Nanoceramics: Materials, Properties, Methods and Applications-Part II

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Abstract:
The overwhelming interest of nanotechnology including nanoparticles, nanocrystals, nanostructure and nanoscales of all ceramic materials especially nanoceramics is one of the most exciting fields so that it incorporates materials sciences, cement, concrete and other building materials particularly bricks, ceramic wall and floor tiles and also advanced bioceramics industries. In this review article, the author wishes to indicate the importance of nanomaterials and/or nanoparticles for improving the physicochemical and mechanical properties, thermal shock, thermal expansion, firing resistance and microstructures of the resulting nanoceramic products.

1. Introduction

Nanoceramic is a type of nanoparticles that is composed of ceramics, which are generally classified as inorganic, heat-resistant, nonmetallic solids made of both metallic and nonmetallic compounds. Research has been emphasized on this material because of its unique properties on a nanoscale. On a macroscale, ceramics hold brittle and rigid traits and would break upon high impact against other hard objects. In the past few decades, research has shown that on a nanoscale, nanoceramics take on a larger variety of functions than all known materials [1]. Nanoceramics were discovered in the early 1980s and formed using a process called sol-gel which mixes nanoparticles within a solution and gel to form the nanoparticle. Later methods involved sintering, which is formation of solid through pressure and heat. Because of these methods, advancement in researching the properties of nanoceramics was possible. These properties include being dielectric, ferroelectric, piezoelectric, pyroelectric, ferromagnetic, magnetoresistive, superconductive, and electro-optical. Recently, a lasering process involving polymers and particles of ceramics to form a completely different architecture of nanoceramic called structure nanotruss. This structure gave the nanoceramic particles a flexible and durable property unlike its macroscopic properties, which are brittle and rigid.

2. Properties of Nanoceramics

The nanoceramics have unique properties because of their size and molecular structure. These properties are often shown in terms of various electrical and magnetic physics phenomenon which include:

1. Dielectric: An electrical insulator that can be polarized (having electrons aligned so that there is a negative and positive side of the compound) by an electric field to shorten the distance of electron transfer in an electric current.

2. Ferroelectric: Dielectric materials that are able to be polarized in more than one direction (the negative and positive sides can be flipped through a certain electric field).

3. Piezo-electric: Materials that accumulates an electrical charge when mechanical stress (bending, pressured, etc.) is applied to it.

4. Pyro-electric: Material that can produce a temporary voltage when there is a certain temperature change, hot or cold.

5. Ferromagnetic: Materials that can to sustain a magnetic field after being magnetized by source.

6. Magnetoresistive: Materials that can change electrical resistance in accordance to an external magnetic field.

7. Superconductive: Materials that exhibit zero electric resistance when cooled to a critical temperature.

8. Electro-optical: Materials that change in optical properties when place in an electric field.

Nanoceramic is more than 85 % air and is very light, strong, flexible, and durable. The reason for these qualities to all be present in a single material is fractal nanotruss, a nanostructure architecture developed by Julia Greer [2]. They developed a technique in order to construct these nanostructures using a "three-step process" for creating these complex structures [3]. The nanoceramic Greer is currently using is called alumina, or aluminum oxide, and its maximum compression is about 1 micron from a thickness of 50 nanometers. After its compression, it can revert to its original shape without any damage on the structure. Since this is nanotruss was coated with alumina, different molecules could yield a different result.

3. Sintering

Sintering is the process of consolidating nanoceramic powders using high temperatures to pressure the powder into a solid material. High temperature sintering results in a rough material that deteriorates the properties of ceramics and requires a lot of time to obtain an end product. This technique also limits the variety of geometry possible for the final form of the nanoceramic. Because of these disadvantages, microwave sintering has been developed to overcome the time and energy problems of the previous technique. In microwave sintering, radiation is produced from a magnetron, a device that produces electromagnetic waves, to vibrate the powder and thus heating them. This method allows for heat to be instantly transferred across the entire volume of material instead of a graduate temperature gradient [1]. They first start placing the nanopowder in an insulation box that is composed of low insulation boards in order for the microwaves to pass through it. They place it in an insulation

box in order to increase the temperature because nanoceramics don't absorb microwave energy well in room temperature. Inside the boxes are suspectors that absorb microwaves at room temperature to act as an initial heat source and initialize the sintering process. When the box is placed in the microwave furnace, the microwaves heat up the suspectors, to about 600 ℃. Then, the nanoceramics start to absorb the microwaves and bind the nanoparticles together.

4. Applications

Nanoceramics, due to its vast amount of mechanical properties, have a large variety of functions and can be applied to many fields. These fields include energy supply and storage, communication, transportation systems, construction, and medical technology. There are also physical applications such as nanoceramics in nanotruss architecture. In this form, nanoceramic holds very flexible and durable properties so a macroscale version would make the material very light and yet be as strong as current building materials like concrete or steel. Medical technology has been using nanoceramics for bone repair and developing material that can enter the body without conflict [4]. Really, many of the technology have not advanced enough to have a large scale test for these properties. Further advancement in a large scale production of nanoceramics is needed to fully utilize the functions. Ceramic floor and wall tiles are building materials which are designated for use as floor and wall coverings both indoors and outdoors regardless of shape or size. The ceramic bodies in general, are formed of a mixture of clays, quartz, fluxes, colors and other raw materials. They undergo some processing such as milling, screening, blending and wetting. Tiles are shaped by pressing methods at room temperature [5,6]. Floor tiles are belonging to the 1st and 2nd groups with W. A. only up to 6 % [7-9]. There are two different types of floor tiles:

A) Floor tiles: They are dense bodies, fully vitrified, colored frequently by the color of the natural clays used in the batch or by colors, and can be obtained in various colors as well as different textures which can be achieved by additions. Many of these types are smooth surfaced and some are slightly self-glazed, others are made non-surface. They are characterized by high resistance to abrasion, weather stains, etc.

B) Quarry tiles: The quarry tiles are thicker and frequently larger than floor tiles and having a simple coarser surface texture with W. A. between 2-5 %. These types of tiles are used mainly in floors of kitchens, public buildings, balconies, terraces, porches, silos and yards as well as wall linings.

C) Decorated floor tiles: These are ready dense unglazed tiles with designs in various colors of used clays. The shrinkage of the various colored bodies used in the same tile must be compatible. There is a wide range of bright colors in these colored types.

The floor tiles production all over the world is based on the available local raw materials and it is usually established near the sites of these raw materials. Since, it is considered that the transportation of raw materials over large distances is uneconomic. In addition, the simple shape of the floor tiles and the convenient size of each tile in the production, make it possible to mechanization and automization of the industry [10,11]. Many attempts were made in the world, each to use local raw materials in the production of floor tiles [12-15]. Accordingly, it was found that tiles with water absorption of 7-9 % can be prepared by replacing the expensive refractory clay type

with local clay mixtures [12]. Fig. 1 demonstrates the classification of ceramic products whereas Fig. 2 shows Triaxial diagram showing areas of commercial wares.
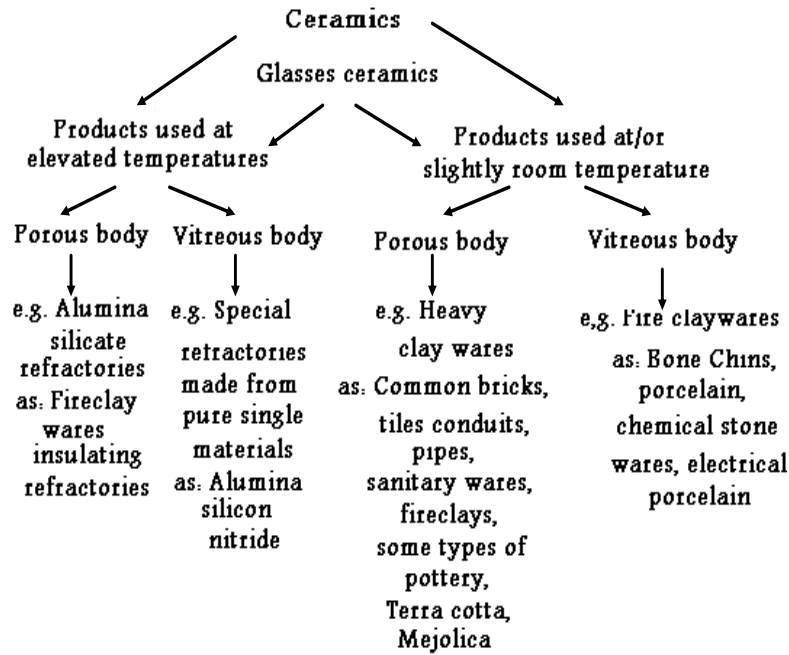

