



Effect of Quartz and Red-Clay to Phonolite Ratios on Some Properties of Floor Tile

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Abstract — This research paper presents the procedure of preparing and testing of experimental ceramic slabs in order to examine and investigate some of their physical and mechanical properties. These slabs were divided into two types; type (I) includes slabs made of either Quartz, Phonolite, Red-Clay, Green-Clay or Kaoline, whereas, type (II) includes slabs various in their Quartz and Red-Clay to Phonolite ratios. Other constituents of type (II) slabs such as Green-Clay, Kaoline were intentionally kept comparable for the sake of comparisons. Methods of preparing and testing of the experimental ceramic slabs were performed according to the technical constraints and facilities available at the Investment Complex for Manufacturing Building Materials (INCOMA) at Misurata-Libya. Several tests and measurements have been done on the experimental ceramic slabs of both types. The physical properties of type (I) slabs and raw materials ceramic powder were investigated with the determination of their water absorption, thermal shrinkage, heat loss, bulk density and fluidity. The breaking strength and impact resistance of type (II) slabs were also determined. These results were enhanced with surface and cross-sectional metallographic investigations for both types of experimental slabs. It is thought that the presence of a considerable amounts of oxides such as CaO, K₂O and Fe₂O₃ in the Red-Clay decreased significantly the fractural strength, but improved the impact resistance of the experimental ceramic slabs. Conversely, the presence of high alumina and silica contents together with trace amount of ZrO₂ in the Phonolite probably behind the improvement of their fractural strength, and decline of the impact resistance of the experimental ceramic slabs.

Index Terms: Floor tiles, physical and mechanical properties, RedClay, Quartz, phonolite, ratios.

I. INTRODUCTION

Raw materials required for the production of ceramic tiles includes: Quartz & Phonolite and Clays[1]. Quarts and Phonolite serve as the primary source of Silica and Alumina respectively, to give tiles the desired

strength, whereas, Clays are to serve as a binder[2]. Ceramic tiles are divided into several groups, according to their method of manufacture and their water absorption. Water absorption is associated with the porosity of the material. Consequently, choosing raw materials is one of the most important aspects to control the quality of ceramic tiles. The preparation of these materials before firing also had significant effects on the final product[3-7]. However, at the heart of ceramic production is a pyrotechnology - how the raw materials change during firing. Ceramic tile production comprehends many different processes according to each different finished product. Ceramic processing is used to produce commercial products that are very diverse on size, shape, detail, complexity and material composition, structure, and cost[5]. Their strength as a consequence possible applications, depends both on the constituents of ceramic composites, and on their degree of homogeneity. Such measurements are very important because their mechanical properties are often not uniform across the whole surface[4]. Price of ceramic tiles also depends on purity of texture, accuracy of color, shape and properties.

Despite all of the efforts to improve the performance of ceramic tiles, problems on quality of the tile production in this regard still open and needs further investigations. According to our personal visit to INCOMA and the useful discussion with their management staff members, ceramic tile process industry at INCOMA generates a considerable amount of wastes due to various and unknown reasons for them.

The aim of this research paper is to investigate the effect of Red-Clay and Quartz to phonolite ratios on some of the physical and mechanical properties of floor ceramic tile materials in general and to the ceramic tiles produced at INCOMA in particular.

II. PRODUCT MIX OF CERAMIC TILES

In ceramic tiles production, clays serves various functions such as a binder, a suspension aid and an inexpensive source of alumina[2, 4, 5], their plate-like structure and the physicochemical nature of their surface enable them to form, with water, colloidal suspensions

and plastic pastes. Most kaolins are primary clays[6, 7]. In addition, it is refractory due to its low impurity content. The action of the water tends to grind up the clay into a much smaller particle size. The sources of clay-forming minerals are aluminous rocks, particularly those containing feldspar.

