Ceramic bricks made from municipal solid waste incineration-derived clay and ashes: a quality study

Estudio de calidad de ladrillos cerámicos producidos con mezclas de arcilla y cenizas de incineración de residuos sólidos municipales

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ABSTRACT

This paper presents analytical data from tests on bricks made from different clay/ash mixtures. The ashes used were obtained from a Hoffmann-type brick-making furnace equipped for coal and municipal solid waste co-firing. The bricks were physically and chemically characterised and their quality was determined by porosimetry, water absorption, breaking load and tensile strength tests. Brick loading capacity increased by up to 10% with ash mixtures and decreased at higher percentages, whereas water absorption increased brick loading capacity for all mixtures. This study showed that clay/ash bricks complied with all the quality standards required in Colombia.

Keywords: brick, incineration, co-firing, municipal solid waste, volatile ash, encapsulation.

RESUMEN

Se presentan los resultados de pruebas analíticas realizadas en ladrillos fabricados a partir de diferentes mezclas de arcilla y ceniza. Las cenizas fueron obtenidas de un horno ladrillero tipo Hoffman equipado para coincinerar carbón y residuos sólidos municipales. Las muestras fueron caracterizadas física y químicamente y su calidad se determinó a través de pruebas de porosimetría, absorción y esfuerzo de tensión y de ruptura. La capacidad portante de los ladrillos aumenta con contenidos de ceniza hasta del 10% y decrece en proporciones mayores. La absorción de agua es superior para todas las combinaciones de mezcla. Este estudio reporta que ladrillos elaborados utilizando mezclas arcilla/ceniza cumplen con todos los requisitos de calidad exigidos en Colombia.

Palabras clave: Ladrillos, Incineración, Co-encendido, Residuos Sólidos Municipales, Cenizas Volátiles, Encapsulación.

Introduction

New construction materials are currently being tested and one possibility concerns the partial substitution of clay in bricks by cheaper and less dense materials such as paper mill residue, rice husk, cotton waste, plastic fibres, etc. (Raut, Ralegaonkar and Mandavgane, 2011, pp. 4037-4042; Raut, Ralegaonkar and Mandavgane, 2013, pp. 269-275; Kumar, Kumar et al, 2010, pp. 585-590). Using wet fly ash from municipal solid waste incineration as a replacement agent within bricks may represent major environmental benefits rather than their final disposition in landfills (safer and cheaper) and reduced clay consumption (Abdrakhimov and Abdrakhimova, 2011, pp. 231-234;). Encapsulating bottom

and fly ash produced by co-incineration of MSW in clay used for bricks becomes a feasible solution for the proper disposal of such ash and as a clay substitute material (La Grega, M.D., Buckingham, P.L. and Evans, J.C., 1994; Akhtar, Alam and Akhtar, 2011, pp. 7338-7346)). There is a restriction on clay brick manufacturing in China because of a lack of clay (Lingling, Wei et al, 2005, pp 243-247). The use of these ashes as a replacement material in other processes like cement and concrete block and structure production has been studied, leading to ambiguous outcomes due to high moisture content producing setting problems and poor quality in final pieces (Anastasiadou, Christopoulos et al, 2012, pp. 165-170; Vijayaraghavan, James, and Marithangam, 2009, pp. 227-334). Moreover, similar research carried out in countries such as Germany, England and China has given favourable results (Chih-Huang, Deng-Fong and Pen-chi, 2003, pp. 679-685; Xu, L, Guo W., Wang, T and Yang N., 2005, pp. 243-247; Shakir, Naganathan and Mustapha, 2013, pp.131-138; Pérez-Villarejo, Eliche-Quesada et al., 2012, pp. S349-S354).

The ash used for this study was obtained from the bottom and exhaust gases of a Hoffmann-type brick furnace when typical coal consumption was partially substituted by municipal solid waste incineration (co-fired) in Tabio, Colombia (García C.A., 2007; García, C.A., Moreno, J.C., Giraldo, L. and Sapag, K., 2007, pp. 1-11; García, C.A., Sanchez, F. and Moreno, J.C., 2006, pp. 1500-1508). The final pieces manufactured with clay and ash must

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meet local requirements concerning seismic-resistance regulations (NCR 10) to guarantee structure stability, according to ICONTEC NTC 4205-1.

