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Study of Raw Materials for Making Ceramic Product and Physico-Mechanical Properties of Ceramic Product Made from Rock Residue Powder†

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The present research work is on the use of the rock waste material (rock residue powder) for manufacturing ceramic product. The mineral phases and chemical composition of raw materials (fire clay and rock residue) were determined by X-ray diffraction and standard chemical analysis method respectively. In the present investigation, the mixture of various proportions of rock residue and fire clay material are used to make ceramic body specimens and these specimens fired at 800 ºC. The (unfired and fired) ceramic bodies were investigated to determine their physical and mechanical properties. All the results were indicated that the physico-mechanical behaviour increases with increasing rock waste content.

Key Words: Ceramic product, Waste material, Rock residue powder, XRD technique, Chemical analysis, Properties.

INTRODUCTION

Waste recovery is a very important from the public health, the environmental and industrial perspectives. The use of solid waste as useful raw material is strongly recommended, since it reduces the negative environmental impact associated with landfill and preserves non-renewable nature. In the last few years, many researchers were interested in studying the problem of industrial solid wastes. This paper presents some of the results from a continuous study of recycling waste into ceramic product.

Rock waste such as residue powders, which result from cutting, polishing, crushing and machining operations in the rock quarries have a significant and serious impact on the environment. The rock waste is potentially pollutant[1] although only a small part of it is correctly discarded. Most of rock waste is illegally dumped in land and in the environment. This practice causes negative effects on the environment and human health, since the fine powder in suspension can be inhaled[2,3]. The amount of powdery rock waste is estimated to be in the range of 20-25 % of total production[4].

The present research work is on the use of the waste material (rock residue) for manufacturing ceramic product. A typical ceramic body formulated using a rock residue in the form of powder was selected as one raw material and mixture of another raw material is fire clay. The objective of this work is to study the possibility to incorporated rock residue powder in ceramic product, without degrade of their properties.

EXPERIMENTAL

The rock residue powder collected from a private rock-crushing plant located in Thuraiyur, Trichy district of Tamilnadu, India and fire clay material (FC) collected from Government Ceramic Institute, Virdhachalam of Tamilnadu.

The mineral phases of raw materials (RS and FC) were determined by X-ray diffraction technique and also, the chemical compositions of raw materials (RS and FC) were determined by chemical analysis method[5,6].

The raw materials, rock residue powder and fire clay material were dried and ground in an agate mortar. The various proportion of rock residue 0 % (control), 10, 20, 30, 40 and 50 % were mixed with fire clay material by weight basis. The ceramic bodies were moulded into ASTM standard rectangular bars using a uniaxial pressing technique. After forming, the ceramic body specimens were dried in open atmosphere (3 days) and then in an oven dried for 24 h at 110 ºC. Then dried samples sintering were performed in a laboratory muffle furnace at 800 ºC. The heating rate was 5 ºC/min. with 1 h soaking at maximum temperature. Six experimental ceramic bodies were prepared and named RW0, RW10, RW20, RW30, RW40 and RW50.