A large amount of silica occurs as free silica that is mostly in the form of quartz, although most of it is combined with other elements in the silicate minerals[8, 9, 10]. Addition of silica sand decreases its unfired strength and plasticity, but assist to facilitate escape of gases during drying and firing. It also reduces drying shrinkage and increases the whiteness of the fired body[10]. The transformation of residual quartz into cristobalite can then start from 1,200°C onwards[8-10]. It is favored by the rise in temperature, the use of fine grained sand, the presence of certain impurities and a reducing atmosphere.

Kaolin is a very refractory due to its high content of alumina (Al_2O_3). A very fine granularity is a good opacity due to the lamellar shape of the crystals of the kaolinites. Kaolin also characterized by its high melting point which exceeds 1800°C[2]. The amorphous metakaolin, $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ then formed, exhibits a structural organization directly derived from that of kaolinite. The exothermic transformation observed between 960 and 990°C is a structural re-organization of the amorphous metakaolin, sometimes associated with the formation of phases of spinel structure like $\text{Al}_8(\text{Al}_{13,332,67})\text{O}_{32}$ (γ variety of Al_2O_3) or $\text{Si}_8(\text{Al}_{10,675,33})\text{O}_{32}$. In these formulae, represents a cation vacancy. Between 1,000 and 1,100°C (often around 1,075°C), these phases are transformed into mullite stoichiometry ranging between $3\text{Al}_2\text{O}_3$, 2SiO_2 and $2\text{Al}_2\text{O}_3, \text{SiO}_2$. During this reaction, amorphous silica is released. The surplus amorphous silica starts to crystallize in the form of cristobalite from 1,200°C onwards. It should be noted that the impurities present, the degree of crystallinity and the speed of heating influence each of these transformations[11-14].

III. PLASTIC VERSUS NON- PLASTIC BEHAVIOR OF CERAMICS

The plastic ceramic raw materials are essentially the clays and the kaolin that have, as their main function, to supply plasticity and to provide adequate mechanical strength[15]. The large group of non-plastic ceramic raw materials include minerals, rocks and artificial chemicals, that when mixed with water is not plastic. Among the non-plastic raw materials, feldspars, talc and quartz are the most used and decrease the plasticity[15], A part of the non-plastic ceramic raw materials such as quartz acts as a filler, reducing linear shrinkage of the body when drying or firing[16]. On the other hand, non-plastic raw materials are used for sintering, fluxing and melting or to increase the refractoriness[2, 16, 17]. Plastic raw materials include kaolin, clay and bentonite. The classic or "triaxial" ceramic body consists of three major components: clay, quartz which is a non-plastic material and feldspar, that acts as a flux providing the glassy phase[3, 17].

IV. EXPERIMENTAL MATERIALS AND METHODOLOGY

In this section, the steps of preparing and testing the experimental ceramic slabs (tiles) are summarized. These steps were performed according to INCOMA laboratory specifications and their technical constraints together with the guide of ISO 10545-4 (02.2019).

4.1 Experimental materials

The chemical analysis of the ceramic raw materials as received from INCOMA is presented in Table 1. Thirty (30) of two types of experimental ceramic slabs (110x55x7mm in dimension) were prepared.

Type (I) experimental ceramic slabs were made of either Quartz, Phonolite, Red-clay, Green-clay or Kaolin. The chemical composition of these raw materials are presented in Table 2. Each of these raw materials were mixed individually with an appropriate amounts of dis-agglomerate (organic chemical compound) and water as reported elsewhere[18].

Type (II) experimental ceramic slabs were made of a mixture of raw materials mentioned above with a different proportions and classified into two groups (A) and (B) as tabulated in Table 3.

Table 1 Chemical composition (wt%) for the ceramic raw materials of floor tiles at INCOMA[1]

Materials	Composition												
	Al ₂ O ₃	SiO ₂	CaO	Fe ₂ O ₃	Na ₂ O	K ₂ O	TiO ₂	MgO	Cl	ZrO ₂	MnO	SO ₃	P ₂ O ₅
Red-Clay	19.49	58.49	2.82	7.724	0.41	6.12	1.14	--	--	--	3.91	--	0.2
Green-Clay	18.3	62.99	2.41	5.676	1.42	4.89	1.34	1.96	--	0.192	--	0.646	0.15
Quartz	1.44	96.32	0.586	0.657	0.19	0.513	0.184	0.1	--	--	--	--	--
Kaolin	30.33	64.6	0.387	1.824	0.23	0.819	1.48	0.26	0.065	--	--	--	--
Phonolite	21.3	57.17	2.16	2.167	8.12	7.25	0.315	0.46	0.216	0.1739	0.17	--	--

