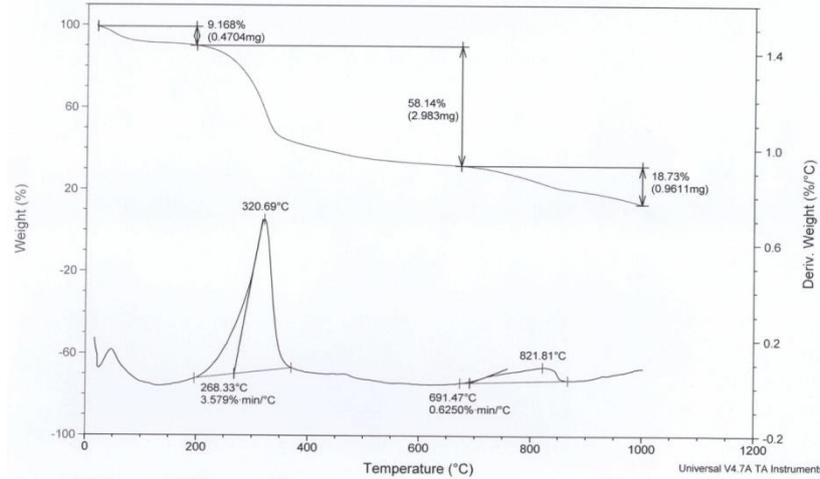


Figure 10: TG-DTA Pattern for Rice Straw

**Testing of Prepared Green Brick(Unfired) Samples**

The linear and volume shrinkage of unfired brick samples were measured according to ASTM standard[12]. The purpose of this test is to obtain values of both linear and volume shrinkage after drying under various processing conditions to enable designers to determine the proper size of mold so as to produce a predetermined size of fired ware. The shrinkage takes place in a constant rate period, and happens due to the amount of layer water evaporated during drying. Effect of RH and RS addition on linear and volume drying shrinkage of clay mixtures are presented in Figures (11&12). By comparing with reference samples it is observed that the linear and volume drying shrinkage percentage of clay/RH and clay/RS specimens decrease as wastes addition increase from 5% to 25% then remain nearly constant .

