A STUDY ON THE EFFECT OF GRANULATED POWDER ON BENDING STRENGTH (MOR) & WATER ABSORPTION OF "MONOCOTTURA" FLOOR TILE

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1. ABSTRACT

In order to study how granule size distribution affects bending strength and water absorption of monocottura tiles, a series of floor tile bodies with different granule fractions were examined through powder compression. The powder used in making body biscuits was obtained from a given formula at fixed level of moisture by a spray dryer. It was then formed through powder compression at a fixed level of pressure. The examination of bending strength and water absorption in the monocottura tiles indicate that each range of powder sizes can be significantly effective on creating certain properties in the final body. Moreover, with regard to tile bodies formed at different ratios of various powders, it was established that a variety of granulations can be employed to achieve desirable properties in the final tile body. Relocation and motion of powder particles during formation of a tile body can take place through the driving force of compression, which is regarded as a major factor contributing to compaction in a biscuit body. Increased compaction in a biscuit is accompanied by reduced porosity and extended grain boundary, which in turn remarkably contributes to better sintering in the firing process and ultimately enhanced bending strength and reduced water absorption.

2. INTRODUCTION

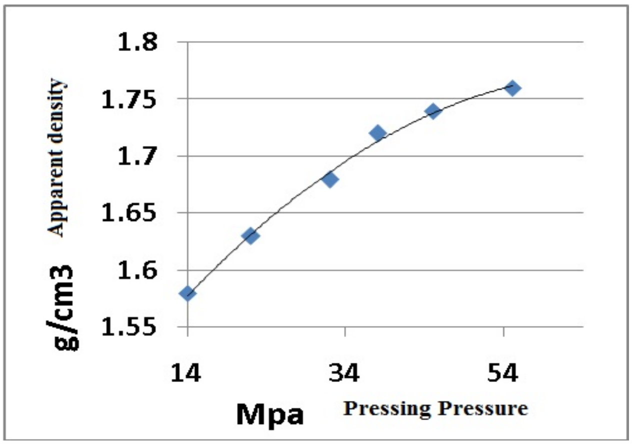
Although specifications of the final product greatly depend on intrinsic characteristics of the raw material, the production process can play an essential role in creating the desired properties too [12]. Throughout the tile manufacturing process, compaction is achieved in a body through compressing the powder obtained from spray dryer, while pressure acts as a conglomerator, packing the powder particles together [5,6]. With regard to the importance of the behaviour shown by various powder sizes during compression and also the characteristic of each granule size in the end product, it is necessary to investigate the effect of these two parameters individually on the bending strength and water absorption of tile body.

2.1. POWDER BEHAVIOUR AT VARIOUS SIZES IN COMPACTION

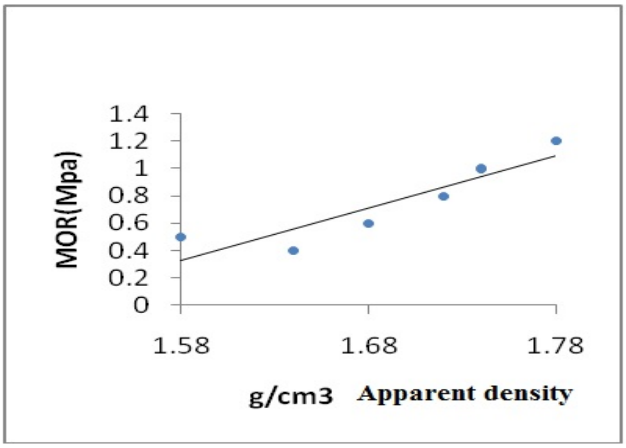
When powder in a variety of grain sizes is put under compression at the initial stage, where it has not been crushed yet, there is the possibility to quickly rearrange powder grains in order to fill up the porosities among the granules. This stage is the first phase to increase the density in a green body [2,9]. In fact, compression leads to relocation of particles inside the system, so that small-sized particles move in between larger ones, this action, leads the green body to increasing density. If we consider that, small powder particles tend to diffuse into the void of a packing of mixed sizes as pressure is applied, then according to equation (1) this diffusion would depend on average particle size (d_A), pressure (P) and compact density (P_B):