Methodology

Ten samples were made for each ratio: 100:0, 99:1, 95:5, 90:10, 80:20 and 60:40. Both clay and ashes were homogenised to 75 μm size (García C.A., 2007). The bricks were produced by extrusion, mixing clay, ash and water. These bricks were calcined in a laboratory using a Barnstead furnace (model 48000), following the typical temperature for a Hoffman furnace slope, as shown in Figure 1.
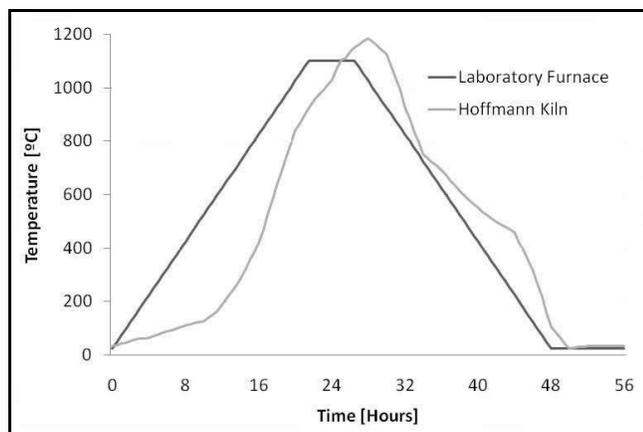

Figure 1. Sintering temperature profile (García C.A., 2007)

The following tests were carried out for measuring the stability of encapsulating the ashes into the clay.

- **Scanning electron microscopy (SEM)** - this test provides morphological study of surfaces on a molecular scale;

- **X-Ray diffraction (XRD)** – this technique is used for determining molecules' crystalline structure;

- **X-Ray fluorescence (XRF)** – this analysis allows finding a sample's chemical composition and finding the quantity of refractory elements;

- **Atomic absorption spectroscopy (AAS)** – this test is used to determine elements in a sample (Dyer, J. R., 1965). These tests were applied to the clays, the ash and the bricks for finding differences, establishing changes and quantifying stabilisation effectiveness;

- **Differential thermal analysis (DTA)** – this test is used for determining phase changes in a sample compared to a reference inert sample and finding data about transformations, such as glass transition, crystallisation, melting and sublimation; and

- **Thermal gravimetric analysis (TGA)** – this is a continuous study of variation concerning the mass of a sample submitted to thermal change in controlled conditions.

These techniques provided data about materials' thermal behaviour. Different volatile elements were determined as well as the temperatures at which the transitions occurred. It showed the possibility that, at any given time, molecule dismembering from the tested bricks might have occurred. Molecule dismembering may have affected these bricks' physical, mechanical or environmental performance:

- **Infra-red spectroscopy (IRS)** - this technique led to identifying existing links within a molecule;

- **Mercury intrusion porosimetry** - is mainly used for studying macro porosity (d >50nm);

- **Thermal programmed reduction (TPR)** is a test that determined the numbers of reducible species present in a catalyst and revealed the temperature at which reduction occured.

Table 1. Equipment used for the XRD, FRX, ATD, AAS and SEM tests

Test	Equipment	Model	Characteristics
XRD	Rigaku	Miniflex	2-70∘ (c/0.02 ∘), 2∘ min - 1
XRF	Phillips	PW 1400	XR tube, Rh anode, Collimat HR.
DTA	Netzsch	STA 409 PC LUX	30 -, 15 ∘ min - 1
AAS	Perkin Elmer	Aanalyst 300	
SEM	Fei	Quanta 200	Res 1.2 nm @ 30kV/3.0 nm @ 1.0 kV

XRD, XRF, DTA and AAS tests were performed in Los Andes University's chemistry laboratories in Colombia. TCLP lixiviation tests were performed at Los Andes University's chemistry department, according US Environmental Protection Agency standard methods and equipment. These heavy metals were studied because of their impact on human health: arsenic (As), barium (Ba), cadmium (Cd), chrome (Cr), mercury (Hg), silver (Ag), lead (Pb) and selenium (Se). Mercury porosimetry analysis and TPR were performed at the Universidad Nacional San Luis' Surface Science and Pore Mediums Laboratory in Argentina and SEM tests were performed at the Universidad Nacional de Colombia's Tests and Research Centre.