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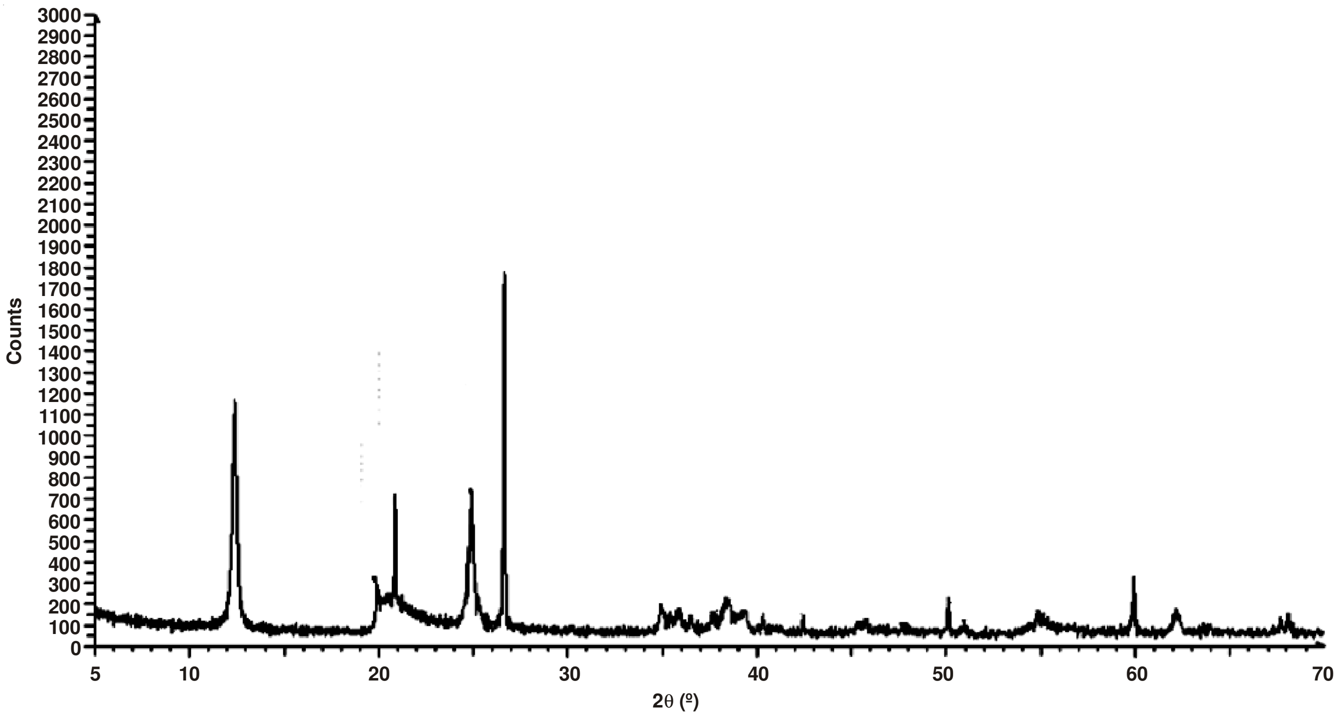

Fig. 1. XRD pattern of the fire clay material (FC)