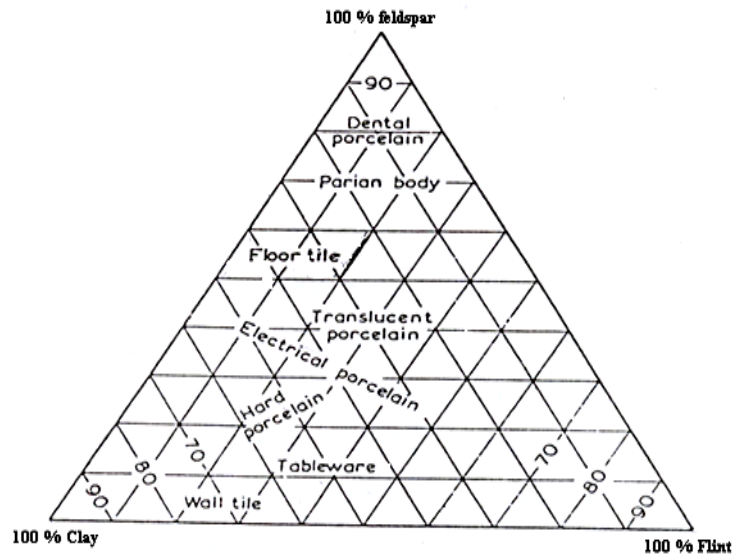**Figure 1.** *The Classification of Ceramic products.*

Figure 2. *Triaxial diagram showing areas of commercial wares.*

5. Raw materials

The chemical composition of raw materials used in the ceramic industry is shown in Table 1 and are frequently classified to:

Table 1. *The chemical composition of ceramic raw materials, wt. %.*

Materials Oxides	Clay	Feldspar	Quartz	Limestone
LOI	9.72	0.67	1.78	42.63
SO$_2$	53.47	75.37	93.63	0.08

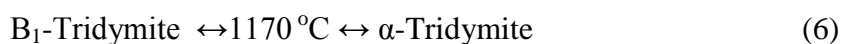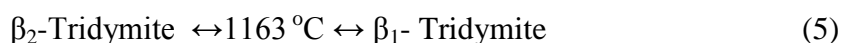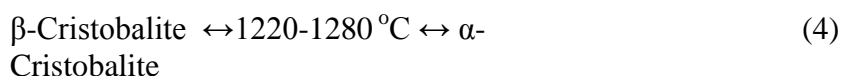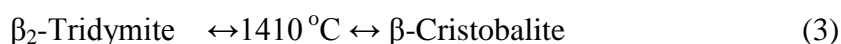| AleO$_3$	26.78	13.62	3.64	0.03
Fe$_2$O$_3$	3.99	0.41	0.08	0.04
CaO	0.60	0.53	0.18	56.84
MgO	1.38	----	----	0.10
MnO	0.03	0.03	0.02	----
K2O	1.18	5.84	0.14	0.05
Na$_2$O	1.15	3.44	0.17	0.12
TiO$_2$	1.12	0.05	0.16	0.01
SO$_3$	----	0.02	0.14	0.02
P2O$_5$	0.51	----	----	----
Cr	----	0.02	0.06	0.08

5.1. Fillers

In practice, the filler of white ware bodies is always silica, either in the form of flint of sand though few bodies often used alumina. Fillers serve to open up the body facilitating the drying process, reducing the drying shrinkage and plasticity. Moreover, they are controlling the thermal expansion and contributing the whiteness of the fired bodies. Fillers are often representing a large ratio of the total body composition. So, the particle size distribution is very important in the determination of the particle packing in the body. This is in turn affects the drying and firing shrinkage. Therefore, the fineness of the filler affects to a large extent the thermal expansion of the fired body. Fillers are high melting, chemically resistant inorganic materials whose main function is to reduce a ceramic body's tendency to warp or distort when fired to high degrees of temperatures that results in the formation of glass in the body. They also play an important part in determining the thermal expansion of the fired body. Free silica in the form of quartz, added as a ground sand or flint, is by far the most common filler used in white wares and many other ceramic bodies. Though alumina is an important ingredient in many refractory and technical ceramic bodies, it is in a few tableware bodies [7,16-22].

5.2. Quartz sand

Quartz sand or silica is the most abundant oxide in the earth's crust. It is estimated that silica occupies approximately 19 wt. % of the outer 25 miles of the earth. White wares often contain silica in the form of silicates, i.e. silica can be combined with other elements to form the silicate minerals, but a large proportion occurs as free silica, mostly in the form of quartz. Since this is the mineral form which is stable under the normal atmospheric conditions. Earthen wares for an example are containing 72-76 % silica. In many countries like UK, flint is the popular form of silica component of white wares, though the continental countries have used quartz sand for many years. Recently, however and mainly for economic purposes, many British manufacturers have taken to the continental practice and replaced flint with sand in their formulations. Sandstones are common rocks, but the sandstone pure enough to use in white ware bodies are infrequent. Many contain iron compounds which stain them brown or red colors [6,10,17,19]. Generally, quartz exists in two forms differing slightly in density. Below 573 ºC, β-quartz, the denser form, is the stable one, while above this temperature, -quartz is stable. The change between the two occurs rapidly as the temperature changes through 573 ℃ and since only a comparatively small displacement in the relative positions of the SO$_4$ tetrahedra is removed. This type of change between high and low temperature modifications of silica is referred to as a displacive change. When quartz is subjected to temperature

above approximately 1000 ℃, bonds between atoms are broken and a new crystal structure results. The mineral cristobalite has such lower density than quartz. Since the change involves a major reconstruction of the crystal. Cristobalite also exists in - and modifications [5,6,9,10,17]. The transformation of quartz to cristobalite is slow and dependent on the temperature and time of firing. Also, on the presence of mineralizers which promote the change. The amount of formed cristobalite for a particular firing temperature depends largely on the size of the present quartz particles. The general scheme [5,6,23] of the polymorphous transformation of quartz at different temperatures may be represented as follows:

$$\alpha\text{-Quartsz} \leftrightarrow 573\,^o\text{C} \leftrightarrow \beta\text{-Quartz} \tag{1}$$

$$\beta\text{-Quartz} \leftrightarrow 870\,^o\text{C} \leftrightarrow \beta_2\text{-Tridymite} \tag{2}$$

$$\beta_2\text{-Tridymite} \leftrightarrow 1410\,^o\text{C} \leftrightarrow \beta\text{-Cristobalite} \tag{3}$$

$$\beta\text{-Cristobalite} \leftrightarrow 1220\text{-}1280\,^o\text{C} \leftrightarrow \alpha\text{-Cristobalite} \tag{4}$$

$$\beta_2\text{-Tridymite} \leftrightarrow 1163\,^o\text{C} \leftrightarrow \beta_1\text{-Tridymite} \tag{5}$$

$$B_1\text{-Tridymite} \leftrightarrow 1170\,^o\text{C} \leftrightarrow \alpha\text{-Tridymite} \tag{6}$$

5.3. Flints

Flints contain small amounts of water, minute air spaces and interstitial amorphous silica due to its mode of formation from the skeletons of sponge-like organisms which dissolved in sea water and were later deposited in chalky deposits. The fine microstructure of flints makes it more reactive than quartz and in particular it converts during firing more readily to cristobalite (another crystalline form of silica) which is profoundly affects the thermal expansion of the body [5,20]. Flint occurs as hard nodules in the middle and upper parts of the chalk deposits often arranged in layers along bedding planes separating the beds rather than evenly distributed throughout the chalk. Therefore, flints were formed by precipitation from silica in solution. This is confirmed by the fact that the white skin of flint gradually varies in composition from $CaCO_3$ to silica. In order to prepare flints for use, flints pebbled from the sea shore or from chalk deposits are calcined up to 1100 ℃ to make it friable, to facilitate its grinding to the required fineness (45-50 % less than 10 μ). During calcinations, flints easily shatter due to expansion of air and water pockets within the structure. This changes its color from black to white as an organic material is burnt off. The specific gravity of flint is 2.62 reduced to 2.50 after calcination due to the exfoliation of the flint on heating and to a lesser extent to the formation of cristobalite (spec. gr. 2.3). About 18 % of quartz is converted to cristobalite during calcination process of flint. The calcined flint is wet ground to the required size and used in a slop form in the body mix. Grinding to the correct fineness is very important since it affects the extent of conversion of quartz to cristobalite during firing of the body [5,6,10,17]. Flints of interest to the potter are divided to Chalk flints which are generally large lumps with a thick chalk crust, wash mill flints which are by-products of the cement industry and by far most important source of free silica for earthenware and tiles and beach flints which are smooth flint pebbles that occur on a number of beaches at the southern coasts of England, and at the northern coasts of Belgium and France, where the surface chalk is completely washed away [5,6,9,10,17]. Flint is composed of extremely small crystals of quartz. The surface of each minute crystal is available for conversion. Consequently, more cristobalite is formed in a body in which flint is the

filler than in the same body in which the ground sand is substituted for flint. Therefore, flint is preferred in the earthenware industry. Since, the contraction which accompanies the change as the cristobalite cools is an important factor in preventing crazing [5,6,9,10,17]. They are added and usually used in ceramic tiles or any ceramic masses as fillers. It is a common accessory constituent in clays used for the production of tiles [24]. Quartz relics act as a source of flaws in ceramic products. To get over it, the grain size of the quartz should be greatly reduced. In this case, it participates readily in the liquid (or glassy) phase formation.