Table (2): Chemical composition (wt%) of the experimental ceramic slabs of group (A)

Tile No.	Group (A)						
	Feldspar	Silica	Kaolin	G. Clay	R. Clay	Dis-ag	Water
A-1	20	10	25	5	40	2	8
A-2	20	12	23	5	40	2	8
A-3	20	14	21	5	40	2	8
A-4	20	16	19	5	40	2	8
A-5	20	18	17	5	40	2	8

Table (3): Chemical composition (wt%) of the experimental ceramic slabs of group (B)

Tile No.	Group (B)						
	Feldspar	R. Clay	Kaolin	Silica	G. Clay	Dis-ag	Water
B-1	20	42	23	10	5	2	8
B-2	20	44	21	10	5	2	8
B-3	20	46	19	10	5	2	8
B-4	20	48	17	10	5	2	8
B-5	20	50	15	10	5	2	8

4.2 Preparation of experimental ceramic slabs

The experimental ceramic slabs of **type (I&II)** were prepared according to the following steps; *First*, is preparing the ceramic paste, *Second*, pressing the ceramic paste and then drying the green slabs and, *finally*, firing the experimental ceramic slabs. The parameters of these steps are summarized elsewhere[18].

4.3 Testing and characterising of the experimental ceramic slabs

Several tests have been performed on both **types (I&II)** of experimental ceramic slabs in order to examine some of their physical and mechanical behavior. Some metallographic investigations were also undertaken to enhance the obtained results.

4.4.1 Physical tests of experimental ceramic slabs

The physical properties of **type (I)** slabs of raw materials were investigated with the determination of their water absorption, thermal shrinkage, heat lose, bulk density and fluidity.

4.4.2 Mechanical tests of experimental ceramic slabs

Two important mechanical behavior for the experimental ceramic slabs including fractural and restitution coefficient were examined with the determination of their breaking strength and impact resistance respectively. Several mathematical equations were used in estimating the physical and mechanical properties of the ceramic test slabs were used as reported elsewhere[18].

4.4.3 Metallographic investigations of experimental ceramic slabs

Metallographic survey for selective experimental ceramic slabs of both **types (I&II)** using digital camera, with a unified magnification (x100) for the purpose of comparisons of the surface and cross-sectional morphology of the experimental ceramic slabs.

V. RESULTS AND DISCUSSION

5.1 Results of the physical tests

Comparisons between Figures (1 to 3) reveals controversial results between phonolite and quartz effect in terms of their water absorption, thermal shrinkage and heat lose. High porosity of quartz as indicated in Figure 4(a) was the main cause of its high water absorption as reported by Fugmann, K. G[6]. Low thermal shrinkage, the compact, flaky and glassy structure of phonolite [see Figure 4(b)] probably because of its high alumina content together with a trace amount of ZrO₂[13]. The high percentage of alumina in Koaline probably was behind its high heat lose if compared with quartz. Heat lose of alumina formers such as phonolite, Red and Green-Clays (refer to Figure 3) were relatively comparable.

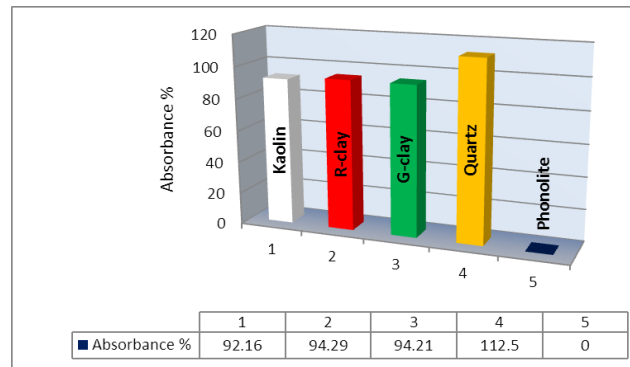


Figure 1. Comparison of water absorption between slabs of type (I)