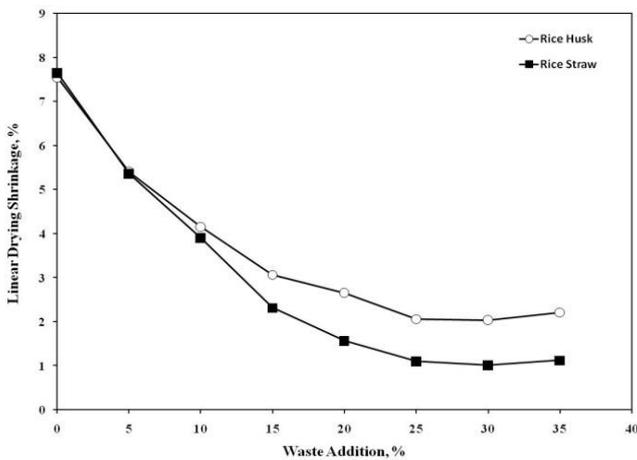


Figure 11: Effect of Waste Addition on Linear Drying Shrinkage

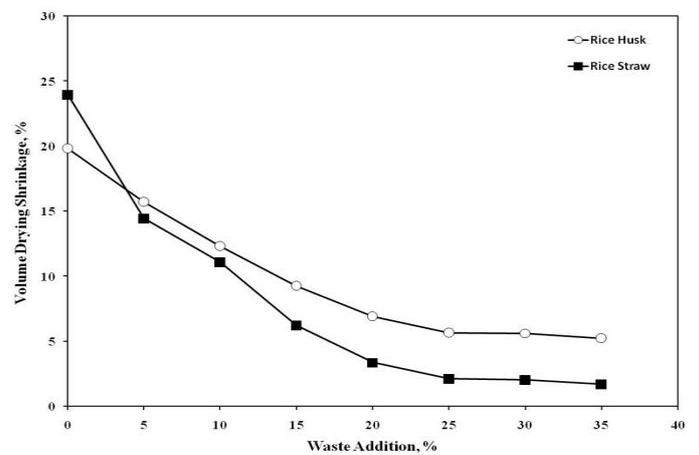


Figure 12: Effect of Waste Addition on Volume Drying Shrinkage

**Testing of Fired Brick Samples**

**Loss OnIgnition**

The LOI values is important to indicate the amount of moisture or impurities lost when the sample is ignited under the firing conditions. The LOI of fired samples were measured according to standard ASTM[11]. Figures(13-a &13-b) summarized the value LOI for the different substitution percentages of RH and RS to the clay at three firing temperatures(700°C, 750°C and 800°C).From Figure 13-a the LOI increases as RH% increases from 0 till 20% for all firing temperatures, but in case of substituting of RS percentage to clay as presented in Figure 13-b the LOI increase to double its value at nearly 5%RSsubstitutionforall firing temperatures then remain nearly constant which means that this crystalline phase was in stable condition and

only carbon will evaporate. Fore that it can be concluded that the LOI at 10% RH and 5% RS substitution for 700°C firing temperature is nearly doubled the LOI value of reference samples.

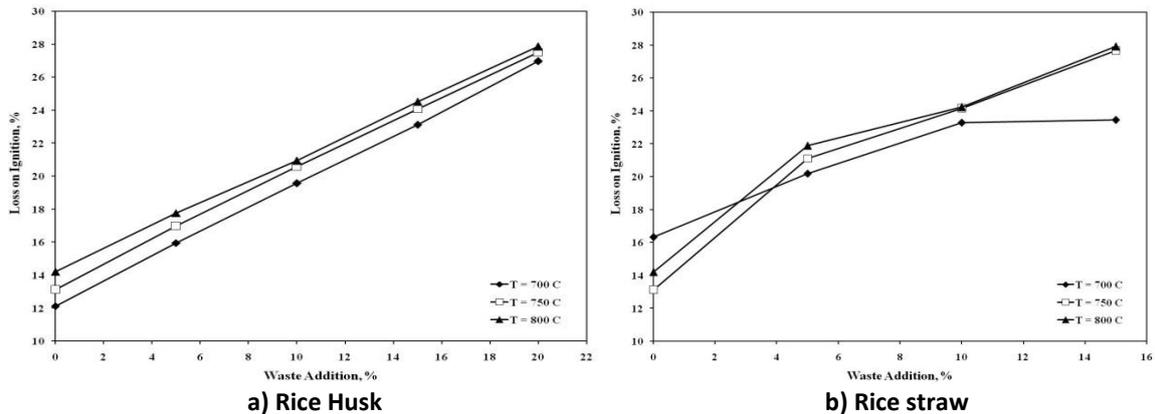


Figure 13: Effect of Waste Addition on Loss Ignition of Fired Samples

**Determination of Firing Shrinkage**

The firing shrinkage gives an approximate idea of the efficiency of the firing process. From figures (14&15), by comparing the linear and the volume shrinkage of reference samples with clay/RH and clay/RS samples, it is noticeable that linear a and volume shrinkage will increase till 5% waste substitution, then decreased till 10% wastes substitution, and finally increased for the higher substitution percentages. Therefore the Clay/RH and Clay/RS of 10% wastes substitution proved their suitability for using in construction building.

