Trends in

Applied Sciences Research

ISSN 1819-3579

Academic
Journals Inc.

www.academicjournals.com

Trends in Applied Sciences Research 9 (10): 574-587, 2014
ISSN 1819-3579 / DOI: 10.3923/tasr.2014.574.587

Optimization of Ingredients for Clay Block Manufacture: Unfired Characteristics

[1]R. Maharaj, [2]C. Maharaj, [3]D. White, [4]C. Penjilia and [5]S. Ramlagan

[1]University of Trinidad and Tobago, Point Lisas Industrial Estate, Point Lisas, Trinidad and Tobago, West Indies

[2]Department of Mechanical and Manufacturing Engineering, University of the West Indies, St. Augustine, Trinidad and Tobago, West Indies

[3]University of Trinidad and Tobago, O'Meara Industrial Estate, O'Meara, Arima, Trinidad and Tobago, West Indies

[4]ABEL Building Solutions, Clay Division, Depot Road, Longdenville, Chaguanas, Trinidad

[5]Asea Brown Boveri, Ave. Balboa, EdificioBBVA, Piso 14, P.O. Box 0816-01349, Panama

Corresponding Author: R. Maharaj, University of Trinidad and Tobago, Point Lisas Industrial Estate, Point Lisas, Trinidad and Tobago, West Indies Tel/Fax: 1 868 642 8888

ABSTRACT

The purpose of this study was to optimize the relative proportions of the input ingredients of clay, sand and water utilized in a formulation by a leading hollow core clay brick manufacturer in Trinidad as a possible solution for the incidence of production of defective products. The influence of incremental changes in the proportions of ingredients on important physical characteristics of clay bricks such as compressive strength, extruded surface finish, flexural strength, shrinkage, density and extrusion pressure and the rheological properties of dynamic modulus (G^*) and phase angle (δ) were investigated before the firing stage. The optimum formulation was found to be the addition of an additional 5% clay content to the manufacturer's original mixture utilizing with a water/clay ratio of 16% composition. Further research is recommended on optimization of the firing process by testing fired properties with these optimum ingredients.

Key words: Unfired properties, kaolinite, extrusion, compressive strength, flexural strength, scanning electron microscopy, rheology

INTRODUCTION

A brick or block can be described as a kneaded unit of clay-bearing soil, containing sand and lime or cement, which is usually fire hardened or air dried and used in construction. The fired brick has been found to be used as early as 2900 BC in early Indus Valley cities (Possehl, 2002) and today the fired brick is still the most common type used as they are the long lasting and strongest building material. By weight, bricks basically consist of Silica (sand) 50-60%, Alumina (clay) 20-30%, Lime 2-5%, Iron oxide $\leq$7% and Magnesia less than 1% (Punmia and Jain, 2003). The main manufacturing processes are shown in Fig. 1.

In Trinidad, there exist many naturally occurring clay deposits whose ceramic producing potential has been previously investigated (Knight *et al.*, 1996). It was found that the clays can be distinguished by color (varying from grayish to reddish brown) and particle size with unfired flexural strengths ranging from 0.1-0.7 MPa (Knight *et al.*, 1996). The major difference in these

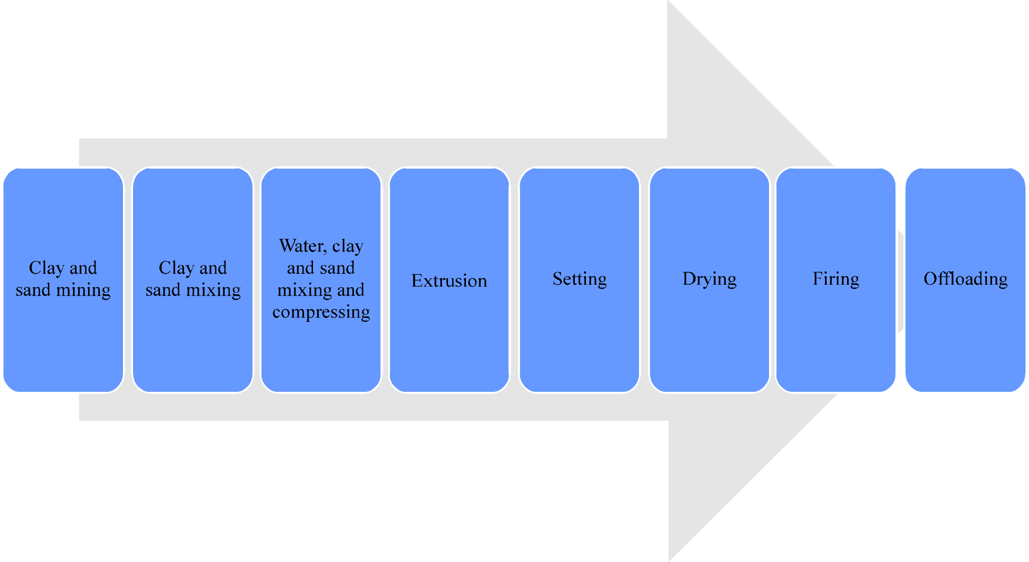

Fig. 1: Main processes in the manufacture of hollow clay blocks

clays arises from the different weight percentages of Aluminum oxide and Iron (III) oxide. Trinidad clays basically contain Kaolinite of chemical composition $Al_2Si_2O_5(OH)_4$, as its main mineral. Trinidad and Tobago is one of the largest producers of clay blocks in the region uses the Grey Gumbo and Red Mottle types in its manufacturing process (Ramdath, 2012). The sand used in the manufacturing process is predominantly silicon dioxide with a trace amount of aluminum oxide.