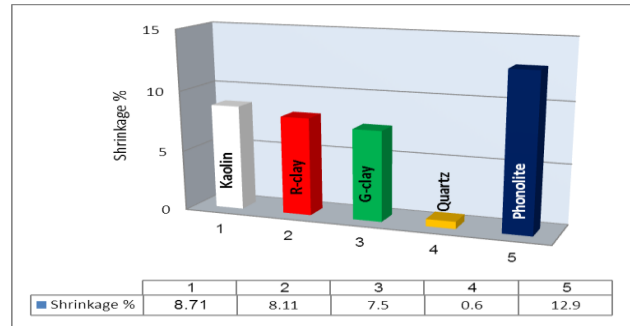


Figure 2. Comparison of shrinkage between slabs of type (I)

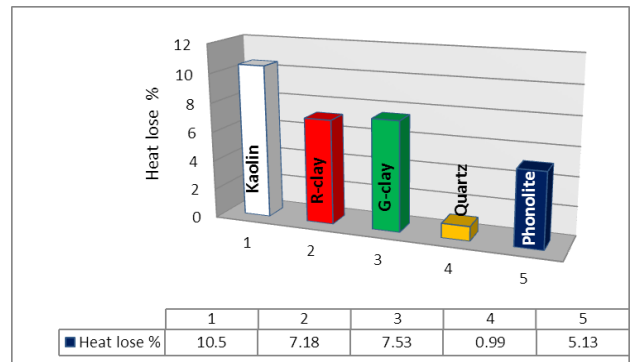


Figure 3. Comparison of heat lose between slabs of type (I)

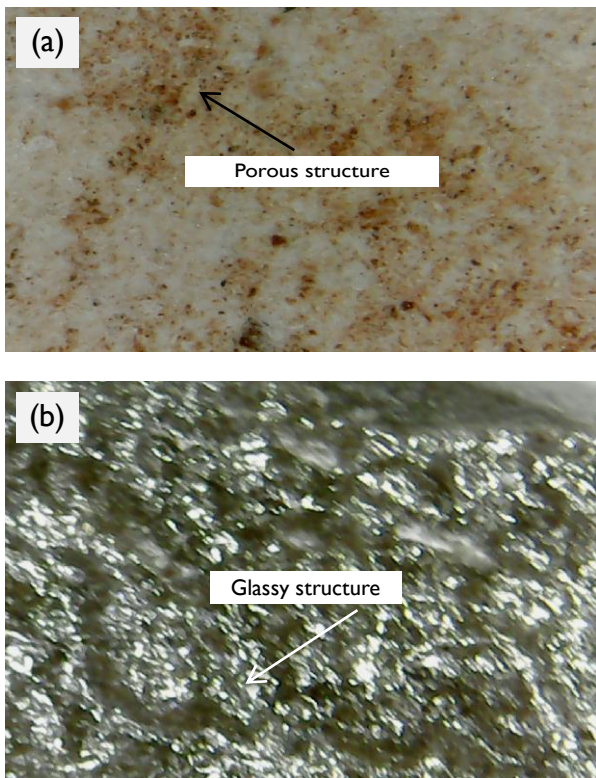


Figure 4. Comparison between surface morphology of (a) Quartz, and (b) Phonolite. (Mag. 100)

The presence of some oxides such as, CaO, MgO, SO₃ and P₂O were the main cause of high fluidity and low density of Green-Clay (see Figures 5 & 6) as reported by Fugmann K. G[6], if compared with other raw ceramic materials.

Large isolated particles with irregular morphology located at quartz and feldspar (in the phonolite) probably agglomerated with glassy matrix was observed [see Figure 7(a)]. Its origin is probably due to the differences in thermal expansion coefficients of these grains with respect to the rest of slab matrix [see Figure 7(b and (c))].

During the sintering process, the development of liquid phase is progressively closing the capillaries that constituted the open porosity and fine closed arises[13, 15], and thus improving the fractural strength of the ceramic slabs [see Figure 7(b and (c))].