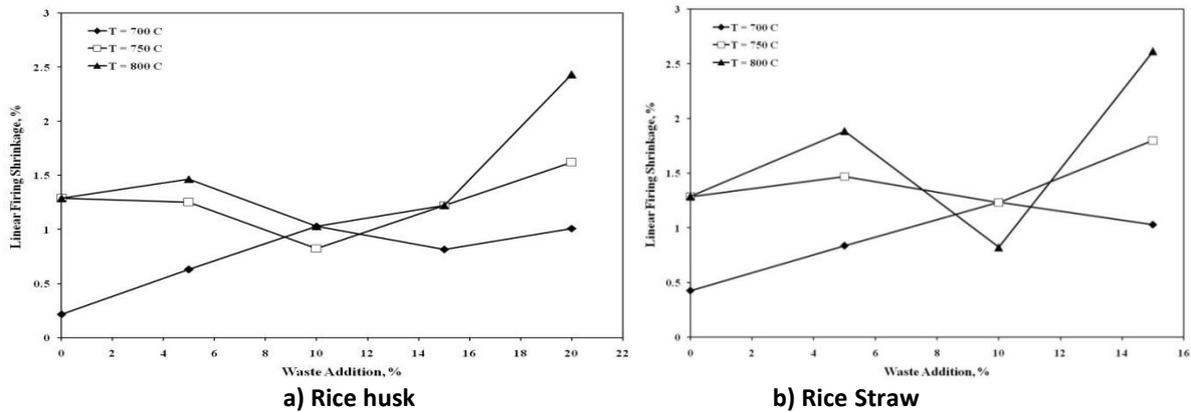


Figure (14) Effect of Waste Addition on Linear Firing Shrinkage

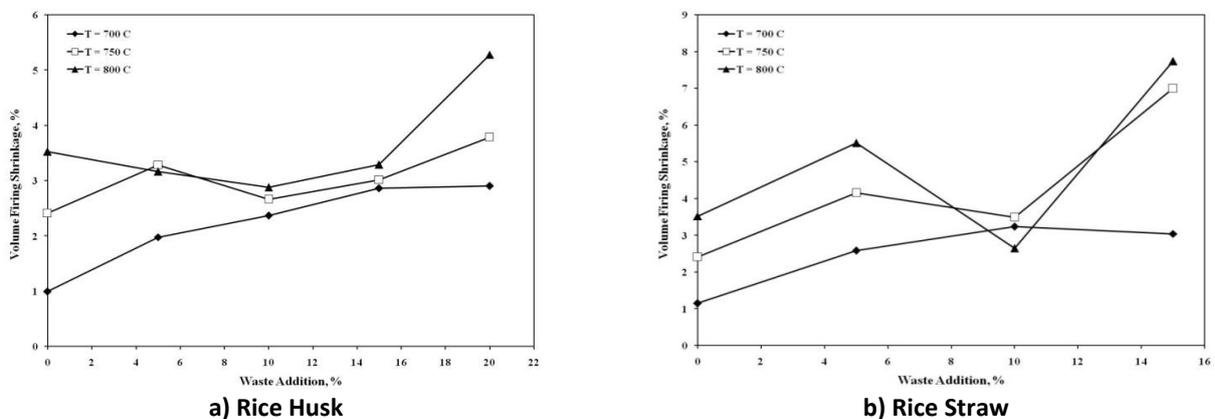
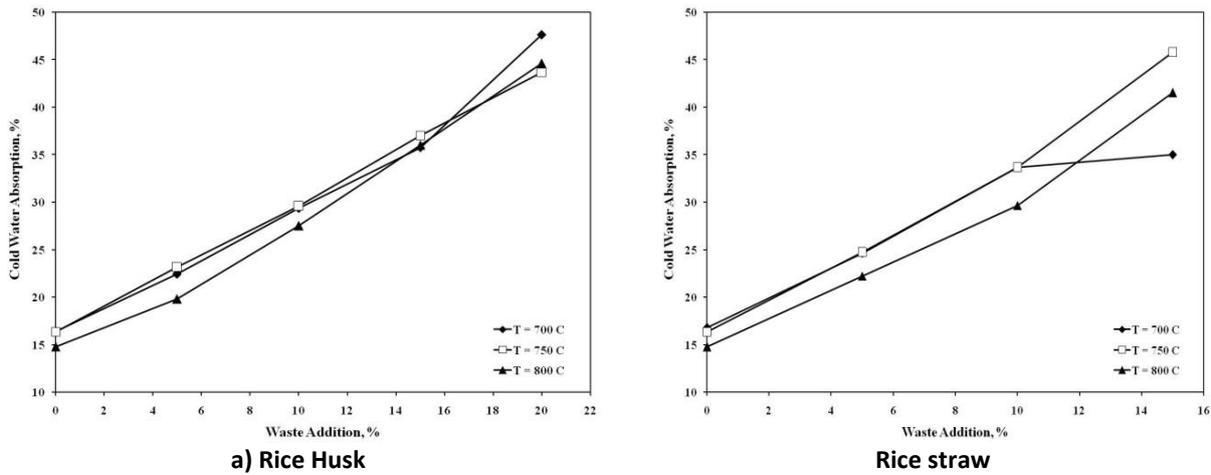