The following tests were performed to measure the quality of the clay/ash mixtures:

- **Water absorption:** to ascertain the capacity of exerting attraction on the water in contact with a brick. This measurement is directly correlated to porosity (ICONTEC, NTC 4017);

- **Mechanical resistance to compression:** this assay determines a brick's structural capabilities (ICONTEC, NTC 4017); and

- **Initial absorption rate:** measures the capacity of a dry structural unit for capillary absorption; this factor affects the quality of the link between brick and mortar (ICONTEC, NTC 4017 and ASTM, Standard C4302).

Results and Discussion

Table 2 shows the chemical composition of the clay and ashes used. FXR and AAS tests reported a possible affinity between clay and ash. High inorganic matter content can be found in ash consisting of oxides from different metals. Silica content in the clay was low; the silica:alumina rate was close to 2.4:1 and potassium content was significant, so this could have been illitic clay. SiO_2 percentage in the ash (41%) was lower than in the clay, as Al_2O_3. The ashes tested had an acceptable heavy metal concentration according to government (DAMA, 2001 and Colombian Ministry of the Environment regulations, 2002).

Infrared analysis showed similar absorption bands for different clay/ash ratios, indicating no formation of new compounds as a result of reactions between ash and clay, although new structure formation generated loss in definition of characteristic bands for the tetrahedral layer as well as for the octahedral layer. The

corresponding bands were softened due to sample calcination and thus represented deformation of Si-O-Si link. Starting clay structure alteration could be seen by the disappearance of some of the bands and widening and displacement of most of them. Figure 2 shows results for the 90:10 sample.

Table 2. Chemical composition of clay, ash and heavy metals found in the ashes. XRF and ASS analysis. Maximum accepted in lixiviation tests

| Substance	CLAY	ASHES	Metal	CONCENTRATION (mg L-1)	
	%	%		Ashes	Maximum
SIO_2	58.5	41.0	Arsenic (As)	1.120	5
Al_2O_2	24.7	16.0	Barium (Ba)	2.260	100
CaO	0.5	12.5	Cadmium (Cd)	0.430	1
MgO	0.3	2.5	Chrome total (Cr)	3.450	5
Fe_2O_2	4.8	8.6	Copper (Cu)	1.560	
P_2O_3	0.5	8.4	Mercury (Hg)	0.023	0.2
TIO_2	0.9	1.1	Magnesium (Mg)	4.720	-
K_2O	1.5	0.8	Nickel (Ni)	3.370	-
MnO	0.8	0.7	Lead (Pb)	1.230	5
N_2O_3	0.0	3.5	Silver (Ag)	0.083	5
$CaCO_2$	0.0	2.5	Selenium (Se)	0.021	1
Fe_2SIO_4	0.0	0.4			
Humidity	7.5	2.0			
Apparent Porosity	1-5%	15-20%			
Bulk density [g cm³]	2.7	1.22			
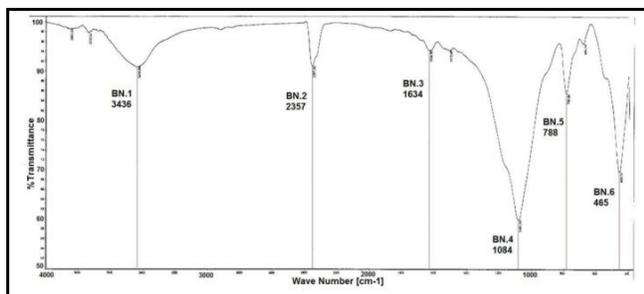
Figure 2. IR performed on the 90:10 clay:ash mixture. % transmittance of wave number [cm-1].

Band No. 1. This band oscillated around 3,437 cm^{-1}. It was wide and showed medium intensity which could be attributed to O–H stretching. This phenomenon could be explained by water absorption caused by tension vibration of hydroxyls associated by hydrogen bridges between interlaminar water;

Band No. 2. This band oscillated around 2,357.88 cm^{-1} and was characteristic of Si–H from SiH, SiH2 vibrations;

Band No. 3. This band oscillated around 1,643.03 cm^{-1}. It was weak and characteristic of water's H-O-H link deformation;

Band No. 4. This band showed oscillations around 1,084.2 cm^{-1}. It was strong and corresponded to Si–O stretching of the tetrahedral layer. There was a spike attributed to Al-O link vibration of the octahedral layer. From 1,000 cm-1 it was complex because of blowing of OH deformation absorption bands from the octahedral and tetrahedral layers;

Band No. 5. It had oscillations around 788,40 cm^{-1}. It was a weak band and harmonic for Si–H from SiH, SiH2 and SiH3 vibrations;

Band No. 6. This band oscillated within 460 cm^{-1} and 470 cm^{-1}. It showed Si-O-Si links associated with amorphic silica corresponding to octahedral vibrations. This band, and another close to 3,720 cm^{-1}, reflected di-octahedral hydroxyls thereby confirming

that this clay had a 2:1 trilaminar structure material, and was probably illitic.