$$\text{Eq.(1): } D_{AB} \approx d_A(P/\ P_B)^{1/2}$$

Eq.(1) shows the particle size and pressure as the factors which increase the diffusion and cause further density in the tile body, while the compression in the body structure reduces the movement of particles within the body structure. Therefore, the greater the diffusion of smaller particles into the space among of larger particles, the more compression is enhanced, increasing the density of the biscuit [8]. Increased density contributes to reduced porosity and ultimately raised level of bending strength [4,11]. Hence, in the case of applying a variety of powder sizes, the density will be increased in the formed body. As for Eq. (1), there are two points to be mentioned regarding typical diffusion: 1) Instead of a transport of particular molecules, the transport of numerous molecules takes place. In fact, mass conduction occurs. 2) The driving force of this diffusion is the pressure gradient rather than the density gradient [8].The role of pressure in raising density is considerable at the first stage of formation, but as particles are crushed and body compaction is increased, this parameter becomes less significant in raising the density, as shown in curve (1-A). Curve (1-B) shows the correlation between bending strength and apparent density in a pressed body. As can be seen, the modulus of rupture (MOR) increases linearly when the apparent density increases. [5,6,7,9].



CURVE 1-A



CURVE 1-B

2.2. EFFECT OF POWDER COMPACTION ON TILE BENDING STRENGTH AND WATER ABSORPTION

The effect of compaction in a ceramic body on bending strength and water absorption can be found in the impact of porosities and grain boundaries during sintering. Accordingly, increased compaction leads to extended grain boundaries. Extending grain boundaries helps to improve sintering, while the porosities are the factors of reducing the grain boundaries and this, in turn, the barriers to grain growth during firing. Furthermore, the number of open pores in a ceramic body affects its water absorption. As a ceramic body is sintered, some changes occur in the formed particles and body porosities. The changes that occur during the firing process are related: 1- Changes in grain size and shape (grain growth); 2- Changes in pore shape; 3- Changes in pore size. The driving force which encourages the particles to join together and grow is the particles' tendencies for reducing the surface energy. Meanwhile, some particles disappear by combining to form larger particles as surrounding cavities get shrunk away. Such a phenomenon is conducive to body shrinkage throughout the firing process. Figures (1-A,B) display schematically the initial stages of sintering within three particles across a common boundary, where a connecting zone is first created, which is called a neck.

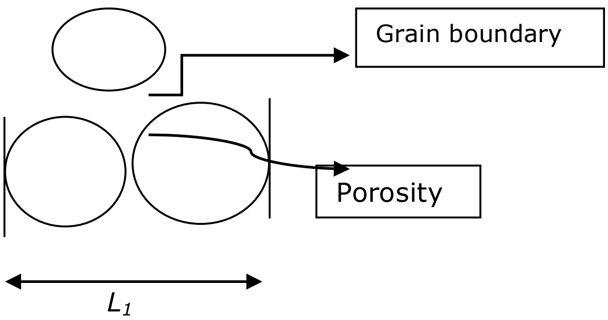


Figure 1-A

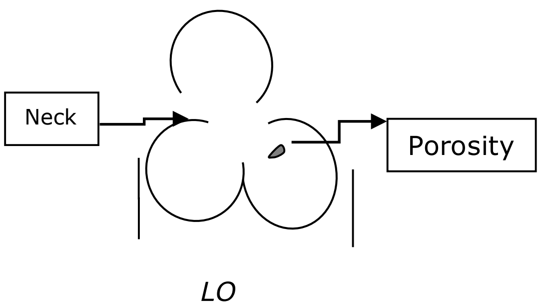


Figure 1-B

As may be observed above, the common boundary between particles plays a key role for initiation of sintering and grain growth. In other words, as the body compaction increases, common boundaries extend, which in turn leads to improved sintering and reduced porosity [10]. In bodies such as porcelain tiles, the high mechanical strength and very low rate of water absorption (about 0.5%) are the result of low true porosity. Due to the effect of porosities in reducing the bending strength and increased water absorption, the amount of porosity is of great importance in the biscuit, and the growth of pores during sintering depends on pore size in the green body. Therefore, the rate of compaction in the green body affects the degree of densification of the fired product. [3]