The relative proportions of the ingredients used in the mixing formulations determine the behavior and the properties of the ceramic end product as the inherent mineral and chemical composition influence their plasticity and drying and firing characteristics (Onal and Sarykaya, 2009). The composition and the homogeneity of the ceramic mixture have a profound effect on the rheology and properties of the final fired clay body (Aras, 2004).

For clay-shale blocks manufactured in the Canadian Atlantic provinces, compressive strength values of the final products varied for different raw materials (Davison, 1963). An extruded kaolinitic clay, silt and sand mixture produced samples that had unfired compressive strengths between 1 and 4 MPa, with compressive strength decreasing as moisture content increased (Maskell *et al.*, 2013). A formulation containing Kaolinitic clay extracted from a Trinidad mud volcano effluent with some additional quartz, mica and potash feldspar had unfired compressive strength of 1.9 MPa and mean linear shrinkage of 5.7% (Knight *et al.*, 2000). A compressive strength of 2.5 MPa for a natural bentonite clay from Burgsvik, Sweden was obtained (Pusch, 2006). Particle size and shape of an unfired clay block affects its strength and water permeability (Johari *et al.*, 2010). The effect of composition on the ultimate physical characteristics of the ceramic can be associated with the desaglomeration and dispersion of the particles within the ceramic system. It is critical in the industrial ceramic processes to obtain homogenous and stable systems by controlling the dispersion of the particles which controls its structure. This characterization can be accomplished by using rheology (Ayadi *et al.*, 2013). Three clays namely Illite (I), Montmorillonite (M) and Kaolin (K) were utilized to study the effect of clay composition on the rheological behavior of the ceramic suspensions. It was found that the rheological properties of the ceramic suspensions were mainly governed by the percentage of Montmorillonite (Ayadi *et al.*, 2013). They also demonstrated that in order to obtain low viscosities (0.20-0.40 Pa sec), as required by the ceramic industry, the clay mixture should not contain more than 16% of Montmorillonite. Although the technique of rheology has been used to characterize concentrated suspensions, studies involving the influence of mineralogical changes on rheological properties has been limited (Andreola *et al.*, 2004). Furthermore a literature survey on the use of rheology on Trinidad clays and ceramics properties has provided no results.

Within recent times a principal producer within the brick manufacturing industry has been experiencing major defects such as cracking and excessive shrinkage in the final brick product. Such defects if not mitigated, can have significant negative impacts on the economic bottom line and reputation of the manufacturer. One of the possible root causes for these defects has been associated with non-optimization of block ingredients. Since the relationship between the composition and the physical, performance and rheological properties of clay based ceramic materials is well known and documented (Andreola *et al.*, 2004). The objective of this study is to optimize the composition of the ingredients (clay, sand and water) used by the manufacturer of the bricks by measuring its effect on physical characteristics such as compressive strength, extruded surface finish, flexural strength, shrinkage, density, extrusion pressure and rheology. This objective will be accomplished by measuring the effect of incremental deviations of the proportion of clay, sand and water to the original formulation on the outlined physical characteristics.

MATERIALS AND METHODS

Specimen preparation: Samples of clay (C), sand (S) and the original formulation (O) of a mixture of clay and sand, obtained from the processing plant, were oven dried at a temperature of 60°C until constant mass was achieved. The clay and the sand/clay mixture were manually pulverized to <1 mm particles. Twenty different formulations of clay, sand and water (W) were produced in five series based on variations to the clay:sand ratio of O. Each series utilized four moisture contents by weight as shown in Table 1.

The dried materials of each series were thoroughly mixed, then combined with the requisite quantity of water, hand kneaded and consolidated in a mixing bowl, to best simulate the mechanical processes in the plant. The consolidated material was then transferred to the extruder and hydraulically compressed to produce continuous lengths of 12 mm by 12 mm cross-section extruded material. These were cut into ten 25 mm specimens for compressive strength testing and ten 50 mm lengths for flexural strength testing. Physical properties evaluations were conducted on the specimens before the mechanical tests were conducted. The specimens were then labeled and oven dried to constant mass at 60°C. It must be noted that full sized block specimens of varying compositions could not be obtained as this would have affected the existing plant operations. Therefore, a modification was required to ASTM C67-11: Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile (ASTM International, 2011).

Table 1: Formulations of Clay (C), Sand (S), and Water (W) mixture

Series 1	Series 2	Series 3	Series 4	Series 5
O+10% S+17.3% W	O+5% S+17.3% W	O+17.3% W	O+5% C+17.3% W	O+10% C+17.3% W
O+10% S+16% W	O+5% S+16% W	O+16% W	O+5% C+16% W	O+10% C+16% W
O+10% S+15% W	O+5% S+15% W	O+15% W	O+5% C+15% W	O+10% C+15% W
O+10% S+14% W	O+5% S+14% W	O+14% W	O+5% C+14% W	O+10% C+14% W

Physical properties

Linear Shrinkage: The percentage linear shrinkage, S_L of each formulation was determined from the average linear shrinkage calculated across ten specimens. Measurement of the length of each specimen was obtained from freshly prepared specimens and repeated on the oven dried specimens. The linear shrinkage of an individual specimen was calculated using the Eq. 1:

$$S_L = \frac{L_d - L_w}{L_w} \times 100 \tag{1}$$

where, L_w and L_d are the lengths before and after drying, respectively.