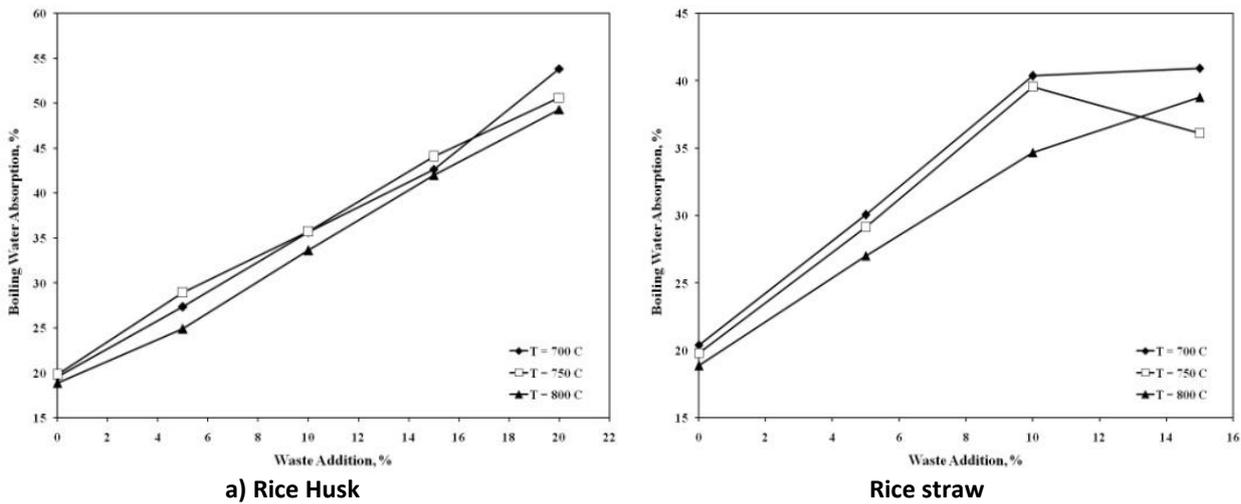
Figure 15: Effect of Waste Addition on Volume Firing Shrinkage,

**Determination of Cold and Boiling Water Absorption**

Water absorption test is used as an indication for the specimens resistance to immersion. It is related to pore volume connected to the surface of sample. It is a measure to the open porosity [17], also it is closely related to density. According to ASTM [15] the suitable Clay/RH or Clay/RS must have water absorption  $\leq 25\%$ . In case of cold water addition as presented in Figure(16) the standard 25% water absorption is achieved at 10% percentage substitution for both RH and RS of clay for the three fired temperature (700°C, 750°C, and 800 °C). But it is remarkable that in case of boiling water Figure (17) the standard 25% water absorption is achieved at 8% only of RH and 5% of RS substitution for the different temperatures. The water absorption decreases may be due to pores are rounding and becoming smaller when compared to solid state and completely isolated from the surface at the end of sintering, and then closed because of vitreous phase formations. This phase penetrates into pores, and closes them, and then separates from neighbouring pores. That mechanism may be due to the reduction in water absorption with increasing sintering temperature [8]. Therefore the Clay/RH is the suitable one for using in constructions.