5.4. Fluxes

A flux is a material which reduces the fusion temperature of the materials or mixtures to which it is added. It is not strictly possible to divide material to fluxes and refractory materials, since whether or not a material acts as a flux depends not only on the material itself but also on the material to which it is added. It is good mention that as the alkali content increases in a fluxing material, its fluxing action increases. The F_2O_3 content of the fluxing material must be in its lowest content, or it will impart its color to the fired ceramic body. Also, the grain size of the fluxes is of great and vital importance [5-7,10,16,20-22]. Fluxes are constituents which melt when the ware is fired and cooled. The glass solidifies and provides the bond which holds the whole mass together. This accounts for most of the fired strength of the pottery ware. Cornish stone, feldspar and nepheline syenite are fluxes [10,18,25].

5.5. Feldspar

It is essentially a mixture of two feldspars namely, orthoclase-potash feldspar, $KAlSi_3O_8$ or $K_2O. Al_2O_3. 6SiO_2$ and albite-soda feldspar, $NaAlSi_3O_8$ or $Na_2O. Al_2O_3. 6SiO_2$. Small amounts of mica and quartz are usually present. The feldspar is often extracted by normal quarrying or mining with visual control over the extracted material at the quarry face. Briefly, the method starts by crushing the rock to less than one inch by means of jaw and gyratory crushers which followed by wet milling to pass a 30 mesh sieve. The three stage froth flotation process which follows, first removes the mica, garnet and other similar materials. Then, the feldspar is frothed from the quartz. Finally, the separated feldspar undergoes a further flotation to reduce the quartz as much as possible. The concentrated feldspar and quartz which are separated by this method are then dried and stored. Although the feldspar contains about 5 % of quartz and the quartz contains 6-8 %of feldspar [6,10,17,25]. The temperature at which the individual fluxes start to melt is largely depending on the total alkali content and the particle size of the material. Mica, particularly in the extremely finely divided form in which it occurs in the clays, is one of the first of glass-formers to melt. The outer fluxes all resemble one another at the lower temperature, e.g. pressed discs of Cornish stone, feldspar and nepheline syenite when fired up to 1100 ℃ have very much the same appearance-hard and with sharp well defined edges. As the temperature is raised, the expected variation becomes apparent with the nepheline syenite showing the greatest tendency to flow. So, increasing the amount of flux in a body will not necessarily reduce the fired porosity, particularly at relatively low temperatures [18,25]. Schuller and Jager [26] showed that the viscosity of feldspar melts increased with the rise in K_2O / Na_2O ratio. Schuller [11] found that soda feldspar seems to increase the reactivity of the melt and promotes the attack on the aggregates of primary mullite ($3A_2O_3.2SiO_2$ or A_3S_2) at a higher temperature. Table 2 illustrates the chemical analysis of some commercial fluxes.

Table 2. Comparative analyses of some commercial fluxes, wt. %.

	Feldspar	Cornish stone	Neph. Syen. 1	Neph. Syen. 2
SiO_2	67-70	72-74	61.0	56.0
TiO_2	67-70	0.05-0.15	0.0	0.1
Al_2O_3	16-18	14-16	23.0	25.0
Fe_2O_3	0.1-0.3	0.1-0.3	0.07	0.08
CaO	0.3-0.5	1.5-1.9	0.6	0.75
MgO	0.03-0.15	0.05-0.20	0.10	0.0
K_2O	9.0-11.0	4.0-4.5	4.5	9.2
Na_2O	2.5-3.3	3.0-4.0	10.0	8.1
LOI	0.4-0.7	1.0-2.0	0.6	0.75

5.6. Nepheline Syenite

It contains a high alkali ratio (9-10 %) and therefore it is considered as a powerful flux. So, it can be used in a body composition of glazes and porcelain enamels have increased considerably over the last 20 years. It is claimed that the substitution of nepheline syenite instead of feldspar in the ceramic body reduces the firing temperatures resulting in saving time and fuel. Also, it increases the firing ranges. There are two main sources for nepheline syenite in Canada known as "Lakefield variety" and in Norway known as "North Cape variety". The second variety has a total alkali content and K_2O/Na_2O ratio greater than those of the first. Therefore, the Norway variety has a higher fluxing action than the Canadian [7-9,16,20-22].

5.7. Cornish stone

It is clear from Table   that Cornish stone is not a powerful flux as either feldspar or nepheline syenite where its total content is considerably less, but it was however very widely used as a flux in this country being the only native deposit of fluxing material commercially viable and therefore its use has declined recently, and feldspars and nepheline syenite being imported to replace it. In fact, the Cornish stone is effectively a mixture of kaolin and feldspar. Generally, it is graded on the basis of its color. There are three Cornish stone varieties known as "Hard purple", "Mild purple "and "Dry white". All varieties contain feldspar, quartz, clay minerals, mica, fluorspar (CaF_2) and other minor impurities. The purple varieties are the richest in feldspar, while the white variety is the poorest. Hence, the hard purple is the strongest flux and the dry white is the weakest. The color is mainly due to the impurity content exist in the fluorspar of the stone. During firing, as the feldspar and fluxing ability increases, the fluorspar content increases too. This often accompanied with the emission of fluorine. This is usually detrimental to both environment and human health [7,16,17,20-22].

5.8. Talc

It is a magnesium silicate and a member of montmorillonite group having the formula of 3 MgO. 4 SiO_2. H_2O and it is a very cheap source of magnesia which is acting as a flux. The presence of Talc in bodies that fired at high temperatures results in the formation of cordierite. This imparts low thermal expansion and therefore good thermal shock resistance. This makes it to be more suitable for the production of table wares. The only disadvantage is the very narrow range of firing which makes it easily distort on firing. Furthermore, according to its lower expansion, they become more difficult to match with the glaze of a suitable expansion or decorate to an acceptable standard. It is believed that lime-bearing talc in particular gives bodies of low

shrinkage the high strength and good crazing resistance. They are successfully used in the manufacture of both table wares and wall tiles [5,7-9,16,20-22].

5.9. Clays

Generally, clay minerals are often formed from the decomposition of igneous rocks as granite which is composed mainly of roughly equal proportions of potash mica (K_2O. 3 Al_2O_3. 6SiO_2. 2H_2O), quartz (SiO_2) and potash feldspar (K_2O. Al_2O_3. 6SiO_2). Kaolinitic clay was the decomposition or kaolinization of feldspar in the presence of air and water [5,6,10,16,17] as follows:

$$K_2O.3Al_2O_3.6SiO_2 + 2H_2O \rightarrow Al_2O_3.2SiO_2.2H_2O + K_2O + 4SiO_2 \qquad (7)$$
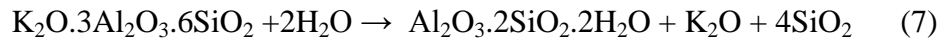
Kaolinite is a crystalline material in which the crystals are being flat and extremely small hexagonal in shape. The crystal size may vary from 5 μ to 1 μ 0r 10^{-4} meters which in turn responsible for their extreme properties [7-9,16,20-22]. It is well known that clays supply the plasticity or workability which simplifies the manufacture of clay wares and also account for the dry strength of the unfired article. So, materially assist in reducing handling losses. The properties of any particular clay are depending mainly on a number of factors including the type and amount of the clay minerals, the particle size and size distribution of the clay and the other minerals invariably present. When the clay is formed from the parent rock, it may have been deposited at its place of origin and hence, it is known as "Primary or residual clay", but if it is transported by means of water or winds or any other means and deposited into a distance away is known as "Secondary or sedimentary clay" [5,6,17]. At the temperature range 100-200 $°C$, the volume of certain argillaceous minerals shrinks as a result of the water loss which causes a dimensional change. At the temperature range 200-300 $°C$, the oxidation of certain organic materials present in argillaceous minerals begins. The degree of oxidation depends on the nature of the organic materials, the amount of oxygen available in the furnace and the readiness with which this can penetrate into the mass of the argillaceous material in order to promote the oxidation. In general, the degree of oxidation increases with rising temperatures [5,6,10]. The most important clay mineral for potters is kaolinite (Al_2 Si_2 O_5 $(OH)_4$ or Al_2O_3. 2SiO_2. 2H_2O or AS_2H_2), which is seen in the electron microscope to crystallize into minute hexagonal plates. The Kaolinite is a decomposition product of feldspar which starts to decompose in the temperature range 450-500 $°C$ to metakaolin (Al_2O_3. 2SiO_2 or AS_2) and water vapor [9,17].