Bulk density: The mass of the dried specimen was determined using a top loading balance. The volume was calculated from the physical dimensions of the specimen.

Qualitative surface finish: The extruded samples were visually examined for the degree of surface finish. Samples were observed and evaluated as very smooth, smooth, rough and very rough based on the sample set containing at least 70% similar type.

Mechanical properties

Compressive strength: The compressive strength tests were conducted using a Hounsfield model H50KStensometer. The specific compressive strength was calculated dividing by the bulk density.

Flexural strength: The flexural strength, termed the Modulus of Rupture (MOR), was found by the three point bend method as described in ASTM C293-02 using Hounsfield model H50KStensometer.

Extrusion pressure: The extrusion was accomplished using a laboratory extruder and hydraulically compressed using the tensometer which recorded values of force required for the flow of extrudate.

Rheological study: The rheological properties of the clay blends were determined using an ATS RheoSystems Dynamic Shear Rheometer (Viscoanalyzer DSR) performed under the strain control mode. The test geometry used was the plate-plate configuration (diameters 25 mm) with a 1 mm gap and the measurements were conducted at room temperature and a frequency range of 0-16 Hz that corresponds to a shear stress varying between 0 and 580 Pa. The data obtained at different oscillating shear frequencies and temperatures were store in the computer and the results obtained were analyzed using the Viscoanalyzer software. The value of the rheological parameters associated with the complex modulus and phase angle were calculated at the different oscillating frequencies and temperatures using the instrument's software.

Microstructural examination and elemental analysis: Microstructural examination of the fracture surface of randomly selected Series and respective water addition samples were conducted using Scanning Electron Microscopy (SEM). Total 5 random samples were chosen for analysis. Gold sputtering of the fracture surfaces was used to avoid charge build-up on the samples during analysis. Secondary electrons were employed at an energy level of 30 kV. The fracture surfaces of the samples were examined after being subjected to flexural strength tests.

Elemental analysis was performed using Energy-dispersive X-ray spectroscopy (EDS). Elemental maps were produced at the location shown in the SEM images.

The primary purpose of the SEM and EDS analysis was to determine if there was a thorough mix of the clay and sand for this study.

RESULTS AND DISCUSSION

Figure 2 shows a graph of linear shrinkage versus increasing clay content. There was very little variation in shrinkage at and above Series 2, for the 14% water composition. Most values for shrinkage of all Series were less than the 5.7% mean value found by Knight *et al.* (2000) for the Trinidad mud volcano effluent.

Table 2 shows the associated standard deviation values for the Fig. 2 data points. The 14% water composition produced the lowest average value of standard deviation values among the different water compositions. Correspondingly, Series 3 produced the lowest average standard deviation of all the series.

Figure 3 is a graph of bulk density versus increasing clay content. The 14% water composition had the least variation in density as the clay content increased, while the 16% water composition produced the widest variation.

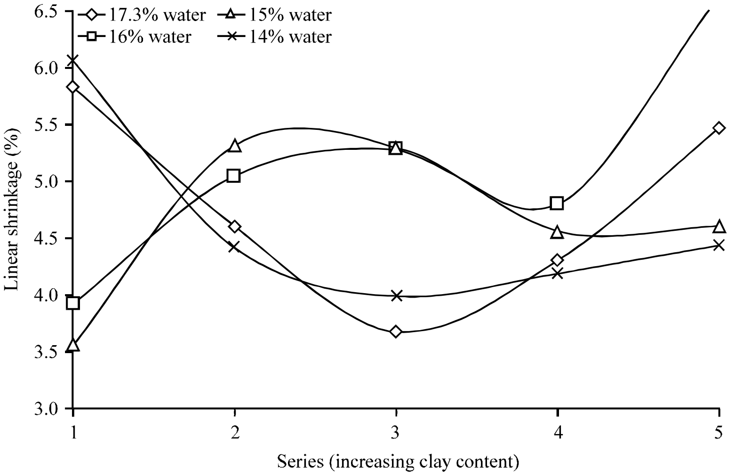

Fig. 2: Linear shrinkage versus increasing clay content

Table 2: Linear shrinkage versus standard deviation values

	Linear shrinkage standard deviation (%)				
Series	17.3% water	16% water	15% water	14% water	Average
1	1.17	1.63	1.30	2.10	1.55
2	1.50	2.24	1.43	1.05	1.56
3	1.81	1.58	1.08	0.69	1.29
4	1.67	1.70	1.89	1.47	1.68
5	1.92	4.95	1.44	0.83	2.29
Average	1.61	2.42	1.43	1.23	