Some phases were identified using X-Ray diffraction analysis. The movement of the peaks indicated diffractogram modifications and therefore in the phases found. These peaks were compared and analysed with charts reported by the Joint Committee on Powder Diffraction Standards for 1995 and the International Centre for Diffraction Data database (version 2.16, PA 19073 USA). Silicates and aluminates were found, as well as typical minerals like quartz. Phases identified in the clay at 1,100°C were mullite and quartz. Mullite was also identified as the main phase at 1,100°C in the ash, followed by quartz and a low proportion of cristobalit. Diffractograms of ash concentration lower than 20% were similar to those of pure clay. Diffractograms concerning ash concentration mixture were closer to those of just ash.

Figure 3 summarises diffractogram results for a specific clay:ash mixture. According to the pertinent literature, quartz becomes modified into cristobalit at high temperatures and illitic changes into mullite, which is precisely what the XRD tests showed (Maritan, L., Nodari, L., Mazzoli, C., Milano, A. and Russo, U., 2006, pp. 1-15).
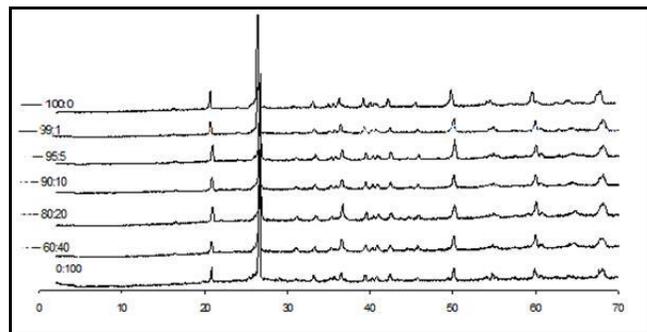

Figure 3. XRD Diffractograms for clay:ash mixtures

The cristobalit and mullite phases were more stable than those for illite and quartz. XRD modifications implied a change in clay's crystalline structure. Some heavy metals from the ash could have been introduced into the clay's matrix according to their cationic interchange capacity (CIC), but this was not measured. This clay had a CIC allowing some metals present in the inter-laminar layers to exit and other cations around the matrix to enter. Some spectra are shown in Figure 4.
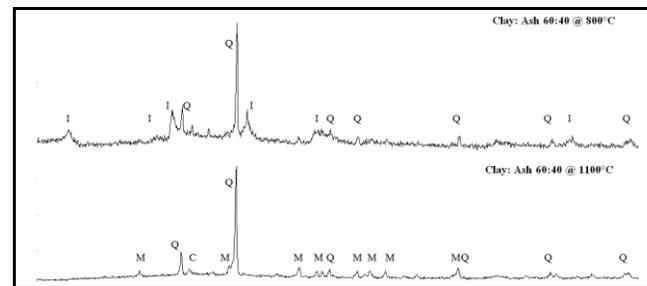

Figure 4. XRD Diffractograms for 60:40 clay:ash mixture (Illite, Mullite, Quartz)

Previous analysis has confirmed some aspects cited by Sun, D.D., Chang, L et al., 2001, pp. 916. They mentioned that stabilisation happens in two stages. The first consists of encapsulation where the clay forms a physical barrier around the metal, thereby preventing lixiviation. However, if the clay matrix breaks, the material could emerge into the environment. The second stage starts with absorption and ends with metal incorporation into the

ceramic matrix. This results in a chemical link formation between metal ion and clay, becoming part of the matrix and eliminating metal lixiviation happening. The first stage occurs at low temperatures and is due to an ionic interchange where the metals are attracted towards the negatively-charged clay's crystalline structure's surface layers. This phenomenon is known as external adsorption.

As the clay was mostly illite, external adsorption and also internal adsorption may have been present. Interlaminar cations in the structure may have been replaced by surrounding heavy metals. At temperatures close to 1,100°C it can be seen that the illite present in clay became mullite. Metals which had been initially adsorbed by the illite were incorporated into the new mullite matrix.