$$Al_2O_3.2SiO_2.H_2O \rightarrow 450\text{-}500\ ^oC \rightarrow Al_2O_3.2SiO_2 + 2H_2O \qquad (8)$$
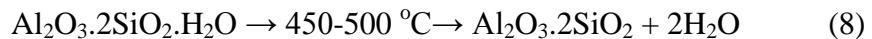
The reaction is accompanied by an expansion so that the ware at this step is larger than when it entered the kiln. At about 980 oC, a sudden evolution of heat accompanies the complete breakdown of the structure with the formation of mullite (3Al2O3. 2SiO2 or A3S2) and quartz minerals.

$$Al_2O_3.2SiO_2 \rightarrow 980\ ^oC \rightarrow 3Al_2O_3.2SiO_2 \qquad (9)$$
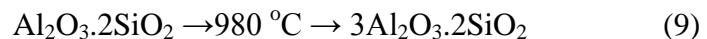
5.10. Limestone

It is an important ingredient in wall tiles. It is used in wall tile body composition because of its ability to produce bodies of very low moisture expansion which it is particularly very important in tiles, where the highly porous body would otherwise be prone to adsorbmoisture in use and expand, with the inevitable crazing of the glaze [6,10,17]. Lime is often used at about 10-12 wt. % addition in tile bodies reduces the

amount of glassy phase formed in firing. This is the feature of the body which is prone to moisture expansion. Its behavior during firing is more complex. So, it cannot be classified simply as a filler or flux. On firing, it is first starts to decompose to CaO and $CO_2\uparrow$ evolved as follows:

$$CaCO_3 \rightarrow 700\text{-}900 \, ^{o}C \rightarrow CaO + CO_3\uparrow \qquad (10)$$

Then the resulting CaO can react with other constituents to form anorthite-lime feldspar ($CaAl_2Si_2O_8$) rather than enter into the glassy phase. This reduction in glass content virtually eliminates firing shrinkage and the considerable problems which were associated with variations in size due to normal fluctuation in firing. Limestone (ground chalk or $CaCO_3$) which is commonly known as "Whiting" could also be used as an important glaze material [27]. Tiles [7,12] are generally classified according to their degree of water absorption (W.A.) into three main groups:-i- Low water absorption (W. A. < 3 %), ii- Medium water absorption (W. A. = 3-6 %) and iii- High water absorption (W. A. > 10 %). On the other hand, the use of granite or kaolinized granite for the manufacture of red tiles by rapid firing was done giving a firing temperature 40 ℃ lower than the best comparable clay body one [11,27]. In some compositions, the firing temperature was found to reach 1000 ℃ [28,29]. Belous; Kushel; Evplov and Ohkrimenco [30] studied the effect of both chemical and structure composition on the properties of floor tiles. They found that the presence of ZrO_2 had no significant effect, but the bending strength was increased by increasing the Al_2O_3 content and was lowered by more SiO2 content. The compressive strength reached a maximum value 3100 MPa in presence of 95 % Al_2O_3. Most of the traditional white wares ceramics specially floor tiles fall into the category of polycrystalline aggregate in a glassy matrix, e.g. multiphase body with phases having too different coefficients of thermal expansion which may tend to develop some defects. So, for bodies made of polycrystalline aggregate in a glassy matrix, the mechanical strength and the physical properties, i.e. the ceramic parameters, are greatly influenced by the stresses developed in the glassy phase as well as corroded relics of quartz grains and numerous small mullite crystals [31]. Quartz, which is one of the gradients in the floor tile composition, is characterized by a relatively higher thermal expansion than other components, that reaches a value of 26.2 x 10 -6 between room temperature and 1000 ℃ [32]. So, quartz controls the total thermal expansion of the tile body. Therefore, the extensive cracking is often result from the expansion changes accompanying the phase conversions of silica in the form of quartz, in addition to the difference in expansion between the glassy matrix and the quartz phase was proved in the work done by Genin [33], Kalnin and Coworkers [34,35] found that both young' s and their modulus as well as the strength of white wares were increased with the total amount of mullite which was also claimed by Mortel [36] in case of fast fired bodies and by Giamlem et al. [37] with the addition of anorthothite as a flux to rise the bending strength of the produced articles. Schamberg et al. [38] stated that the bending strength of the tiles can be increased with more thn 20 N/mm2 by preventing the formation of gehlenite compound by the addition of about 8 % barium compound in the tile composition and firing at 800-1200 ℃. On the other side, the strengthening of the glassy matrix of the tile bulk compositions was by increasing its Al_2O_3 and decreasing its K_2O contents [39-42], whereas the MgO, K_2O and Na_2O are factors governing the maturing temperature, liquid phase formation, crystallization of phases and as a result, the different properties developed.

In recent trends, to save raw materials and to reach good mechanical as well as ceramic properties of floor tiles and moreover to use both industrial byproducts and

wastes, the industrial waste materials containing oxides suitable for tile composition were introduced in the tile mix composition. The most important waste materials among this field of application are both fly ash and cement kiln dust. Aggarwal et al. [43] found that fly ash from a thermal power station and aluminium industry waste can be used in the manufacture of wall tile up to 42 wt.%. The bending strength of ceramic tiles was increased and their water absorption decreased by the addition of fly ashes and slags from coal-fired power plants. The sintering effect of slags was due to the increased amount of FeO and that of ashes to the amount of alkalies in the composition [44-48]. Pinalova et al. used the tailing from a coal benification for the manufacturing of ceramic materials. A typical waste 16-20 mm diameter contained 7-16 % moisture and 75-85 wt. % ash was examined. The waste was added to the ceramic batch and ground to a particle size 0.5 mm, mixed with water and plasticizers, and then pressed or extruded at 20-30 kg/cm^2. The products were dried, fired at 800-1110 ℃, and then optionally glazed at 980 ℃ to give tiles and facing materials. On the way, to increase the capacity of tile firing furnaces and decrease the energy consumption in tiles manufacture, silica flour was used in the ceramic batch composition to produce tiles glazed and fired by glost firing at 1050-1250 ℃ for 20-60 minutes only [49]. Also, tiles had 1.0-1.5 % shrinkage and 13.8-14.5 MPa bending strength were produces at the Slavyanshoe plant [50] by using the waste slurry obtained during the production of water glass. This waste has a high content of SiO_2 (66.67 %) and alkalies (Na_2O, 2.5-3.0 %) but a low content of Al_2O_3 (0.5-0.6 %). Also, to decrease volume and weight with retained strength and decrease the firing temperature of the tiles [51], the raw mixture of tiles contains wastes from asbestos cement production was used.