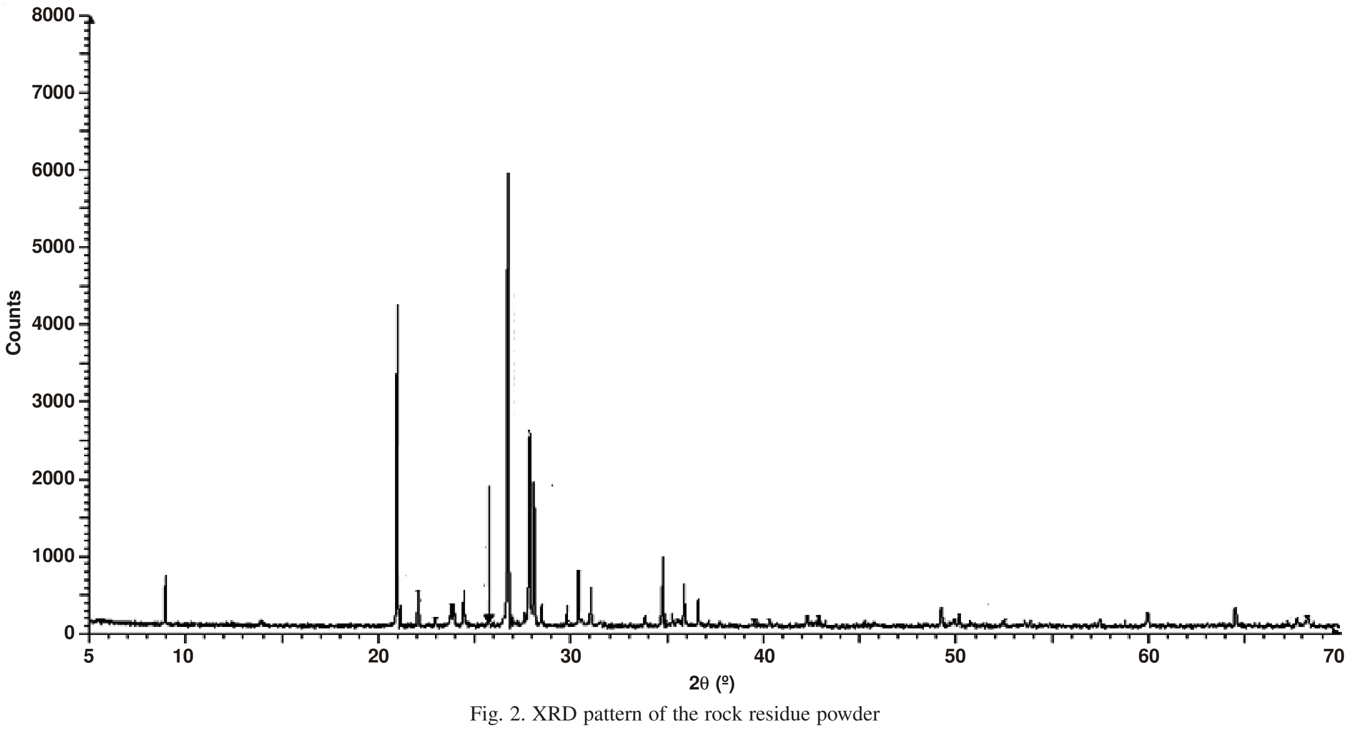

Fig. 2. XRD pattern of the rock residue powder

The ceramic bodies (RW0-RW50) were tested to determine plasticity, drying density, firing density, compressive strength (green strength & fired strength), drying shrinkage, firing shrinkage, appearance and colour by using standard methods[7-10].

RESULTS AND DISCUSSION

Study of the raw materials (fire clay and rock residue powder) for making ceramic product: The raw materials (fire clay and rock residue powder) used for ceramic product, were subjected to XRD technique and chemical analysis.

X-ray diffraction analysis: X-ray diffraction technique has been used most frequently for qualitative identification of mineral phases of raw materials (fire clay and rock residue powder) and the X-ray diffraction patterns obtained are presented in the Figs. 1 and 2.

The X-ray diffraction pattern of sample of the fire clay material is shown in Fig. 1. The identified minerals are: Kaolinite -(1.542, 1.790, 1.902, 2.387, 2.384, 2.400, 2.504, 3.581, 4.263, 4.456 and 7.181 Å), montmorillonite-(1.491 Å), quartz-(1.375, 1.671, 1.819, 2.128, 2.238, 3.346 Å and 4.263 Å) aragonite (carbonate mineral)-(1.983 Å) and hematite-

(2.291 Å). These results were obtained from the searching and matching of d-spacing values in the JCPDS file[11].

The X-ray diffraction pattern of the rock residue powder (RS) is presented in Fig. 2, the identified minerals with *d*-spacing values are: kaolinite-(1.54 Å, 1.742 Å, 1.795 Å, 2.500 Å, 3.041 Å, 3.456 Å), montmorillonite-(9.956 Å, 2.579 Å, 2.646 Å), quartz-(1.301 Å, 1.372 Å, 1.391 Å, 1.441 Å, 1.601 Å, 1.816 Å, 1.847 Å, 2.101 Å, 2.134 Å, 2.234 Å, 3.197 Å, 3.336 Å, 4.0274 Å, 4.239 Å), albite-(2.461 Å) orthoclase-(3.733 Å), calcite-(2.886 Å, 2.992 Å, 3.871 Å) and hematite-(2.275 Å,2.938 Å).