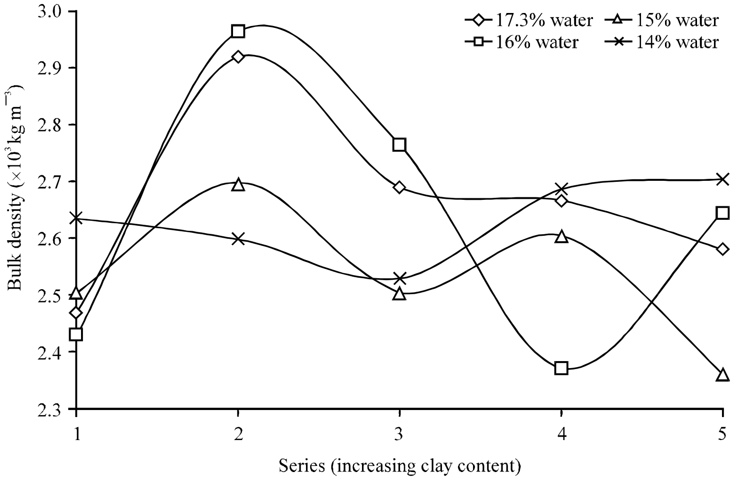

Fig. 3: Bulk density versus increasing clay content

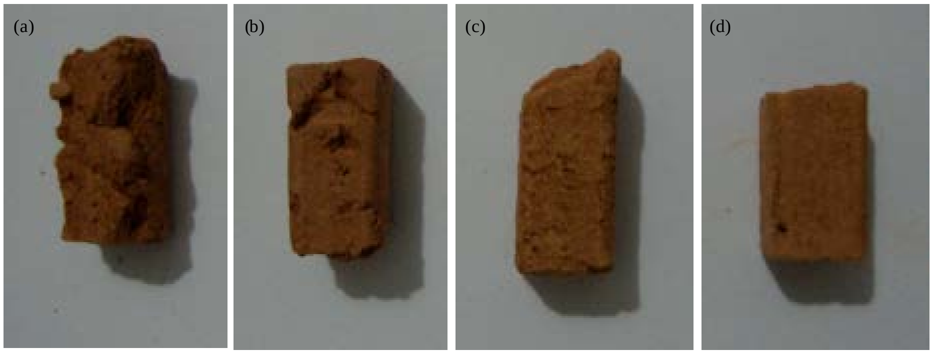

Fig. 4(a-d): Visual examination grading of quality extruded surface. Sample is (a) Very rough (b) Rough (c) Smooth and (d) Very smooth

Table 3: Qualitative surface finish of extruded specimens

	Qualitative surface finish			
Series	14% water	17.3% water	16% water	15% water
1	Rough	Rough	Very rough	Smooth
2	Very smooth	Very smooth	Smooth	Rough
3	Very smooth	Smooth	Rough	Very rough
4	Smooth	Rough	Rough	Smooth
5	Rough	Rough	Smooth	Rough

Rough surfaces in the extrudate are sites of possible stress concentrations that can lead to fracture originating at these sites. The qualitative rubric used to visually examine the extrudate is shown in Fig. 4.

Table 3 documented these observations. A distinct pattern is observed for Series 3 clay content as the smoothness increased with added water content. It is noteworthy that overall Series 2 had the smoothest surface of all the series.

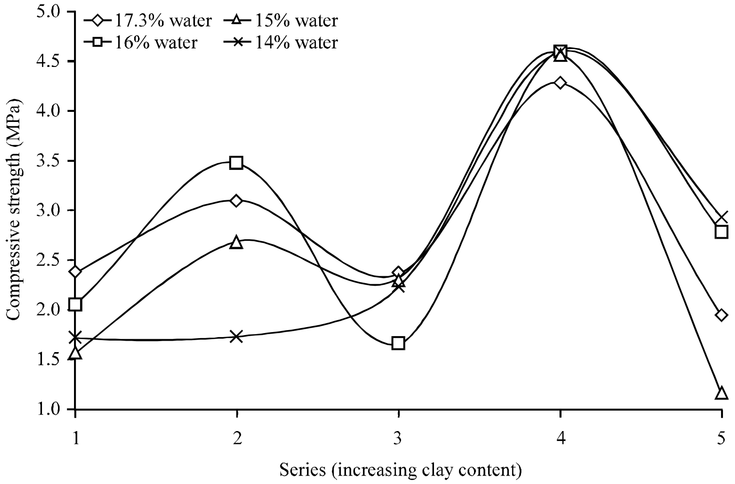

Fig. 5: Compressive strength versus increasing clay content

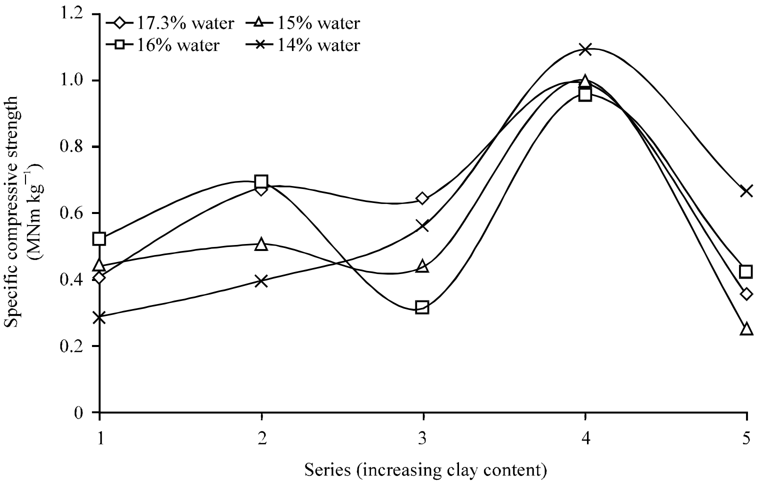

Fig. 6: Specific compressive strength versus increasing clay content

A graph of compressive strength versus increasing clay content is presented in Fig. 5. All curves produced maximum compressive strength for Series 4 formulation. The values of compressive strength are similar to the findings of Maskell *et al.* (2013) who obtained values between 1 and 4 MPa for clay that also contained Kaolinite as its main mineral. However, there was no firm relationship between compressive strength and moisture content.