Some investigators [52,53] used mining tailings and metallurgical wastes in the production of ceramic floor tiles by dewatering, grinding, pressing and firing at 1000-1100 ℃. Mining tailings consist of argillaceous shale and sandstone, where the argillaceous shale serves as a binder, while sandstone as a flux substituted for pegmatite. Ceramic tiles of a low porosity and good mechanical properties were obtained from mixtures containing argillaceous shale 70-76, sandstone 15-20, chamotte 3-5 and bentonite 1-5 wt. % [54]. Nowadays, the use of Portland cement and electrostatic precipitated dust of cement kiln as a waste material which are the main sources of air pollution in many countries, which resemble the present day problem, be a matter of great importance due to the accumulation of large quantities of the kiln dust and its characteristics such as: light weight and fine particles so that it can be transported easily by air to many agriculture and population areas. Keijiro et al. [55] manufactured high strength ceramic tiles from slurry containing cement and mineral materials, e.g. feldspar, siliceous stone and SiO_2 by moulding the slurry followed by drying and firing. The cement is selected from Portland and/or white Portland cements. Abdel-Fattah et al. [56-58] studied the electrostatic precipitator dust collected from a cement kiln as received from the factory. The dust was fired between 1000-1250 ℃, then two calcines (70:30) and (50:50) were prepared by blending the raw dust and kaolin. It was stated that the dust consisted mainly of dolomitic limestone, minor amount of alkalies and quartz together with 2 $C_2S.CaCO_3$ and 2 $C_2S.CaSO_4$. The formed phases in the 1st calcine mix, was found to be mainly of gehlenite (C_2AS), but in the 2nd mix, it was mainly of β-C_2S. It is also very important to note that the use of CaO sources in the ceramic field and tiles production is recommended as a whiting and a flux [59]. The cement kiln dust is a mixture of finely divided particles and partially calcined raw materials with condensed volatile salts [54,55]. So, the use of dust along with kaolin in the form of calcined mix proved also

to be used successfully in replacing feldspar in porcelain bodies. Previous investigators [58,59] also reported the utilization of alkaline wastes collected from cement kilns to produce porcelaineous bodies. Due to the nature of the dust composition which contains mainly a high CaO content together with alkali oxides (Na_2O, K_2O) with a considerable part of the composition sometimes of FeO, the kiln dust always has powerful fluxing effects. So, it would be much suitable to use as a component of ceramic bodies. However, it must be recommended that the oxides present in the kiln dust are in the form of a complex silicate nature or a compound form. So, it can be expected that they would behave differentially from the corresponding free oxides in ceramic bodies, which would limit the possible use of this kiln dust. Therefore, the use of the cement kiln dust in the ceramic body batches, especially in the production and manufacture of floor tiles, need studies to utilize its role and to reach a good, a suitable and preferable conditions for producing tiles having maximum ceramic parameters characteristics [60,61]. The possibility to reuse the EPCKD waste as a source of CaO in the conventional ceramic industry was studied. He could be used about 20-25 wt. % of EPCKD waste to produce wall and floor tiles with good characteristics when compared with the control bodies. The physical properties in terms of water absorption, bulk density and apparent porosity, mechanical properties in terms of bending strength, thermal properties in terms of dry and firing shrinkage as well as thermal expansion were investigated (Figs. 3-5). Table 3 shows the dry and firing shrinkages of ceramic products containing different ratios of CKD at different firing temperatures. It is concluded that the addition of EPCKD waste improved and enhanced all the characteristics of the prepared ceramic bodies [61]. Darweesh and khalil [62] illustrated that the addition of 5-10 wt. % CKD waste to the Alumina cement improved the specific properties of the cement hydrated up to 28 days in terms of chemically combined water content, bulk density, apparent porosity, compressive strength, but the higher amounts of CKD content more than 10 wt. % reflected negatively on these properties (Figs. 6-8).

Table 3. *The dry and firing shrinkage of ceramic products with various CKD content at different firing temperatures.*

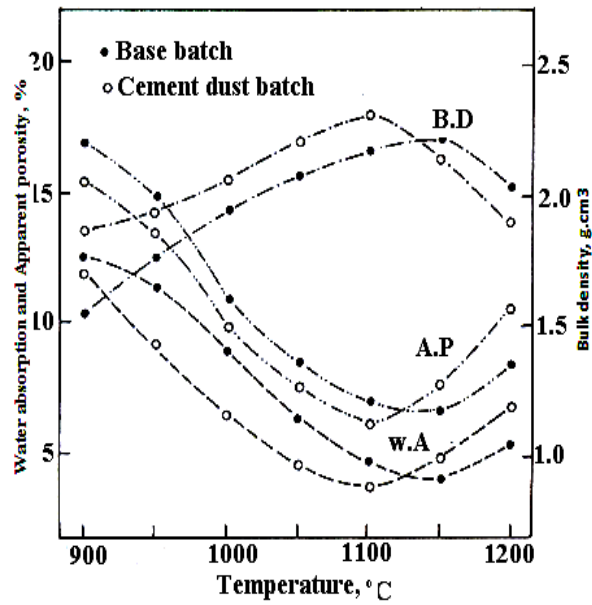|	CKD content, wt %					
	0	5	10	15	20	25
Firing temperature, ℃	0	0	0	0	0	0
	0	0	0	0	0	0
	1000	0.022	0.043	0.105	0.165	0.189	0.251
	1050	0.106	0.152	0.286	0.482	0.535	0.603
	1100	0.313	0.406	0.654	1.108	1.185	1.223
	1150	0.473	.518	0.654	1.312	1.342	1.363

Figure 3. *Water absorption, bulk density and apparent porosity of the ceramic bodied of the base batch and the batches containing CKD waste.*
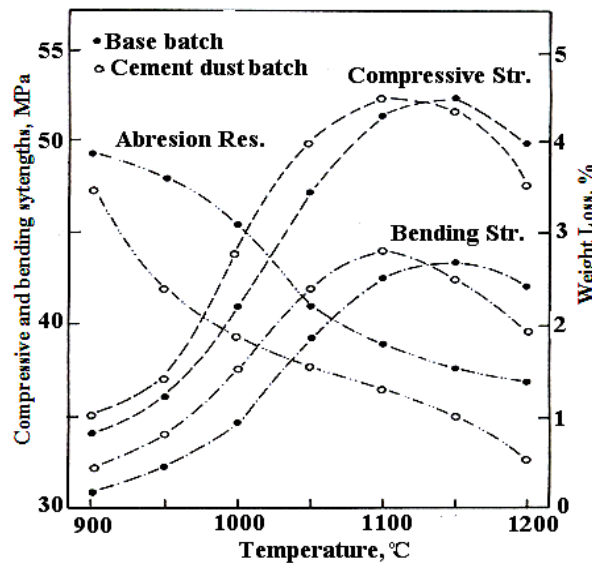

Figure 4. *Compressive and bending strengths as well as abrasion resistance of the base batch and the batches containing CKD waste.*
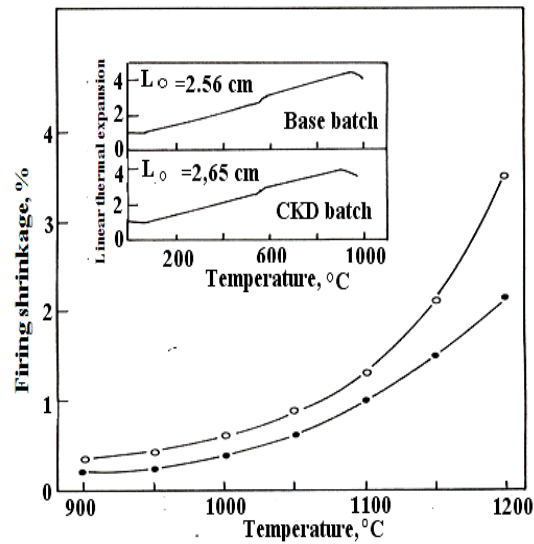
Figure 5. *Firing shrinkage and linear thermal expansion of the base batch and the batches containing CKD waste.*
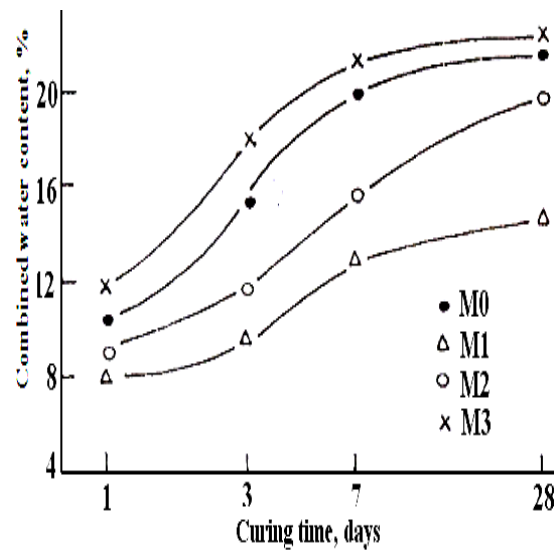