All the peaks are assigned in accordance with those reported in the literature[12-14], joint committee on powder diffraction standards 11 (JCPDS) and the international centre for Diffraction data (ICDD) powder diffraction file[15].

The XRD analysis shows the presence of kaolinite, montmorillonite, quartz, feldspar mineral (albite and orthoclase), carbonate mineral (calcite and aragonite) and hematite are present in the both raw materials and these present minerals are summarized in Table-1.

TABLE-1
MINERAL CONSTITUENT IN THE RAW MATERIALS BY USING XRD TECHNIQUE

Minerals	Fire clay	Rock residue
Kaolinite	✓	✓
Montmorillonite	✓	✓
Quartz	✓	✓
Calcite	✓	✓
Aragonite	✓	✓
Albite	-	✓
Orthoclase	-	✓
Hematite	✓	✓

Chemical analysis: The chemical analysis of the raw materials (fire clay and rock residue powder) were made by using standard method followed by rock and mineral analysis[6].

The present investigation deals with the quantitative estimation of mineral content was determined by using method of chemical analysis and it has been concluded the presence of silica (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3), titanium dioxide (TiO_2), calcium oxide (CaO), magnesium oxide (MgO), potassium oxide (K_2O), sodium oxide (Na_2O) and phosphorus pentoxide (P_2O_5) was detected in the raw materials (fire clay and rock residue powder) and this results are given in Table-2.

The constituent chemical composition and their respective influence on raw materials are as follows[16].

TABLE-2
CHEMICAL COMPOSITIONS (wt. %) OF THE RAW MATERIALS

Chemical composition	Raw material (wt. %)	
	Fire clay	Rock residue
SiO_2	59.600	59.140
Al_2O_3	18.620	15.340
Fe_2O_3	16.480	6.880
TiO_2	2.810	1.050
K_2O	0.030	3.130
MgO	0.340	3.490
CaO	0.670	5.080
Na_2O	0.010	3.840
P_2O_5	0.020	0.200
SiO_2/Al_2O_3	3.201	3.885

(1) Al_2O_3 and SiO_2 - this is responsible for the refractoriness, shrinkage and plasticity of a sample; (2) Fe_2O_3 - this constitution determines the colour of the sample after firing.

The basically characterized by high amount of SiO_2 and Al_2O_3. The large amount of SiO_2 is associated with crystalline phase quartz and combined to Al_2O_3 in the aluminosilicates structure[16-18]. Most part of the alumina is combined to form the structure of aluminosilicates as the kaolinite. The raw materials (fire clay and rock residue) are present low amount of Al_2O_3 and high amount of SiO_2, whose SiO_2/Al_2O_3 ratio varied between 3.21 and 3.856. This result indicates that the studied raw material have high amount of clay mineral[17,18]. The amount of earth-alkaline oxide (CaO and MgO) that are auxiliary fluxes also is low, indicating that the studied raw materials and non-carbonatic[17,18].

Generally, the raw materials have high value of silica, alumina and iron oxide are useful to make quality of ceramic product. From the samples (FC and RS), the chemical composition ($SiO_2 + Al_2O_3 + Fe_2O_3$) is high values (94.7 % and 81.36 %), its indicative is suitable to make quality ceramic product. These results agree with the literature[19].

Physical and mechanical properties of ceramic product: Parameters relevant to the study of the main properties of ceramic materials are plasticity, drying density, firing density, compressive strength (green strength and fired strength), drying shrinkage, firing shrinkage, appearance and colour. To determine these factors which affect the quality of ceramic product considerably, is main theme of this study.

At least five specimens were used in each test for all categories and the average values are presented and discussed in this section. In the present study, the ceramic bodies are analyzed for some important properties reports are summarized in Table-3.