Figure 6 is a graph of specific compressive strength versus increasing clay content. No major shift in the trends was observed in comparison to Fig. 5. However, up to Series 4 in Fig. 6, there was a more gradual increase in specific compressive strength.

Standard deviation values for the Fig. 5 data points are documented in Table 4. The 16% water composition curve produced the lowest average value of standard deviation amongst the water composition categories. Correspondingly, Series 1 produced the lowest average value of standard deviation within the different series.

Flexural strength versus increasing clay content is documented in Fig. 7. The surface finish of the extruded samples is related to its associated flexural strength. This association is demonstrated

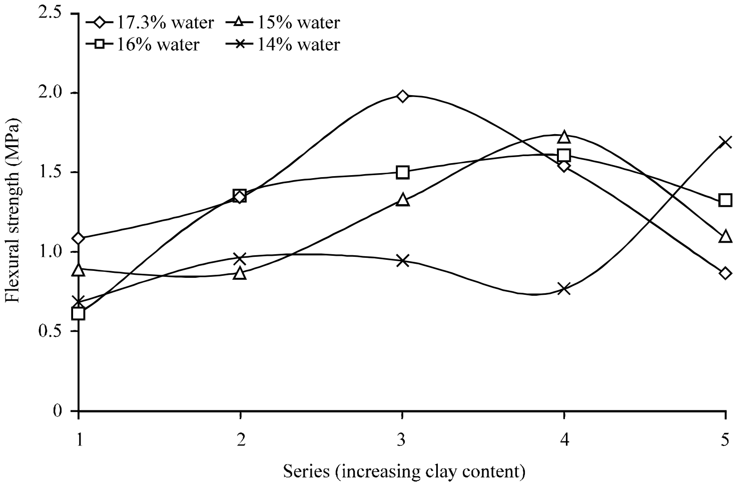

Fig. 7: Flexural strength versus increasing clay content

Table 4: Compressive strength standard deviation values

	Compressive strength standard deviation (MPa)				
Series	17.3% water	16% water	15% water	14% water	Average
1	0.68	0.63	0.79	0.29	0.60
2	1.09	0.99	0.88	0.75	0.93
3	1.01	0.38	0.94	0.73	0.77
4	1.57	1.51	1.85	2.25	1.79
5	0.90	0.81	0.33	1.07	0.78
Average	1.05	0.86	0.96	1.02	

Table 5: Flexural strength standard deviation values

	Flexural strength standard deviation (MPa)				
Series	17.3% water	16% water	15% water	14% water	Average
1	0.27	0.27	0.28	0.25	0.27
2	0.50	0.43	0.28	0.40	0.41
3	0.40	0.57	0.52	0.34	0.46
4	0.58	0.49	1.46	0.32	0.71
5	0.37	0.71	0.25	0.32	0.41
Average	0.42	0.49	0.56	0.33	

with the flexural strength values for Series 3 in Table 3, where a smoother surface results in a higher flexural strength. However, no clear pattern could be discerned for the other Series. The 14% water composition curve had the most consistent values for Series 4 clay content and below. The 16% water composition curve produced consistent and relatively high values of flexural strength for Series 2 clay content and above. Considering all Series, higher ranges of flexural strength were obtained compared to the results of Knight (1996) who reported values ranging from 0.1-0.7 MPa for Trinidad clays.

Standard deviation values for Fig. 7 data points are documented in Table 5. Table 5 reveals that 14% water composition had the lowest average standard deviation value among the different water compositions. Correspondingly, Series 1 produced the lowest average standard deviation of all the series.

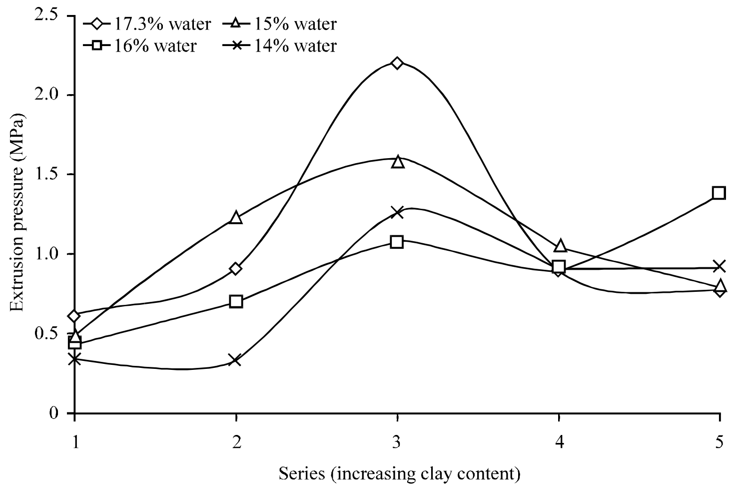

Fig. 8: Extrusion pressure versus increasing clay content

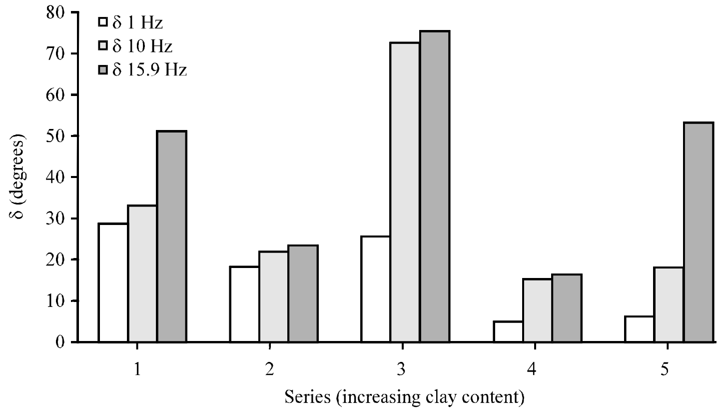

Fig. 9: Effect of mineral additives on the phase angle, δ of various blends at different loading frequencies

Figure 8 shows graphs of extrusion pressure versus increasing clay content for different moisture additions. The 14% water composition produced the lowest average value of extrusion pressure. The maximum extrusion pressure occurred for Series 3 clay content at all water compositions except the 16% water composition. The minimum extrusion pressure occurred at Series 1 clay content for all water compositions.