Figure 6. *Chemically-combined water content of the various alumina cement pastes containing cement kiln dust waste hydrated up to 28 days.*
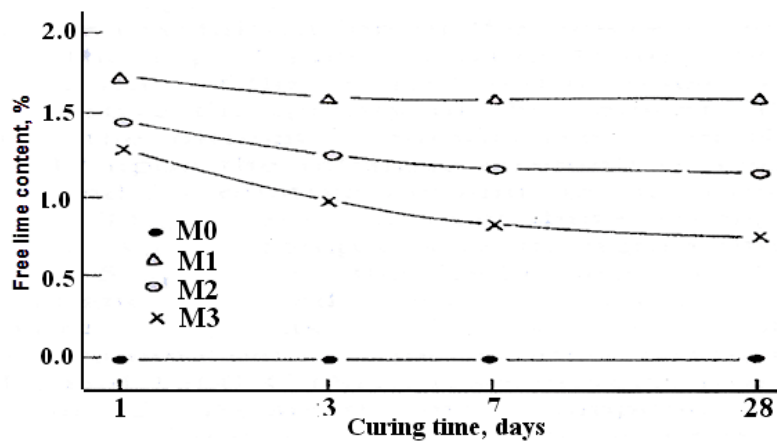
Figure 7. *Free lime content of the various alumina cement pastes containing cement kiln dust waste hydrated up to 28 days.*
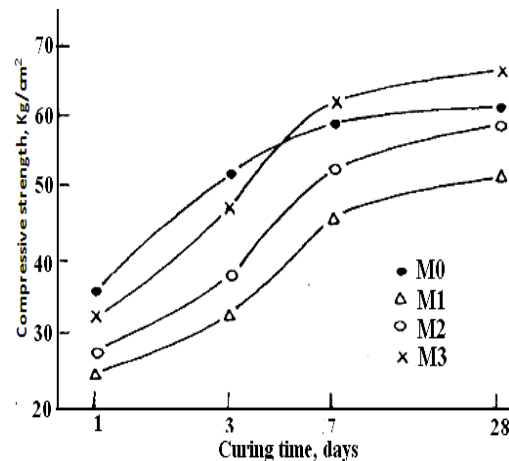
Figure 8. Compressive strength of the various alumina cement pastes containing cement kiln dust waste hydrated up to 28 days.

6. Methods of investigation

6.1. Densification Parameters

White ware products are known as "Vitreous" in which water absorption is less than 1 % or "Porous" in which water absorption is more than 1 % and may be reached to 18 % as in wall tiles. Generally, there are two types of pores, open and sealed pores. Open pores are voids which allow the ingress of a penetrating fluid from the surface of the article. The voids are created due the migration of gases during the drying and firing. Sealed pores are often formed during firing when bubbles of gases are frozen into the glassy matrix or when open pores are sealed by the molten material. Some clays and bodies are bloated when over fired due to the melting action of the fluxes together with the evolution of gases from such impurities as calcium sulphate, $CaSO_4$ [5-10,16,20-22,27]. Porosity is measured by comparing the volume of the pores to the volume or weight of the material itself. There two widely known expressions for porosity are "Apparent porosity" and "Water absorption". The Apparent porosity is the ratio of open pore volume to the total volume, whereas the water absorption is the ratio of the open pore volume to the weight of the test material as follows: The 1 cm diameter / 1 cm thickness disc-shaped specimens were prepared under 20-22 KN loading using a suitable piston. At first, the prepared specimens are left to dry on air for 24 hours and then at 105 ℃ for another 24 hours. The dried specimens are then fired at different firing temperatures using soaking time of 1-2 hours. The used furnace must leave to cool slowly over night. The fired ceramic bodies after cooling are then subjected to densification parameters [63,64], in terms of water absorption (W. A, %), bulk density (B. D, g/cm^3) and apparent porosity (A. P, %) which could be calculated from the following relationships:

$$W.A, \% = W1\text{-}W2/W3 \times 100 \qquad (11)$$

$$B.D., g/cm^3 = W3/ W1\text{-}W2 \qquad (12)$$

$$A.P., \% = W1\text{-}W3/W1\text{-}W2 \times 100 \qquad (13)$$

Where, W1, W2 and W3 are the saturated weight in air, suspended weight and the dry weight, respectively.

Darweesh [61] studied and discussed the water absorption, bulk density and apparent porosity of some ceramic batches containing different contents of CKD

waste up to 25 wt. %. Table 4 shows the chemical composition of the starting raw materials used in this study. They reported that the water absorption and apparent porosity decreased with firing temperature up to 1100 ℃, but only decreased with CKD content up to 10 wt. % and then decreased with further increase of CKD content as shown of the Figs. 9 and 10, respectively, while the bulk density increased as shown in Fig. 11.

Table 4 The chemical composition of raw materials, wt. %.

| Materials Oxides	T-Clay (TC)	Feldspar (F)	Sand (S)	Limestone (L)	Homra (H)	Leached C. Dust (LCD)
L.O.I	9.72	0.67	1.78	42.63	-	24.51
SiO_2	53.47	75.37	93.63	0.08	58.22	12.84
Al_2O_3	26.78	13.62	3.64	0.03	28.25	1.86
Fe_2O_3	3.99	0.41	0.08	0.04	8.16	1.53
CaO	0.60	0.53	0.18	56.84	0.79	52.51
MgO	1.38	---	---	0.10	0.46	1.84
MnO	0.03	0.03	0.02	---	---	---
K_2O	1.18	5.84	0.14	0.05	1.46	1.65
Na_2O	1.15	3.44	0.17	0.12	1.32	0.83
TiO_2	1.12	0.05	0.16	0.01	1.34	---
SO_3	---	0.02	0.14	0.02	---	2.43
P_2O_5	0.51	---	---	---	---	---
Cl^-	---	0.02	0.06	0.08	---	---
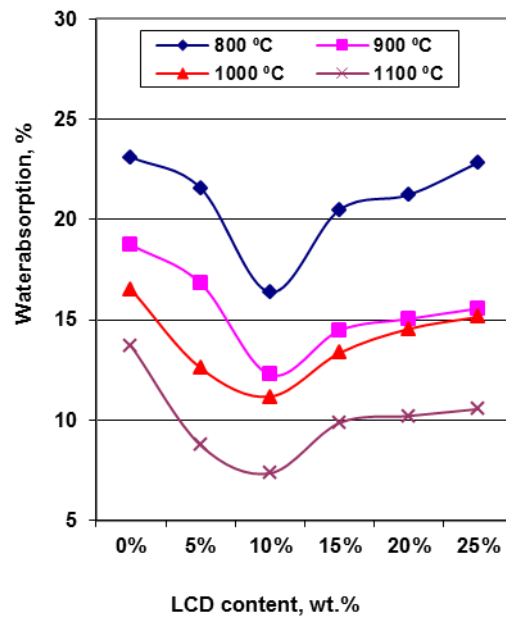
Figure 9. *Water absorption of ceramic products with different CKD contents up to 25 wt. %.*
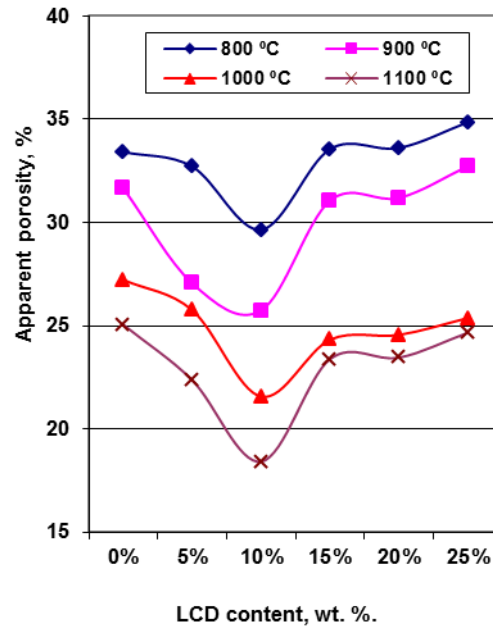
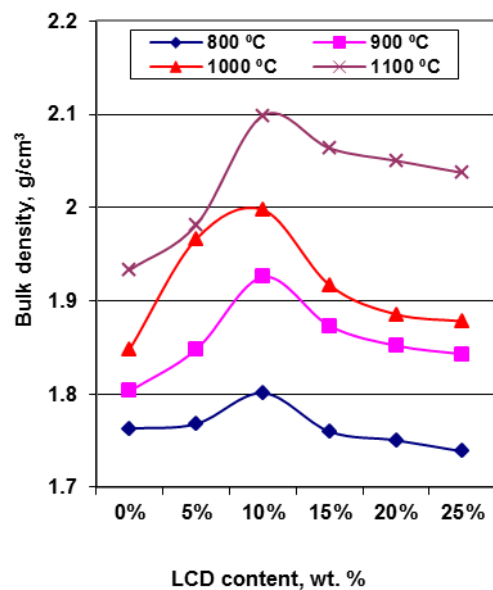**Figure 10.** *Apparent porosity of ceramic products with various LCD contents up to 25 wt. %.*

Figure 11. *Bulk density of ceramic products with various LCD contents up to 25 wt. %.*

6.2. Flexural or Bending Strength

Rod-shaped specimens of the dimensions 1 x 1 x 7 cm^3 are prepared using water as binder under 20-22 KN loading by a suitable piston. At first, the prepared specimens are left to dry on air for 24 hours and then at 105 ℃ for another 24 hours. The dried specimens are then fired at different firing temperatures using soaking time of 1-2 hours. The used furnace must leave to cool slowly over night. The fired rod-shaped specimens after cooling are then subjected to flexural or bending strength measurements [6,9,17] using the three point adjustments system (Fig. 12). The beam load was applied perpendicular to the axis of the sample. The flexural or bending strength [65-67] could be calculated from the following relation:

$$F.S., MPa = 3PL/(2bdx10.2) \qquad (14)$$

where, F.S: flexural strength, MPa, P: the load of rupture, kg, L: span or the distance between the two lower beams (5 cm), b: width of sample, cm and d: thickness of sample, cm.