Plasticity: Plasticity of the clay bodies is great importance in the shaping of ceramic materials[20]. Fig. 3 shows the plasticity

TABLE-3
EXPERIMENTAL DATA OF SOME IMPORTANT PROPERTIES OF THE CERAMIC BODIES MADE FROM ROCK RESIDUE

S. No	Sample code	% of rock residue addition	Plasticity (%)	Dry density (g/cm³)	Fired density (g/cm³) (800 °C)	Compressive strength (Kg/cm²)		Drying shrinkage (%)	Firing shrinkage (%) (800 °C)
						Green strength	Fired strength (800 °C)		
1	RW0	0	26.75	1.781	1.632	27.42	55.67	1.370	1.67
2	RW10	10	25.08	1.888	1.803	28.92	60.70	2.631	2.60
3	RW20	20	22.68	1.975	1.806	31.42	75.23	2.803	2.84
4	RW30	30	20.23	2.025	1.875	33.12	83.64	3.289	2.98
5	RW40	40	18.38	2.184	1.907	35.42	108.26	3.570	3.21
6	RW50	50	16.26	2.349	1.991	37.28	125.72	4.160	3.94

of ceramic bodies as function of the rock residue addition. The plasticity of a normal ceramic body is 26.75 %, When the rock residue content in the mixture varied from 10 to 50 %, the plasticity value decreases from 25.08 to 16.26 %, respectively.

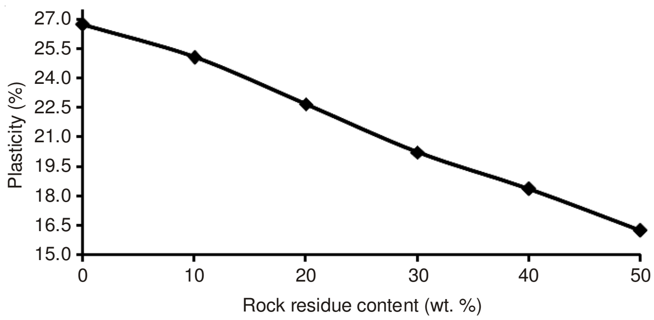

Fig. 3. Plasticity of ceramic bodies as a function of the rock residue addition

Plasticity is an important parameter for the production of ceramic material. Because, ceramic industry mainly uses extrusion shaping and therefore insufficient plasticity creates failures and heterogeneities in clay body and this cause lower mechanical properties[7, 21].

The plasticity has an important technological application, since it indicates the minimum percentage of moisture content necessary to reach a plastic condition[22]. With a high plasticity, there will be more difficulty in drying the samples and causing the appearance of dimension problem or even cracks. It was also observed that the residue addition caused no difficulties during the mixing and extrusion process.

Fig. 3 shows that the percentage of plasticity of ceramic body specimens decreases with increase of the rock residue mixture in the ceramic samples.

Drying density: The density affects a number of properties of ceramic materials but probably the most important effect is on its strength[9,23]. Fig. 4 shows the drying density of ceramic bodies as a function of the rock residue addition.

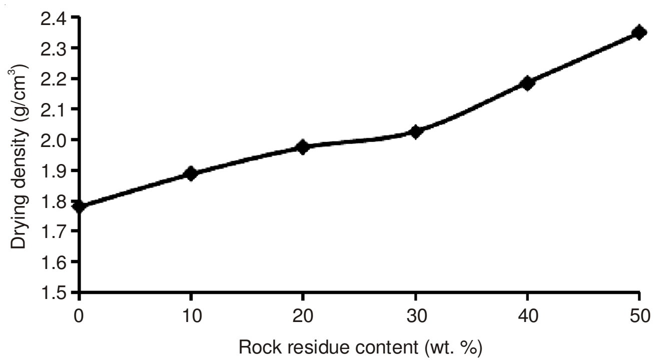

Fig. 4. Dry density of ceramic bodies as a function of the rock residue addition

In the present investigation, the drying density of a normal reference ceramic body RW0 is 1.781 (g/cm^3) and the values of drying density of ceramic bodies from RW10 to RW50 vary from 1.888 to 2.349 (g/cm^3). From the Fig. 4, the drying density of ceramic bodies increases with the increase of the rock residue mix in the samples. Therefore, the dry density of the ceramic body is directly proportional to the quantity of rock residue added in mixture.