The use of the dynamic (oscillatory) testing technique has previously been used by Ayadi *et al.* (2013) to examine the effect of a clay's mineralogy on its rheological properties. At a given combination of time and temperature, the viscoelastic behavior of a clay based material can be characterized by the total resistance to deformation (G*) or its stiffness and the relative distribution of that resistance between an elastic, in-phase component and a viscous, out-of-phase component represented by (δ) or its elasticity. A 0° value δ represents a completely elastic solid and as the value approaches complete viscous liquid behavior, the δ values tend to 90°. In the shear mode, the dynamic modulus (G*) and phase angle (δ) were measured. The in-phase, elastic component can be related to energy stored in a sample for every loading cycle, while the out-of-phase, viscous component represents energy lost per cycle in permanent flow. Figure 9 and 10 demonstrate the effect of the addition of the mineral additives on the rheological characteristics of δ and G* of the ceramic blends, respectively.

The results shown in Fig. 9 clearly show that the incremental additions of both the clay and the sand resulted in an overall decrease in the in the phase angle, δ of the blend. The value of the phase angle, δ of the blend containing 5% clay additive was the lowest closely followed by 5% sand. For the 5% clay additive, all the measured loading frequencies the values of δ decreased by a factor of approximately 5 times compared to the control. The magnitude of δ were <20° in all cases and demonstrated that the 5% clay blend was tending to behave like an elastic solid.

Figure 10 shows that the effect of the addition of the clay and sand mineral additives resulted in a general increases in the values of the complex moduli reflective of stiffening of the blends due to the additives. The chemical composition of the ceramic blends determined the rate of changes of both rheological parameters. The blend containing 5 and 10% clay additives showed superior rheological characteristics as it had a relatively higher value of G* for all frequencies examined.

In terms of the W/C ratio, the results in Fig. 11 demonstrate the influence of water content on G* (stiffness) of the blend containing 5% clay. A maximum value of G* was obtained at a W/C ratio of 16% indicating that the sample was stiffest at this concentration of water.

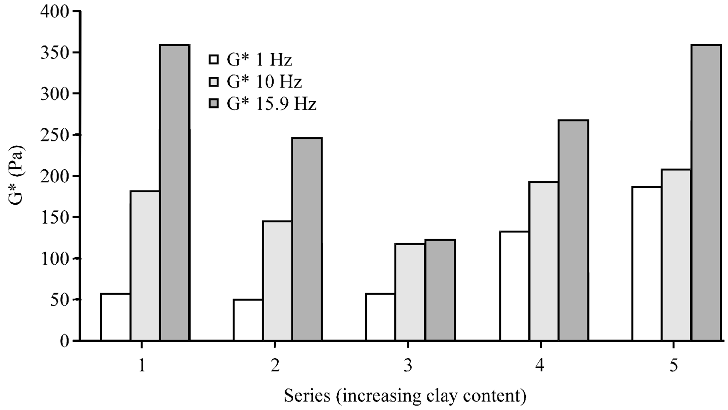

Fig. 10: Effect of mineral additives on the complex modulus, G* of various blends at different loading frequencies

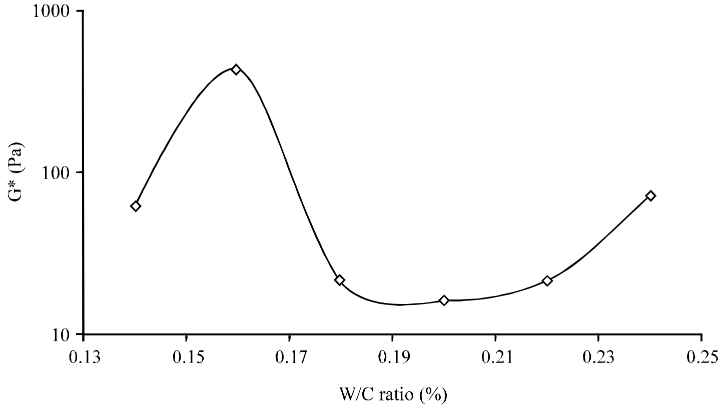

Fig. 11: Effect of W/C ratio on the complex modulus, G* of various blends at different loading frequencies for the blend containing 5% clay additive (Series 4)