The average values of the results for various physical tests performed on type (II) ceramic slabs (see Figures 8 to 12) were found to be consistence with the physical test results obtained on type (I) ceramic slabs.

The importance of the physical tests on both types of ceramic slabs is to investigate the effect of each Quartz and Red-Clay to phonolite ratio on the mechanical properties of type (II) ceramic slabs.

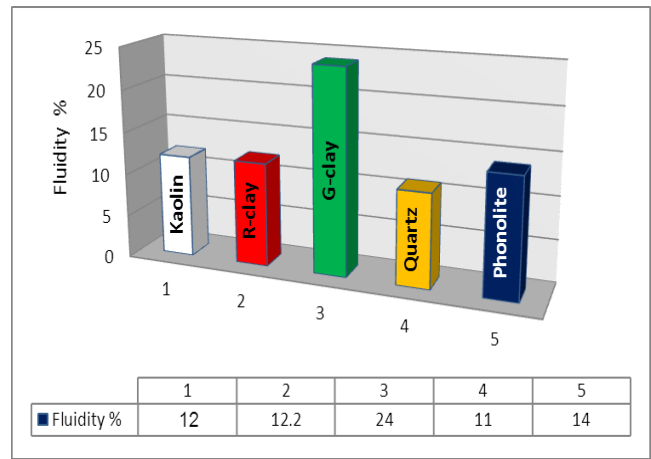


Figure 5. Comparison of fluidity between slabs of type (I)

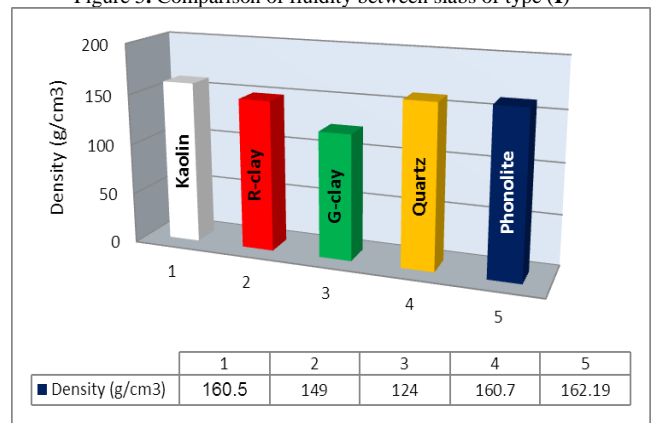


Figure 6. Comparison of density between slabs of type (I)

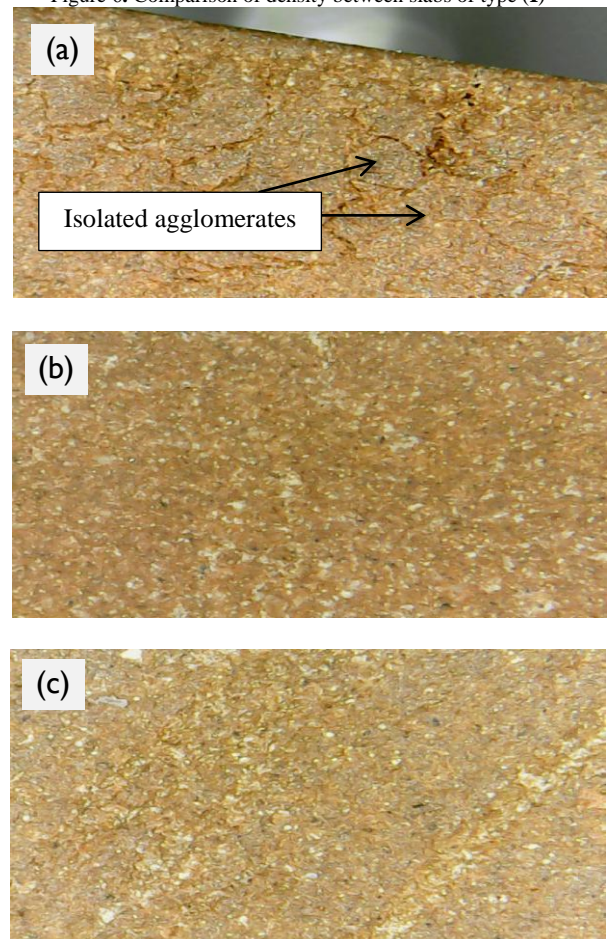


Figure 7. Surface micrographs of type (II) slabs (Mag. 100) (a) and (b) of group B, (c) of group A