2.3. CHARACTERISTICS OF EACH POWDER SIZE

The powder produced by a spray dryer is spherical, the granules containing a single spherical cavity that connects to the external surface by a channel. The volume of a cavity depends on granule size. The bigger granules display a higher ratio of large internal cavity size to granule size. Figure 2-A shows a typical coarse granule and Figure 2-B displays a typical fine granule [1].

Figure 2-A

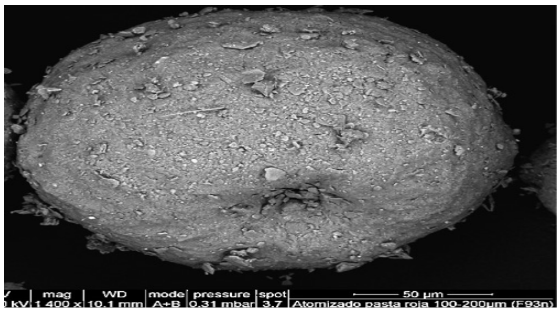


Figure 2-B

Granule mechanical strength in various sizes is different. The mechanical strength of granules can be calculated according to equation 2:

Equation 2:

$$\delta_{fG} = \frac{2.8\, F_{fG}}{\pi\, D^2_G}$$

Where F_{fG} stands for breaking load and D_G is granule diameter. This equation shows that the mechanical strength in different granule sizes is not the same, and it is due to the difference in the size of granule internal cavity for various powder sizes. In fact, the size of a larger cavity leads to less mechanical strength [1]. Smaller granules show a higher level of resistance against crushing, and this leads to greater compression resistance and prevents from reducing the amount of porosity in a green body [12].

3. EXPERIMENTAL ACTIVITIES:

This study investigates the effect of particle size distribution on the body compact by controlling the press pressure, powder humidity and firing conditions. The chemical analysis of the tested body formula was determined, as shown in the following table:

SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	Na_2O	MgO	L.o.I
63.87	19.93	3.12	1.22	4.3	2.09	0.17	5.3

The body was prepared in an industrial plant by wet grinding in a discontinuous ball mill. The density of suspension (1.51 g/cm^3) and 6 g residue was registered on a 200-mesh sieve. The powder under study was obtained from a SITI spray dryer with evaporation capacity of 3500 l/h and the powder formation was carried out by a press (Laies 600 compressor at 260 kg/cm^2 pressure) with 5%±0.2 moisture. For each given granulation, there were 3 prepared biscuits which had been dried up for 50 minutes in dryer at 110°C. The samples were fired in one line of roller kiln (to eliminate the temperature variable within the width of kiln) at 1160$^{°C}$ and cycle of 50 minutes. The measurement of water absorption and bending strength was conducted according to EN-100 and EN-99 standards.

The specifications of powder were determined as follows:

1) The powder after passing through a 20 mesh sieve, residue on a 35 mesh sieve, figure 3 (A), density of powder: 0.848 (g/cm^3)
2) The powder after passing through a 35 mesh sieve, residue on a 60 mesh sieve, figure 3 (B), density of powder: 0.876 (g/cm^3)
3) The powder after passing through a 60 mesh sieve, residue on an 80 mesh sieve, figure 3(C), density of powder: 0.874 (g/cm^3)