Firing density: The firing temperature can also affect the bulk density of the ceramic or building material[9,24,25]. Fig. 5 shows the fired density of ceramic bodies as a function of the rock residue addition. In the present investigation, the firing density of a normal reference ceramic body RW0 is 1.632 (g/cm^3) and the values of firing density of ceramic bodies from RW10 to RW50 vary from 1.803 to 1.991 (g/cm^3). As shown in Fig. 5, the firing density increases with increasing residue addition.

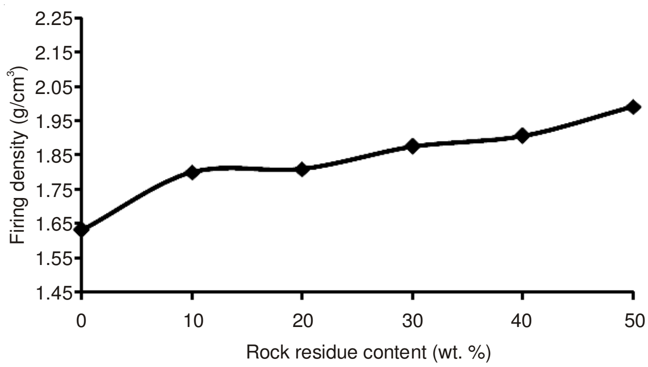

Fig. 5. Fired density of ceramic bodies as a function of the rock residue addition

In the present study, comparing the firing density with the drying density of compositions, it is observed that fired specimens present lower density values. This is mainly due to elimination of the kaolinite constituent water that takes place during the firing stages[26,27]. The organic matter combustion as well as the elimination of water from aluminum and iron hydroxides also contributes to the weight loss[26,27].

Green strength: The green strength of the unfired specimens was examined in terms of the compressive strength.

Fig. 6 shows the compressive strength of the ceramic bodies as a function of the rock residue addition. The values of compressive strength of ceramic bodies with rock residue addition 0 % to 50 % vary from 27.42 to 37.28 (kg/cm^2), where the highest value is displayed for the RW50 specimen.

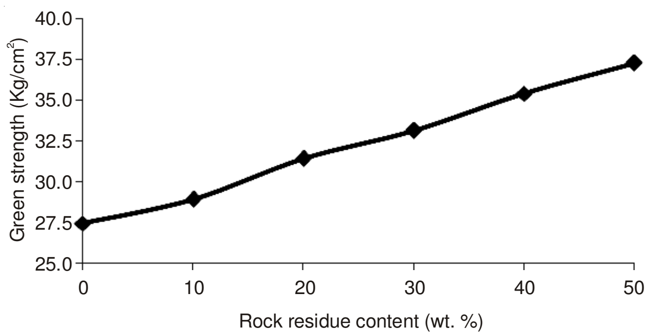

Fig. 6. Green strength of ceramic bodies as a function of the rock residue addition

Strength is the ability of a material to resist failure under the action of stresses caused by a load. The study of this property of material is the concern of a special science *i.e.*, the strength of materials[28]. Strength test is the most important test for assuring the engineering quality of a ceramic material.

Fig. 6 indicates that compressive strength value of the ceramic samples increases with increasing rock residue addition. Therefore, strength of the samples is dependent on the amount of residue in the ceramic samples.

Fired strength: The fired strength of the fired specimens was examined in terms of the compressive strength.

Compressive strength test is very important in view of mechanical behaviour measurement technique for ceramic production[9,29]. Generally, compressive strength decreases with increasing porosity but strength is also influenced by clay composition and firing[30].