Darweesh et. all [52] studied the bending or flexural strength of some ceramic batches containing different contents of CKD waste (Fig. 13). They found that the bending strength increases with firing temperature up to 1100 ℃, but only increased up to 10 wt. % CKD content and then decreased.
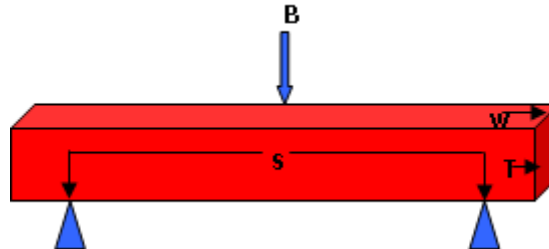

Figure 12. Schematic diagram of the bending strength, B: beam, S: span, W: width and T: thickness.
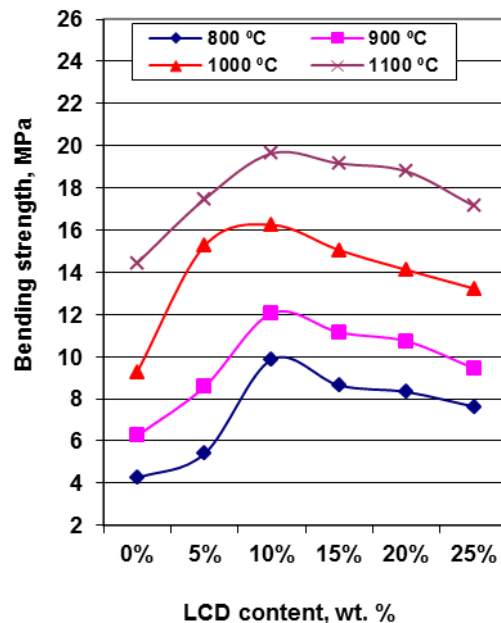

Figure 13. Flexural or bending strength of ceramic products with different contents of CKD waste, %.

6.3. Body glaze

Generally, the glaze is glass layer covered the ceramic vitreous material which is a super-cooled liquid below the point at which it might have crystallized. It is a liquid of very high viscosity due to the facts that A glass rod ultimately deformed and bends when suspended between two supports and on heating, glass softens and liquefies over a temperature range of , but does not exhibit a definite melting point. Normal solids are crystalline and have a definite arrangement of atoms and molecules. This can be shown by X-ray diffraction patterns. Glazes however, have no a definite structure, the atoms are oriented in a random network. This may be appreciated by comparing the structure of crystalline silicate minerals with glazes [7,66,68]. The unit of structure is the silicon tetrahedron this shape is dictated by the size of the atoms, on

a comparative scale oxygen = 1.32 and silicon = 0.39. An example of the Structure of a crystalline silicate and a silicate glass is shown in Fig. 14 which clearly indicates the difference in their structures. They have a random three-dimentional network but no units repeat it selves at fixed and regular intervals. On heating the glaze always softens over a given temperature range due to the varying amounts of energy required to detach different parts of the network which are not structurally equivalent. The interstices within the network may be filled with atoms of other elements which modify the physical properties as color, brilliance and so on. Glazes could be classified to:

A- Lead and Leadless glazes: Lead glazes have a brilliant appearance and con be used to glost firing temperature of 1150 °C above which the lead tends to volatilize while Leadless glazes have been developed and improved. Now, they have a reasonable brilliancy and firing range. However, glazes that mature below 1000 °C often have poor craze resistance. This is mainly due to the fact that high proportions of soda and potash required to give the glaze a low melting point also confer a relatively high expansion but the glaze must have a lower thermal expansion the ceramic body.

B- B- Fritted and raw glazes: A fritted glaze often has one or more material used in its formulation which have been subjected to the fritting operation, i.e. the heating together of components to form a glass which is subsequently ground to a given particle size.

C- Figure 13-Flexural or bending strength of ceramic products with different contents of CKD waste, %.

For a common practice, it often used two frits, i.e. Lead and Borax frits or oxides (PbO and B_2O_3). When these oxides are fitted together, the lead does not generally reach satisfactory limits of insolubility. However, lead borosilicate can be produced satisfactory if the $PbO:B_2O_3$ ratio is carefully selected [6,7,17,66]. The glaze could be classified also due to the type of ware on which the glazes applied as for Earthern wares, Sanitary wares and Porcelain glazes. There is a wide range of glazes available within each group. This classification has been broadened to include firing and maturing temperatures noticing that in the following examples, glaze molecular formulae have been given, where the oxides have been categorized as basic, amphoteric, acidic. The sum of the basic oxide is equal to unity, where the temperatures are referred to the maturing range [5,7-10,16].

1- Majolica (900-1050 °C): [0.7 PbO +0.3 CaO] +0.15 Al_2O_3 + [2.0 SiO_2 + 0.3 B_2O_3].

2- Earthenware (1000-1150 C-Lead glaze; [0.4 PbO+0.3 CaO+0.3(Na,K)$_2$O] 0.25 Al_2O_3 [2.5 SiO_2+0.5 B_2O_3].

3- Leadless glaze: [0.55 CaO 0.3 (Na,K)$_2$O] 0.30 Al_2O_3 [3.0 SiO_2 + 0.8 B_2O_3].

4- Sanitaryware (1200-1250 °C): [0.6 CaO+0.2 (Na,K)$_2$O+0.2 ZnO] 0.35 Al_2O_3 [3.0 SiO_2].

5- Hard paste porcelain (1400 °C): [0.68 CaO+0.20 MgO+0.12 ZnO] 1.0 Al_2O_3 [10.0 SiO_2].

There is a wide range of commercially coloring agents or stains which are used extensively in the ceramic industry. White wares may be decorated using coloring oxides in different ways as: Colored bodies: Colored slips (engobes), on glaze, Under-

glaze and in glaze. Colored bodies and slips have a few percent of the desired color added to the bulk mix during preparation in the slip house. The major disadvantage of using colored bodies is that shading of wares may take place. A ceramic color seldom consists of simply a coloring oxide or compound. This fact is understood by considering the following cobalt blue color known as "Royal blue" or "Maz blue" [5,9,16]. Cobalt oxide 45 %, Whiting 10 %, Flint 18 %, Alumina 5%, Feldspar 22 %. The color may be analyzed in terms of the function of its components. Cobalt is the basic coloring compound giving the intense blue color. Whiting acts as a modifying agent that modified the blue color to give special blue tint and stability. Flint and alumina act as diluents, i.e. as filler giving a slightly weaker but more stable color. Feldspar which acts as a flux is helping in the sintering of the color components during the calcination process noticing that the und glazed colors need about 5 % flux, but on glaze colors need about 70 %. Ceramic colors are usually prepared to fix and carefully control specifications even so, it is difficult to produce decorative wares of consistent quality on a commercial scale. It must always remember that the final color depends mainly on the composition of the glaze, firing temperature and kiln type. These are very important particularly in sanitary ware industry, where the finished product can be sold to an exact standard color while vitreous sanitary ware is generally colored with opaque pastel glazes which have to match other colored fittings, in the cloak or path rooms manufactured from quite different materials. The metamerism phenomena must be taken into consideration which is two color samples may appear to match, under a given illumination, even though their spectrophotometric curves are not the same (Fig. 15). So, the sanitary wares colored glazes are formulated from a color and compatible base glaze to an exact specification. If a different illuminant is used, the samples may no longer match where the perceived color depends essentially on the illuminant and the reflection curve at the surface [5,6,10,17]. The color specification by CIE system gives the chromaticity values or coordinates x, y and Y where these values may be used in conjunction with the CIE chromaticity diagram (Fig. 16).
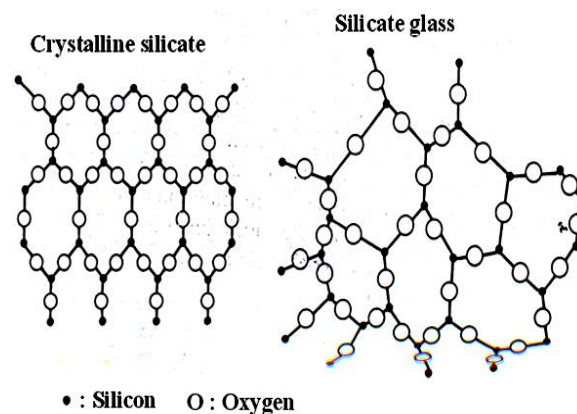