**Figure 16: Effect of Waste Addition Percent on Cold Water Absorption**



**Figure 17: Effect of Waste Addition Percent on Boiling Water Absorption Percentage**

**Determination of Saturation Coefficient**

From Figure (18) the substitution of 18% RH and 10% RS to the clay proved saturation coefficient  $\leq 0.9$  for the three different firing temperatures which is in good agreement with the ASTM standards[17].

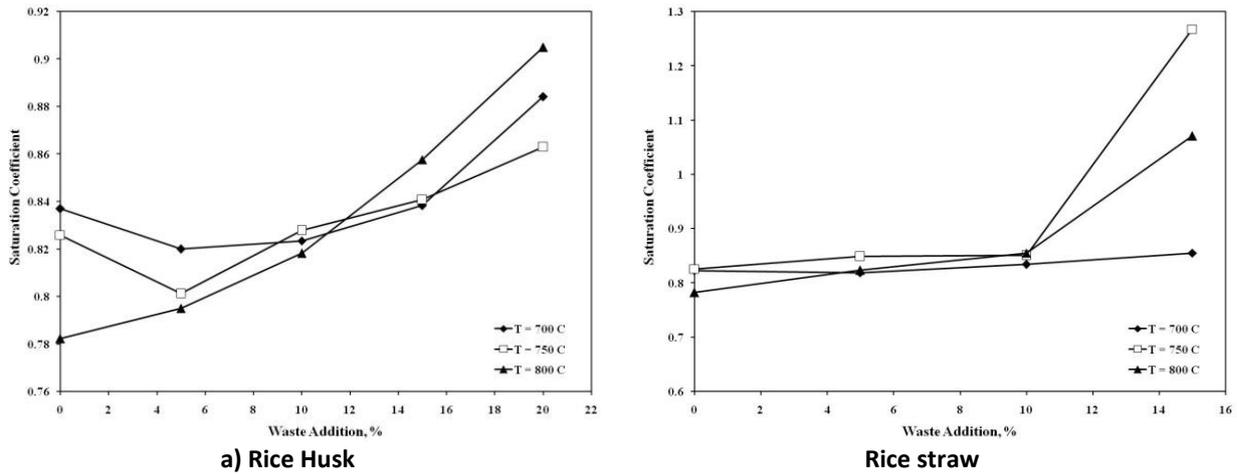


Figure 18: Effect of Waste Addition on Saturation Coefficient, a) Rice Husk, and b) Rice Straw

**Determination of Apparent Porosity, Specific Gravity and Bulk Density**

It is remarkable from Tables (3-5) that the apparent porosity, the specific gravity and the bulk density were increased as waste addition increased and decreased as temperatures increased for all firing temperatures and all percentage of wastes substitution. It has been reported that calcareous or non calcareous low refractive clays in oxidizing conditions widely goes into vitrification at 800°C[21].Based on these results we can consider that 10% RH and 10% RS are suitable for manufacturing of clay mixed bricks at 700°C and less.

**Table 3: Apparent Porosity Percent of Control, Clay/RH and Clay/RS samples at Different Temperatures**

Waste add. % Temp., °C	700	750	800	700	750	800
0	34.25	33.95	32.7	34.37	33.95	32.7
	Apparent porosity for Clay/RH			Apparent porosity for Clay/RS		
5	42.12	42.12	37.17	44.51	42.84	39.53
10	48.39	47.21	44.83	50.90	49.99	43.95
15	52.31	51.68	48.99	50.37	36,23	40.48
20	53.71	53.87	51.08	50.35	36.20	40.45
25	53.80	53.9	52.00	50.30	36.10	40.40