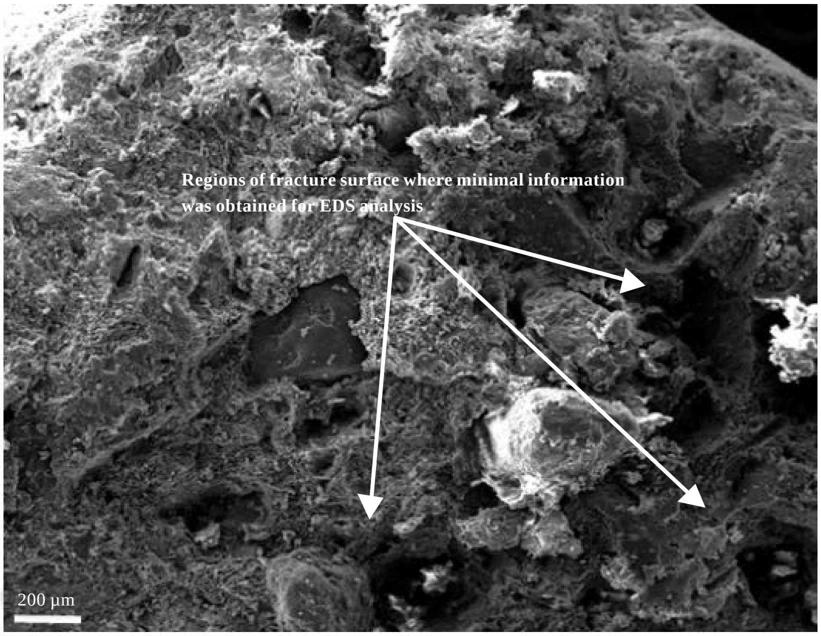

Fig. 12: SEM image showing the fracture surface of series 4 containing the additional 17.3% water content

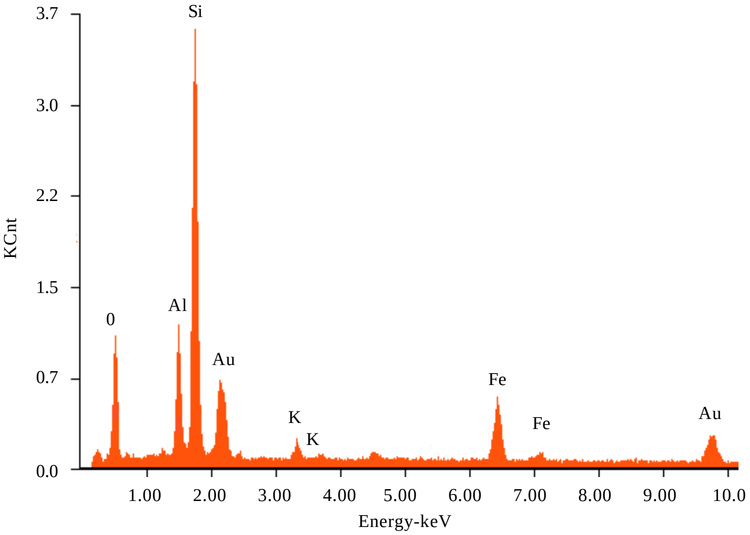

Fig. 13: Elemental composition analysis with count (in thousands) versus X-ray dispersive energy in keV

Figure 12 is a SEM image showing the fracture surface of Series 4 composition which contained the additional 17.3% water content.

Figure 13 shows the elements that were obtained for Fig. 12 using EDS analysis. The presence of gold came from sputtering of the samples with this material. Ramdath (2012) documented Iron (III) oxide and Potassium oxide in the clay type used in this study and the EDS analysis confirms this.

An attempt to determine any variations in material composition due to improper mixing are shown in the elemental map (Fig. 13) of Fig. 12. It was observed that the gold along with other elements were not detected in some regions of the sample (Fig. 12) due to the representative X-rays not reaching the EDS detector. The most likely reason for lack of information in these regions is due to the scattering of the X-rays. These regions were therefore not considered when comparing elements.

A key distinction in material composition difference is Aluminium. Aluminium would be present in the clay but would be in trace quantities in the sand. However, no variation in Aluminium was observed in Fig. 14. Similar results were obtained for the microstructural and elemental analysis of the other Series and water compositions that were analyzed. An inference can be drawn to show that there was thorough mixing of the clay and sand.

The ability to obtain the desired properties of good compressive and flexural strength along with consistency of product quality is of primary importance to clay production. Consistency of strength properties is especially important as slight chemical composition variations in the pit raw material will occur, both on the macroscopic and microscopic scales. It would be undesirable for these slight variations to cause major changes in the properties of the final product. There must be consistency with respect to shrinkage and density. Too much variation within the block product would lead to cracking from excessive internal stresses and therefore a defected product. Variations from improper mixing in this study could be excluded in accordance with Fig. 14 observations.

A weighting system was used for the criteria assessed because of the wide variety of characteristics and properties that are desired in an optimum formulation. This is documented in Table 6. The best in series and water composition were assessed with a high compressive stress having the highest weighting and a low extrusion pressure having the lowest weighting. However, low extrusion pressures are advantageous in the block manufacturing process as this reduces energy input required by the plant.