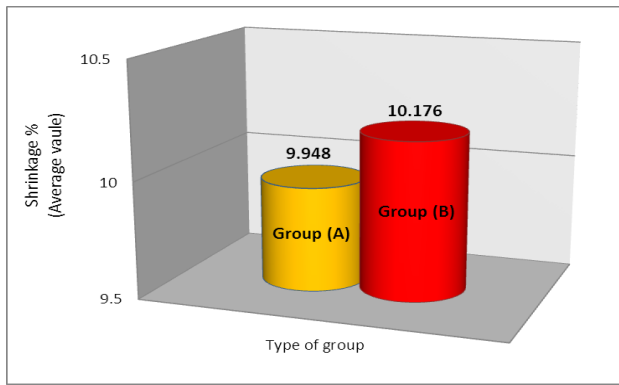


Figure 8. Comparison of shrinkage between slabs of type (II)

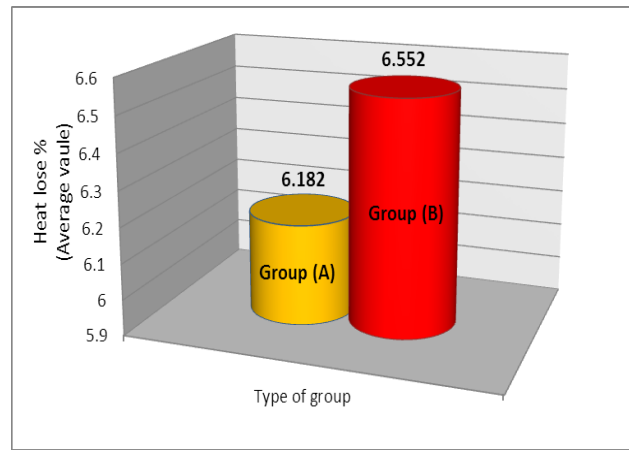


Figure 12. Comparison of heat loss between slabs of type (II)

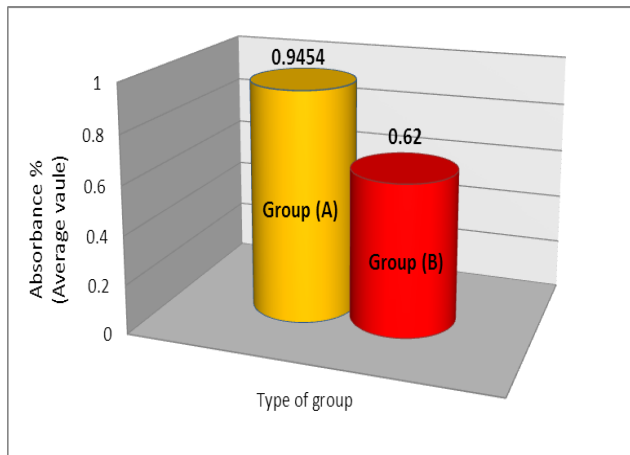


Figure 9. Comparison of water absorption between slabs of type (II)

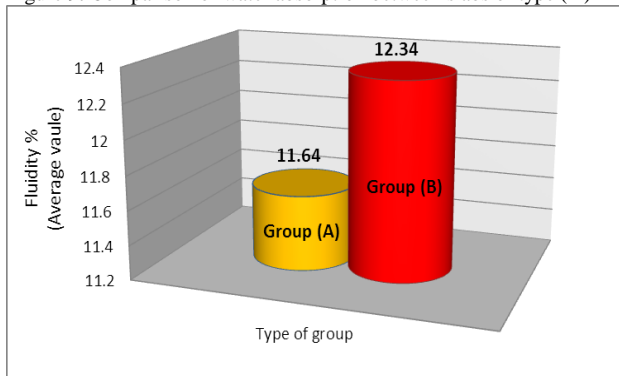


Figure 10. Comparison of fluidity between slabs of type (II)