Figure 5 shows some microphotographs (SEM) where quartz may be identified as a series of large-sized grains (A) and another phase corresponding to the clay's mineral (B).
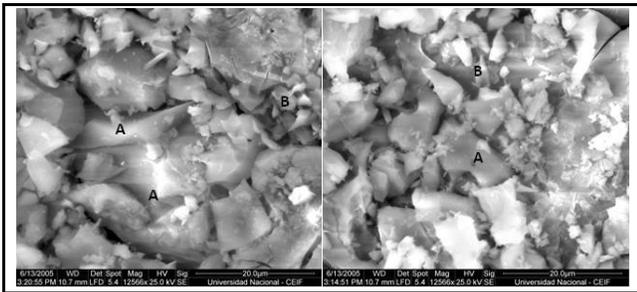

Figure 5. SEM 1. Sample 100:0, and SEM 2. Sample 95:05, where A=quartz and B=mullit

As ash concentration increased in final mixtures, structural changes and grain size distribution difference were observed in SEM images (Figure 5). The amount of larger grains decreased as the presence of smaller grains increased at higher ash ratios. The XRD for each sample was very similar, except for 60% and 0% clay. A higher quantity of metal was identified in the analysed section in these concentrations. A high quantity of potassium was found, confirming clay's illitic characteristics.

Thermal analysis revealed the materials' good thermal stability. Change in quantity of mass during heating time was very low (<1%). This small variation presented at the beginning of the analysis was attributed to remaining water being released. Then quantity of matter remained practically constant during the rest of the assay, showing great thermal stability with temperature. This represents a good characteristic for construction materials which will probably be exposed to high temperatures.
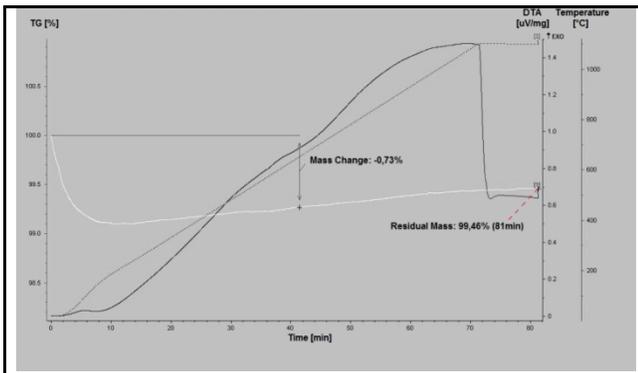

Figure 6. Thermograms from thermal analysis of a 95:5 mixture

DTA in Figure 6 shows mass behaviour at different temperatures. Loss of water inside material was found at 110°C. Brief curvature between 500°C and 700°C may imply a small modification or transition between a material's phases, as metals exchange within the matrix. An endothermic signal was noted close to 900°C, where the material underwent a transition from one phase to another with higher stability. It possibly corresponded to a change from an illitic phase to a mullitic one; however not all the clay became transformed.

According to Figure 6, ash incorporation affects pore formation. Clay has a 2 μm diameter and low quantity of pores. Incorporating 10% ash increased the amount of pores and decreased their medium size diameter to 1.5 μm.

The percentage of lixiviated heavy metals was low in lixiviation trials, implying that stabilisation inside the clay's matrix was strong, and physical stabilisation and chemical and structural stabilisation took place. Lixiviation results and analysis can be found in García-Ubaque, Moreno-Piraján et al, 2007, pp. 352-362.
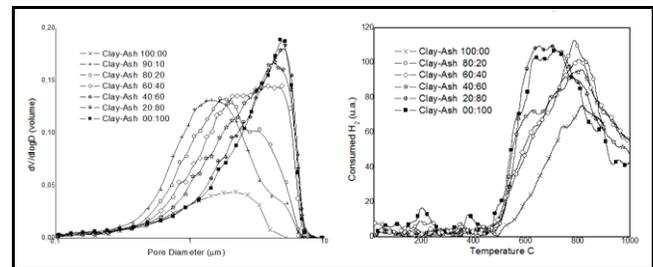

Figure 7. Mercury porosimetry of the bricks and thermal programmed reduction (TPR) results for bricks obtained with different mixtures.