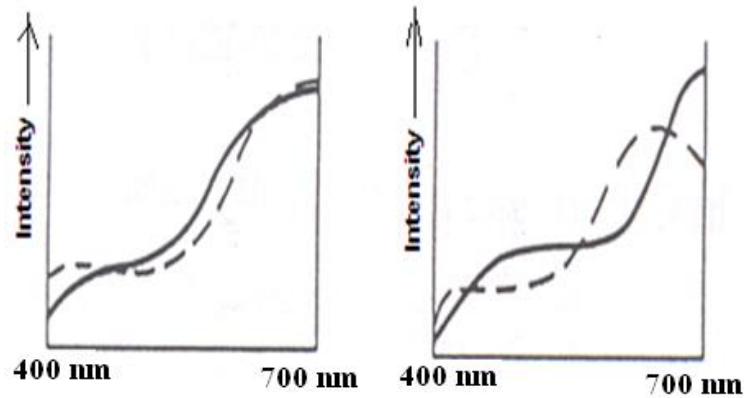**Figure 14.** *Structure of crystalline silicate and silicate glass.*

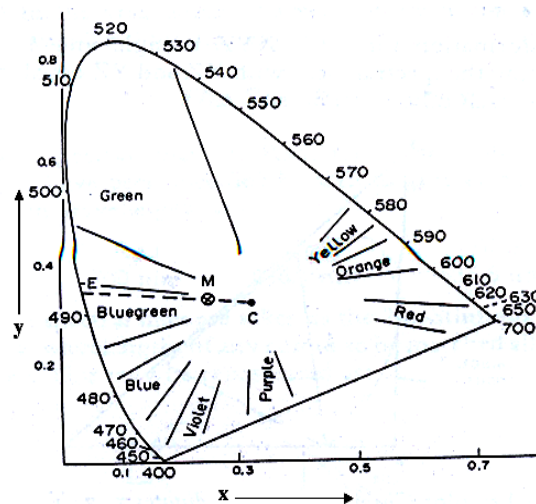**Figure 15.** *The metamerism curves of ceramic products.*

Figure 16. *The CIE chromaticity diagram.*

6.4. Thermal expansion

The difference between the contraction of the ceramic body and the glaze during cooling often causes some faults. The thermal behavior or expansion of ceramic products during firing can be measured by using "Ortom Automatic Dilatometer". In this type of instruments, the temperature can be controlled in the range of 0.2-10 ℃/min. and the rate of heating is 3-5 ℃/min. Darweesh [16] investigated the thermal behavior or expansion of some ceramic composites containing 0, 5, 10, 15, 20 and 25 wt. % cement kiln dust waste (CKD) using the "Orton Automatic Dilatometer" to throw light on the behavior of the samples on heating. Table 5 and Fig. 17 illustrate the coefficient of linear thermal expansion as a function of CKD content and its effect on the thermal expansion of ceramic products containing it. He found that the coefficient of linear thermal expansion decreased with the increase of CKD content up to 25 wt. %. The equipment (Fig. 18) in its fully automated form is a direct reading dilatometer which plots the percentage of expansion against temperature. Specimens between from 55 to 80 mm can be accommodated. The silica correction is added automatically and after the initial setting up the instrument requires no supervision. The Dilatometer is also available in a hand-operated form and the following test procedures relate to this less sophisticated instrument. The test piece with parallel ends is preheated to 900 ℃, cooled and then pushed along the silica tube using thrust rod till it contacts the silica stopping disc. The dial gauge is firmly clamped into

position and the needle adjusted so that a slight pressure is exerted on the thrust rod. The outer ring on the dial gauge is moved to zero reading. The thrust rod should move quite freely within the silica tube. The base of the apparatus should be gently tapped till the dial gauge gives a consistent zero reading. Fig. 19 shows the typical thermal expansion of earthenware body and low sol glaze, where at 500 ℃ the thermal expansion is 0.38 % and 0.32 % for body and glaze, respectively. The difference of expansion is 0.06 %.

Table 5. The coefficient of linear thermal expansion as a function of CKD content and its effect on the thermal expansion of ceramic products.

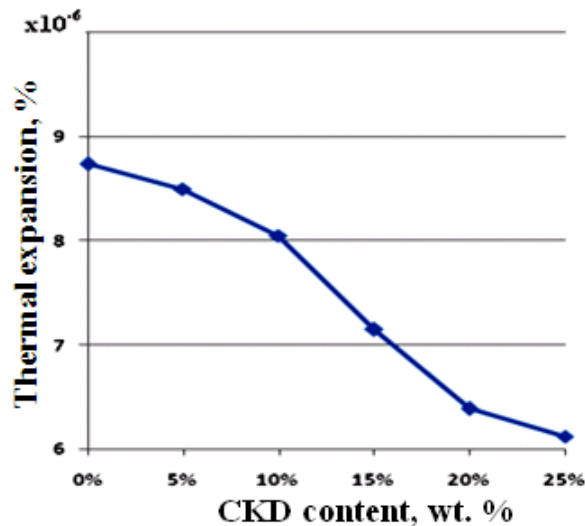| CKD, wt. %	Coeffecient of linear thermal expansion, α/k
0	8.731×10^{-6}
5	8.492×10^{-6}
10	8.049×10^{-6}
15	7.145×10^{-6}
20	6.382×10^{-6}
25	6.120×10^{-6}

Figure 17. The coefficient of linear thermal expansion as a function of CKD content and its effect on the thermal expansion of ceramic products.
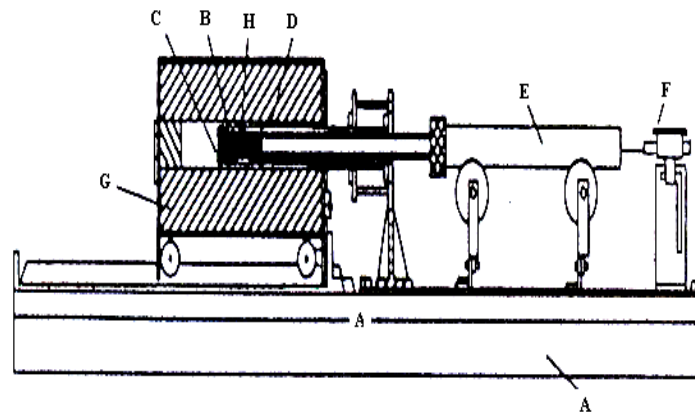

Figure 18. Thermal expansion apparatus (A: Slate base, B: Silica tube, C: Stopping disc, D: Fused silica rod, E: Invar extension rod, F: Dial gauge, G: Furnace, H: Test specimen.
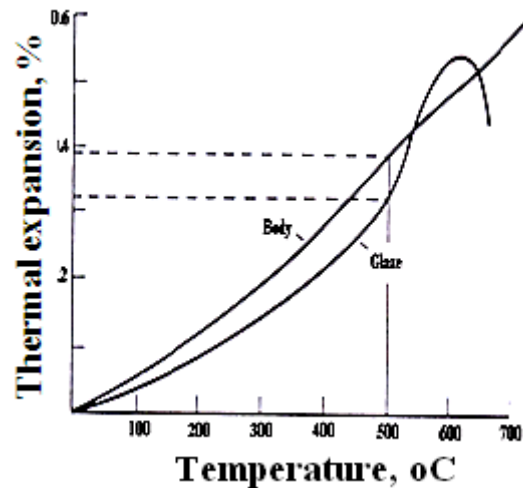
Figure 19. Thermal expansions of earthen ware body and glaze.

7. Conclusion

From the current review article it could be concluded that using of nanoparticles of ceramic materials as clay, limestone, quartz, feldspar and others achieved better results specially the mechanical strength like tensile and bending strengths than those of the normal or traditional particles. The crystals of the resulting ceramic units or products are sharp and well developed. The morphology or the external appearance of the fired units is well defined.

Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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