Figure 3-A

Figure 3-B

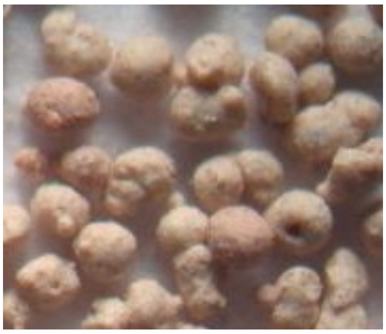


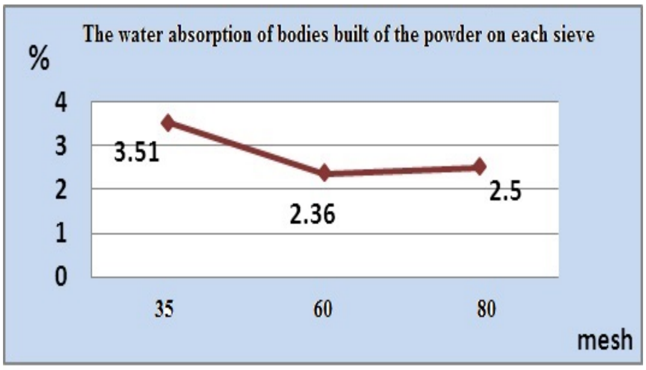
Figure 3-C

(Figures; A to C: Magnification of 30)

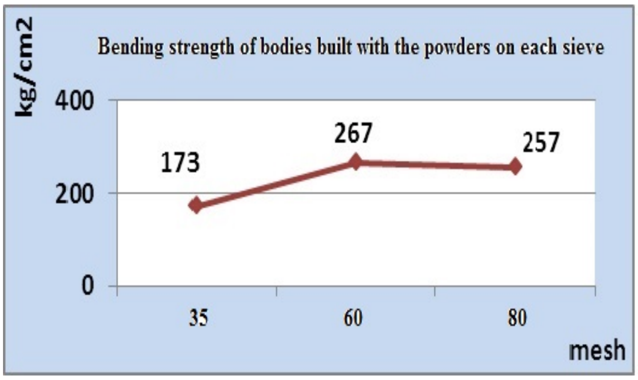
As shown in figure 3(A), the powder consists of granules stuck together with different sizes. In figures 3(B) and 3(C), as the powders become smaller, the real size of the granules becomes more recognizable.

4. INVESTIGATING THE MANUFACTURED BODIES

In the first stage, the bodies which were made of the powder that remained on each sieve were tested, and water absorption and bending strength were determined.