**Table 4: Specific Gravity Percent of Control, Clay/RH and Clay/RS samples at Different Temperatures**

Waste add. % Temp., °C	700	750	800	700	750	800
0	2.66	2.59	2.57	2.56	2.59	2.57
	Specific gravity for Clay/RH			Specific gravity for Clay/RS		
5	2.65	2.54	2.37	2,66	2.57	2.41
10	2.63	2.50	2.41	2.56	2.52	2.26
15	2.56	2.42	2.28	2.47	1.57	1.75
20	2.15	2.30	2.11	2.43	1.53	1.72
25	2.05	2.24	2.00	2.41	1.51	1.71

**Table 5: Bulk Densities Percent of Control, Clay/RH and Clay/RS samples at Different Temperatures**

Waste add. % Temp., °C	700	750	800	700	750	800
0	<b>1.74</b>	1.71	1.73	1.68	1.71	1.73
	<b>Clay/RH</b>			<b>Clay/RS</b>		
5	<b>1.49</b>	1.5	1.46	1.49	1.48	1.47
10	<b>1.34</b>	1.35	1.32	1.33	1.26	1.26
15	<b>1.20</b>	1.22	1.17	1.17	1.23	1.00
20	<b>1.98</b>	0.99	1.06	1.03	1.20	0.99
25	<b>0.97</b>	0.98	1.00	1.02	1.20	0.98

**Table 6: Compressive Strength of Control, Clay/RH and Clay/RS Samples at Different Temperatures**

Waste add. % Temp., °C	700	750	800	700	750	800
	<b>Compressive strength, N/mm<sup>2</sup></b>					
0	15.8	14.0	12.0	16.0	14.0	12.0
	<b>Clay/RH</b>			<b>Clay/RS</b>		
5	9.5	4.3	4.3	5.0	4.7	4.7
10	8.7	4.0	4.2	4.0	4.0	4.2
15	1.0	1.0	1.0	1.0	1.0	1.0
20	0.5	0.5	0.5	0.5	0.5	0.5

**Determination of Compressive Strength**

According to standard ASTM [15] compressive strength is a very important parameter which is used to meet engineering quality in construction material applications. The standard compressive strength according to ASTM[15] must be  $\geq 8.6 \text{ N/mm}^2$

The compressive strength of all fired brick samples(reference, Clay/RS & Clay/RH )at the three different temperatures were determined and the mean values are given in Table [6]. It is decreases as the temperature increases. The best compressive strength was achieved at  $8.7 \text{ N/mm}^2$  at  $700^\circ\text{C}$  for 10% RH waste substitution which is in good agreement with the standard ASTM, but the compressive strength in case of 10%RS was  $4 \text{ N/mm}^2$  which is less than the allowable standard limits. So to obtain good compressive strength of Clay/RS (with 10% waste addition) the temperature must decreased to less than  $700^\circ\text{C}$ . The decrease of compressive strength at  $700^\circ\text{C}$  and above is due to “Bloating” of the samples as a result of verification stage of firing and attributed to the generation of gas within the clay mass [7].

**CONCLUSION**

The properties of clay substitution by different percentages of RH and RS were investigated. Clay/RH of 10% RH substitution proved good agreement with standard ASTM. The physical, chemical and mechanical properties of the the optimum Clay/RH brick were:

- Composition: 95% clay and 10% rice husk
- Firing temperature:  $700^\circ\text{C}$
- Compressive strength:  $8.7 \text{ N/mm}^2 \leq 12 \text{ N/mm}^2$  ( standard limit)
- Saturation coefficient:  $0.83 \leq 0.9$  (standard limit)
- Cold Water absorption:  $25\% \leq 25$  (standard limit)
- LOI: 22%
- Linear fire shrinkage: 0.75
- Volume fire shrinkage 2.3
- Apparent porosity: 44
- Bulk density:  $1.35 \text{ gm/cm}^3$
- Specific gravity: 2.65

**ACKNOWLEDGMENT**

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