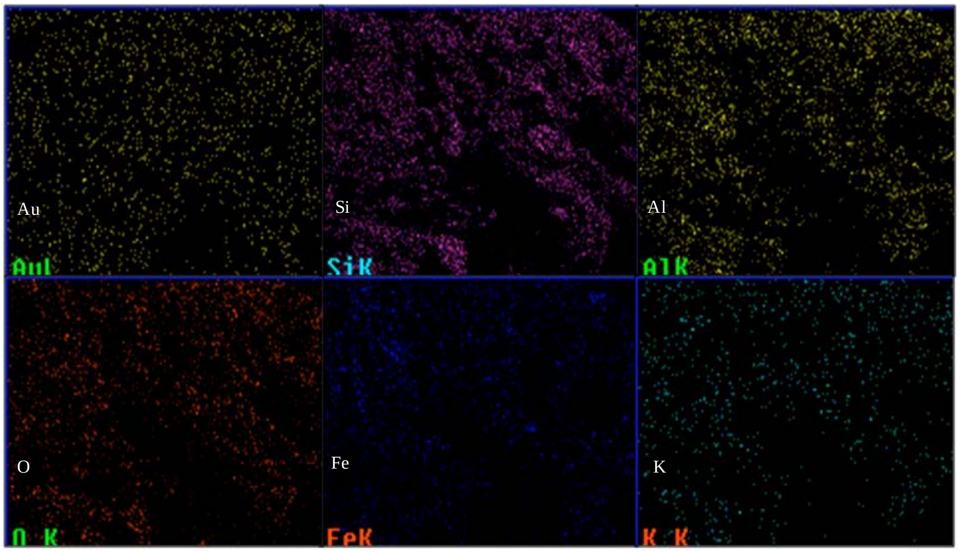

Fig. 14: Elemental map of Fig. 12

Table 6: Criteria and weightings used for determining optimum series and water composition

Criteria	Weighting	Best in series	Best in water composition
High compressive strength	0.25	Series 4	16% water
Lowest deviation in compressive strength values	0.15	Series 1	16% water
Smooth surface finish	0.20	Series 2	17.3% water
High flexural strength	0.10	Series 3	16% water
Lowest phase angle from rheology	0.10	Series 4	16% water
Lowest deviation in flexural strength values	0.05	Series 1	14% water
Low shrinkage	0.05	Series 4	14% water
Low density	0.05	Series 1	14% water
Low extrusion pressure	0.05	Series 1	14% water

Table 7: Scorecard for series and water composition

Series and water composition	Score
Series 1	0.30
Series 2	0.20
Series 3	0.10
Series 4	0.40
Series 5	0.00
14% water	0.20
15% water	0.00
16% water	0.60
17.3% water	0.20

Table 7 reveals the associated scores for the series and water composition, where a higher score is associated with a more desirable product. Series 4 (5% additional clay) obtained the highest score with 16% water composition.

CONCLUSION

A weighting system was used for the criteria assessed due to the wide variety of characteristics and properties that are desired in an optimum formulation. It was found that adding a 5% additional clay content along with 16% water composition to the manufacturer's original mixture produced an optimum formulation for all the properties considered. In terms of plant economics, adding 5% more clay will decrease the unit cost by 1.8%. Further research is recommended for optimization of the firing process by testing fired properties with the optimum ingredients.

REFERENCES

ASTM International, 2011. ASTM C67-11, standard test methods for sampling and testing brick and structural clay tile. ASTM International, West Conshohocken, PA., USA. http://www.astm.org/DATABASE.CART/HISTORICAL/C67-11.htm.

Andreola, F., E. Castellini, T. Manfredini and M. Romagnoli, 2004. The role of sodium hexametaphosphate in the dissolution process of kaolinite and kaolin. J. Eur. Ceram. Soc., 24: 2113-2124.

Aras, A., 2004. The change of phase composition in kaolinite-and illite-rich clay-based ceramic bodies. Applied Clay Sci., 24: 257-269.

Ayadi, A.J., J. Soro, A. Kamoun and S. Baklouti, 2013. Study of clay's mineralogy effect on rheological behavior of ceramic suspensions using an experimental design. Int. J. Res. Rev. Applied Sci., 14: 374-384.

Davison, J.I., 1963. Properties of clay bricks manufactured in the Atlantic provinces. National Research Council of Canada, Division of Building Research, Ottawa.

Johari, I., S. Said, B. Hisham, A. Bakar and Z.A. Ahmad, 2010. Effect of the change of firing temperature on microstructure and physical properties of clay bricks from Beruas (Malaysia). Sci. Sintering, 42: 245-254.

Knight, J.C., 1996. Ceramic potential of some Trinidad clays: 2 fired characteristics. Br. Ceram. Trans., 95: 162-168.

Knight, J.C., D.L. White and N. Deosaran, 2000. Ceramic properties of mud volcanoes effluent. West Indian J. Eng., 23: 22-27.

Knight, J.C., L.H. Grierson and A. Hosein, 1996. Ceramic potential of some Trinidad clays: 1 chemistry and mineralogy. Br. Ceram. Trans., 95: 121-124.

Maskell, D., A. Heath and P. Walker, 2013. Laboratory scale testing of extruded earth masonry units. Mater. Des., 45: 359-364.

Onal, M. and Y. Sarýkaya, 2009. Some physicochemical properties of a clay containing smectite and palygorskite. Applied Clay Sci., 44: 161-165.

Possehl, G.L., 2002. The Indus Civilization: A Contemporary Perspective. Rowman Altamira, Walnut Creek, CA., ISBN: 9780759101722, Pages: 276.

Punmia, B.C. and A.K. Jain, 2003. Basic Civil Engineering. Firewall Media, New Delhi, India, ISBN: 9788170084037, Pages: 446.

Pusch, R., 2006. Mechanical Properties of Clays and Clay Minerals. In: Handbook of Clay Science, Bergaya, F., B.K.G. Theng and G. Lagaly (Eds.). Chapter 6, Elsevier Ltd., Amsterdam, The Netherlands, ISBN-13: 9780080441832, pp: 247-260.

Ramdath, H., 2012. Exploitation of mineral resources-Trinidad and Tobago 2011. Ministry of Energy and Energy Affairs, Government of Republic of Trinidad and Tobago.