In the present study, the results of compressive strength testing of the ceramic bodies made from both clay and rock residue mixtures are shown in Fig. 7. When the specimen is heated at 800 ºC, the compressive strength of the body gradually increased.

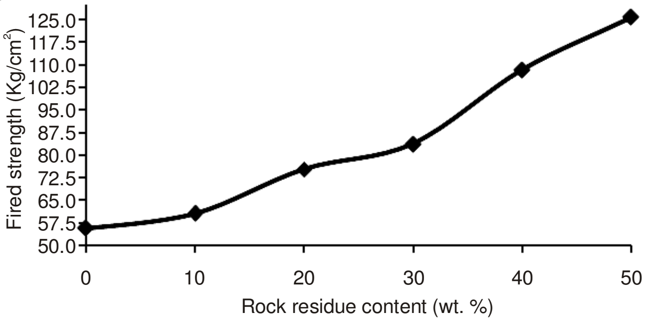

Fig. 7. Fired strength of ceramic bodies as a function of the rock residue addition

The compressive strength is a mechanical property used in brick specifications, which has assumed great importance for two reasons. Firstly with a higher compressive strength, other properties like flexure, resistance to abrasion, *etc*., also improve. Secondly are relatively difficult to evaluate, the compressive strength is easy to determine[31].

The results indicate that the strength is greatly dependent on amount of residue in the ceramic body and the firing temperature. The compressive strength values of all fired samples are increased by increasing the percentage of residue additives. Mix with 50 % rock residue achieved maximum compressive strength value at 800 ºC (sintering temperature).

Drying shrinkage: Fig. 8 shows the drying shrinkage of ceramic bodies as a function of the rock residue addition. When the rock residue content in the mixture varied from 0 to 50 %, the drying shrinkage changes from -1.370 to 4.16 %. The quality of building or ceramic materials can be further assured according to the degree of shrinkage[25,28]. High shrinkage causes destruction of ceramic material both firing and drying stages of production and less linear shrinkage is a factor that may contribute to reduce the risk of appearance of cracks and dimensional defects in ceramic samples[32]. Normally a good quality product exhibits shrinkage[25,28] below 8 %. As a result, there is linear increase in the drying shrinkage as the amount of rock residue in the mixture increases.

Firing shrinkage: The firing temperature is another parameter affecting the degree of shrinkage[24,25].

Fig. 9 shows the firing shrinkage of the ceramic bodies as a function of rock residue addition. The firing shrinkage of a

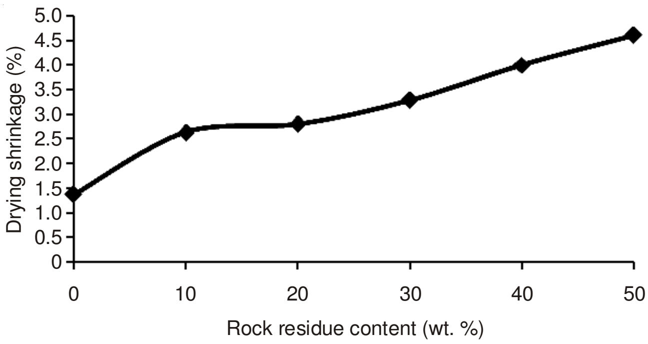

Fig. 8. Drying shrinkage of ceramic bodies as a function of the rock residue addition

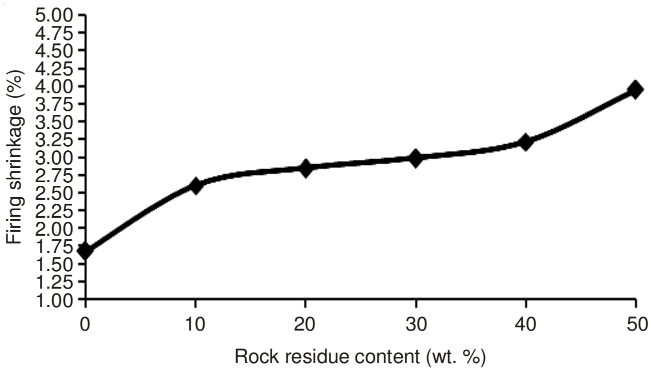

Fig. 9. Firing shrinkage of ceramic bodies as a function of the rock residue addition