Ashes contain different metals, which became transformed into oxides after thermal treatment. A study involving thermal programmed reduction may provide information about these oxides interaction with the clay's matrix (TPR profiles are shown in Figure 7). The reduction temperature was high in all cases (> 500°C), indicating a strong reaction with support. This was consistent with results concerning metal incorporation into the structure. Clay had a higher resistance to reduction at 800°C. As ash content increased, medium hydrogen consumption temperature decreased, implying that a higher quantity of metallic oxide was exposed to reduction at a higher quantity of ash. This data matched the porosity study, where ash components were responsible for porosity, probably forming pore walls, and remaining exposed to the action of hydrogen.

The characterisation tests for masonry bricks indicated that their quality satisfied local standards (Table 3). Absorption increased at low temperatures as ash content also increased due to increased porosity. Load/rupture and resistance behaved the same way when ash quantity changed. Absorption increased for quantities of ash between 5%-10% but decreased significantly with higher quantities. This could be explained because of smaller pore size at 5%-10% than in other samples. Furthermore, pores were not completely integrated to the matrix with high quantities of ash and they became dispersed throughout the brick, generating points of lower resistance. Water absorption increased with increased ash concentration; this was not favourable because it reduced stability as time elapsed (Raimondo, M., Pondi, M., Mazzanti, F., Stefanizzi, P. and Bondi, P., 2007, pp. 926-932). Bricks did not meet local regulations for those having 40% ash content, as the maximum values were exceeded. This could have been attributed to an increase in the material's porosity as

the proportion of ash in the mixture increased (Freyburg, S. and Schwarz, A., 2007, pp. 1727-1733).

According to these findings, samples having $\leq$ 10% ash concentrations had greater advantages regarding resistance and load/rupture properties were maximised and local regulations were met, although cold trap was high. 90:10 mixtures permitted ash consumption and maximise brick properties (Kouakou, C.H. and Morel, J.C., 2009, pp. 27-34).

Other studies (Haiying, Youcai, Jingyu, 2011, pp. 331-334; Pérez-Villarejo, Eliche-Quesada *et al.*, 2012, pp. S349-S354) have concluded that 20% ash content in bricks is a suitable proportion to balance ash disposal and final quality product properties such as water absorption and compressive strength.

Table 3. Compression and absorption trials for blocks manufactured with

Sample No.	Test date	Sample identification	Water absorption %	Load rupture (kg)	Strength		
					kg/cm²	lb/in²	MPa
1	Nov-04	100-0	12.44	14.300	39.95	568.69	3.92
2	Nov-04	99-1	12.74	14.180	39.63	564.22	3.89
3	Nov-04	95-5	13.06	16.260	45.43	646.79	4.45
4	Nov-04	90-10	13.22	16.480	**46.07**	**655.84**	**4.52**
5	Nov-04	80-20	13.50	12.160	33.93	483.05	3.33
6	Nov-04	60-40	**14.88**	10.780	30.16	429.39	2.96
Limit Value (NTC-4205)			**<14.00**				**>3.00**

Data obtained were processed by general liner model (GLM) to establish their statistical significance.

According to GLM results (Table 4), a <.0001 F-value led to concluding that there was no equality of mechanical resistance to compression test for the 6 levels of ash concentration; ash concentration thereby affects bricks' resistance response.

Table 4. Statistical analysis by general liner model (GLM)

GLM procedure					
Dependent Variable: compression					
source	DF	Sum of squares	Mean square	F-value	Pr>F
Model	17	31.51	1.85	17.97	<.0001
Error	72	7.43	0.10		
Total	89	38.93			

R-squared	Coefficient of variance	Root MSE	Average compression		
0.809279	8.355883	0.32	3.84		

Source	DF	Type I SS	Mean square	F-value	Pr > F
AC	5	31.22	6.24	60.55	<.0001
Replica (ash-clay)	12	0.28	0.02	0.23	0.9964

Conclusions

Quality verification tests showed that bricks having low ash content supported higher loads and had higher resistance than bricks made of pure clay. This happened for ash concentrations near10% where the properties became maximised. When ash content increased to 20% or 40%, properties decreased significantly, for example units having 20% decreased by up to a 15% and 40% in resistance. This data agreed with the porosity study: the more the pores, the lower the resistance and higher water adsorption. Pore quantity became reduced art 90:10 ratio but their size increased slightly, whereas the number of pores increased at the other ratios, as well as their size.

Based on stability and quality tests and comparing the results with current local regulations bricks can be made from clay and ash calcined units, optimum ratio being 90% clay and 10% ash.

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