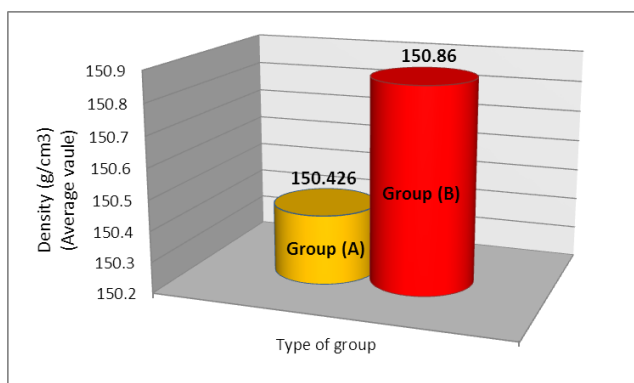


Figure 11. Comparison of density between slabs of type (II)

Some zirconias present in the phonolite have engineered crystal structures which expand when a micro-crack is formed, arresting crack growth and preventing brittle failure[13].

5-2 Results of the mechanical tests

Two important types of mechanical tests were performed on both types of ceramic slabs for the determination of their fractural strength and impact resistance. The results of these tests were enhanced with some of metallographic investigations. Fracture strength test result for the ceramic slabs of type (I) is presented in Figures 13, whereas the fractural strength and impact resistant for the ceramic slabs of type (II) are summarized in Figures 14 and 15 respectively. Inter-particle porosity is most likely responsible for the reduction in mechanical strength of the fired slabs, since they are considered stress concentrators and facilitates fracture (see Figure 16).

Regardless to the composition of unfired (green) slabs, the dynamic evolution of both microstructure and phase composition during firing, makes it difficult to predict the behavior of ceramic slabs with classical model[13]. Zirconia present in the phonolite for example, have a unique crystal structure which combines excellent strength, high fracture toughness, wear resistance, and high temperature resistance[13] as revealed in Figure 5.13. Experimental ceramic slabs of group (B) recorded higher fractural height if compared with slabs of group (A). Probably the presence of some oxides such as CaO, K₂O and Fe₂O₃ in type (II) experimental ceramic slabs reduced their fractural strength, meanwhile improved their impact resistance as revealed in Figure 14 & 15. These results were in consistence with the findings of C. M. F. Veira [15]. Fragile mode of fracture of the experimental slabs of group (A) if compared with the non-fragile behavior of slabs of group (B) as shown in Figure 17.

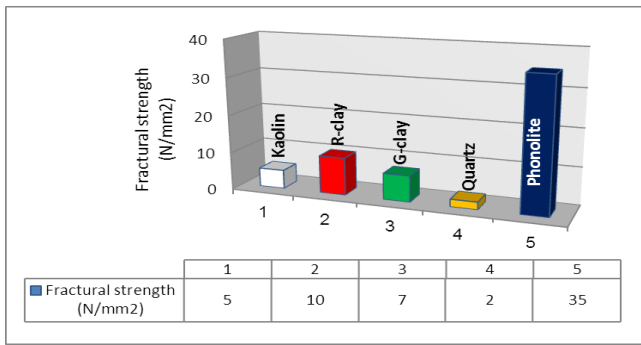


Figure 13. Comparison of fractural strength between slabs of type (I)