normal ceramic body is -1.67 % after heating to temperature of 800 ºC. When the rock residue content in the mixture varied from 0 to 50 %, the fired body shrinkage changes from - 2.6 to 3.94 % with respect to heating temperature of 800 ºC, respectively. A linear relationship between the firing shrinkage and the rock residue proportion was observed for 10 to 50 % residue added.

The linear shrinkage indicates the degree of densification during sintering and is very important for the dimensional control of the finished ceramic product[33].

Appearance and colour: Drying is the most important stage of the ceramic manufacturing process. Small cracks may develop during drying, causing a failure during firing[7,9]. The firing colour is also an important criteria for the classification of a clay bearing material. The chemical and mineralogical compositions of the ceramic raw materials affect the firing colour to a certain extent[34].

In the present study, after drying the samples (RW0-RW50), no defects were observed. Cracks, bloating and other noticeable defects were not observed after firing. The sample RW60 is rejected in the present study due to crack forming and also rough surface created.

The colour of some ceramic bodies was changed from white (before firing) to pale red (after firing) and light grey (before firing) to dark red (before firing). After firing, all the samples presented the characteristic homogenous ceramic product colour, indicating the uniformity of firing. The visual observation for appearance and colour of the ceramic bodies are given in Table-4. Appearance and colours of ceramic bodies were acceptable.

TABLE-4
VISUAL OBSERVATIONS OF APPEARANCE AND COLOUR OF CERAMIC BODIES MADE FROM ROCK RESIDUE

S. No	Sample code	% of rock residue	Appearance	Colour	
				Before fired	After fired
1	RW0	0	No defect and smooth	White	Pale red
2	RW10	10	No defect and smooth	White	Pale red
3	RW20	20	No defect and smooth	White	Pale red
4	RW30	30	No defect and smooth	Light grey	Dark red
5	RW40	40	No defect and smooth	Light grey	Dark red
6	RW50	50	No defect and smooth	Light grey	Dark red
7	*RW60	60	Crack and Rough	Light grey	-

*RW60 sample is rejected in the study

Conclusion

In this study we demonstrated that it is possible to utilize rock residue as alternative raw material resource for the production of the ceramic product. On the basis of the results reported in the present investigation, following conclusions can be drawn:

• It is obvious from the above analysis that the raw materials is made of major mineral-quartz, clay minerals, accessory minerals-calcite/aragonite (carbonate), albite/orthoclase (feldspar) and iron mineral (hematite). These minerals have greater influence on clay mineral structure. Our results show the usefulness of XRD analysis of raw materials.

• The results of the chemical analysis show that the raw materials contain Al_2O_3, SiO_2 and Fe_2O_3 as a major constituents making them suitable as ceramic materials.

• The high plasticity value of the ceramic samples can cause difficulty of drying or firing, causing the appearance of dimensional problem. In the present study, the plasticity of ceramic bodies decreases with increase of the rock residue addition in the samples.

• A linear relationship between the shrinkage and the rock residue proportion is observed for 10 to 50 % rock residue added.

• Density of the ceramic bodies was directly proportional to the quantity of rock residue added in the mixture.

• The maximum green strength and fired strength were observed for specimen (RW50) containing a 50 % rock residue when compared with control specimen (RW0).

• Appearance of ceramic bodies is good (no defect and smooth surface) and colours of the ceramic bodies are acceptable in all the samples.

All the experimental data shows that the addition of the rock residue improves the physical and mechanical properties. It is concluded, that all observation indicates that rock residue is compatible ingredient, so that rock residue can be used as a clay substitute.

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