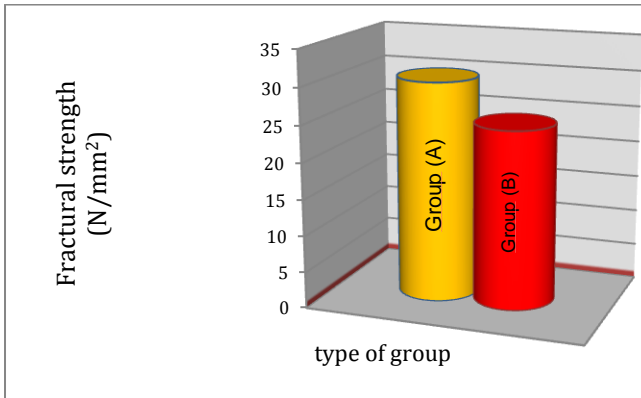


Figure 14. Comparison of fracture strength between various slabs of type (II)

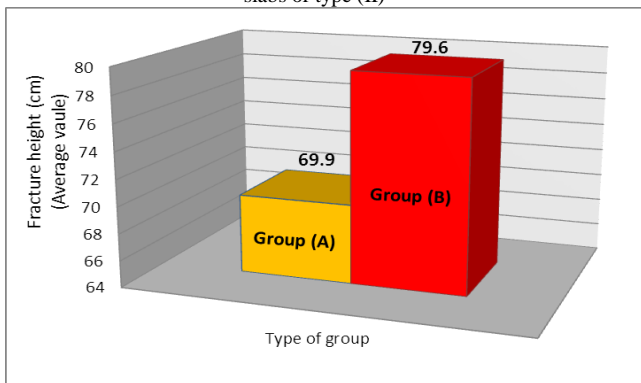


Figure 15. Comparison of impact resistant between various slabs of type (II)

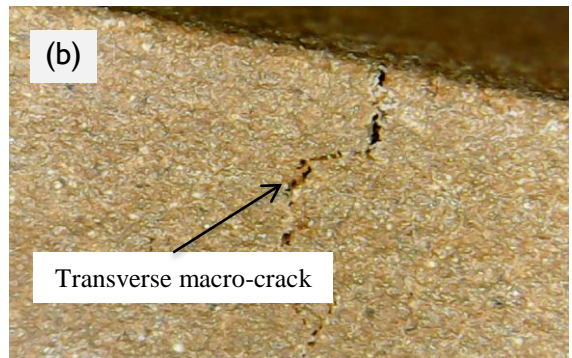
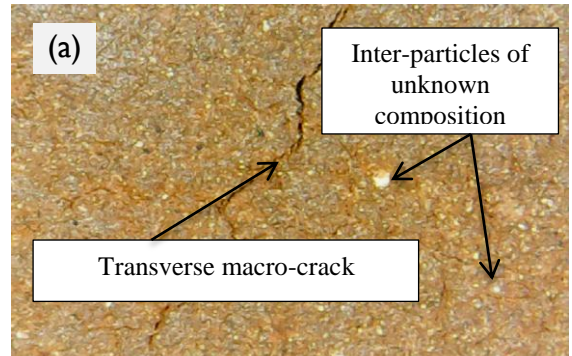


Figure 16. Slab of group B (Mag. 100)

- a) Surface micrograph
- b) Cross-sectional micrograph



Figure 17. Images for the fracture mode of floor ceramic tiles

- a) For group A, shows fragile behavior.
- b) For group B, shows non-fragile behavior.

V. SUMMARY AND RECOMMENDATIONS

6.1 Summary

In this research paper, some of the physical and mechanical properties for the ceramic raw materials (type I) and their mixed slabs (type II) were investigated. The obtained results, revealed that the quartz to phonolite ratio in the mixed slabs were more beneficial to the mechanical properties for the ceramic floor tiles produced at INCOMA than the Red-Clay to phonolite ratio. The presence of a considerable amounts of oxides such as CaO, K₂O and Fe₂O₃ in the Red-Clay decreased significantly their fractural strength and impact resistance of the ceramic floor tiles. Conversely, the presence of high alumina content together with trace amount of ZrO₂ in the phonolite improved the mechanical properties of the ceramic floor tiles at INCOMA.

6.2 Recommendations

1. Several characterization techniques such as SEM, EDAX and XRD must be used to analyse and examine the ceramic raw materials and tiles.
2. Multi-layering for each type of raw material at the storage yard of INCOMA will be useful, to avoid any possible contamination (or mix) and for better distribution of the raw materials.

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