Curve 2

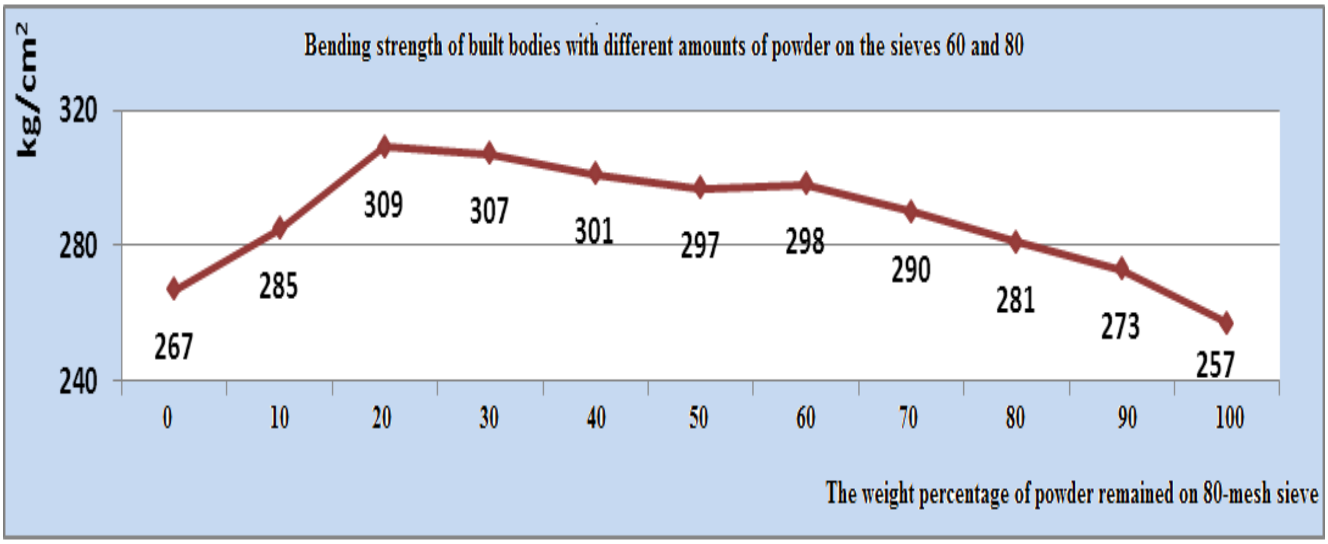


Curve 3

5. INVESTIGATING THE BODIES MADE OF DIFFERENT POWDER SIZES

5.1. INVESTIGATING THE BODIES MADE FROM POWDERS RETAINED ON 60 & 80 SIEVES

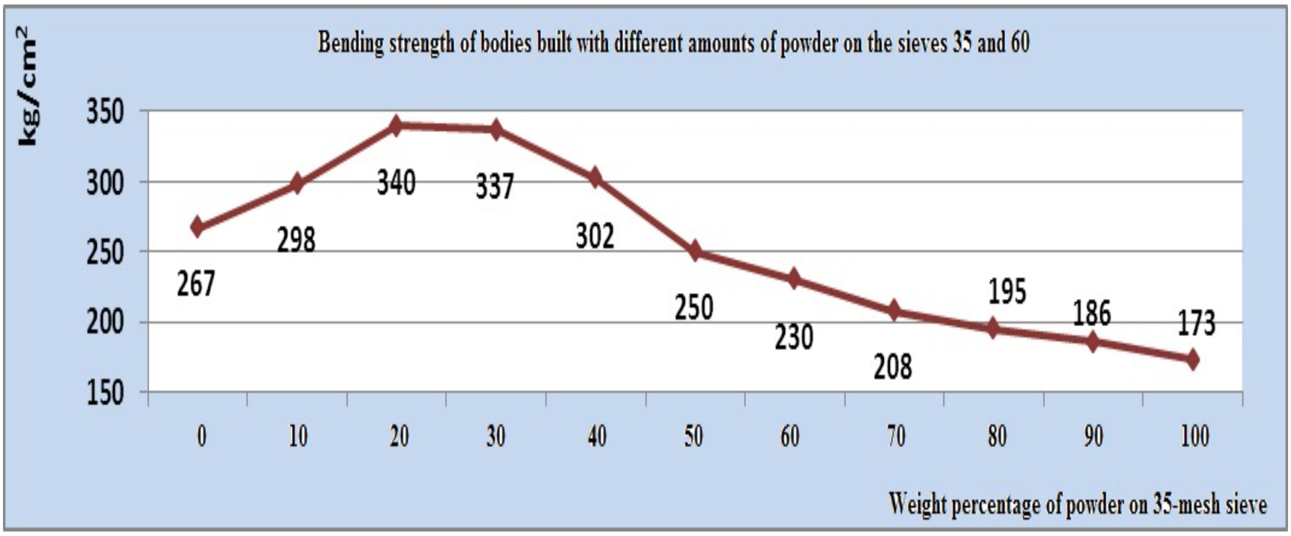
The bending strength of bodies pressed using mixtures of powders retained on 60 and 80 sieves at different ratios is shown in curve 4.



Curve 4

5.2. INVESTIGATING THE BODIES MADE FROM POWDERS RETAINED ON 60 & 35 SIEVES

Bodies formed by pressing, using mixtures of powders retained on 35 and 60 sieves at different ratios, were examined and the results of their bending strength are shown in curve 5.



Curve 5

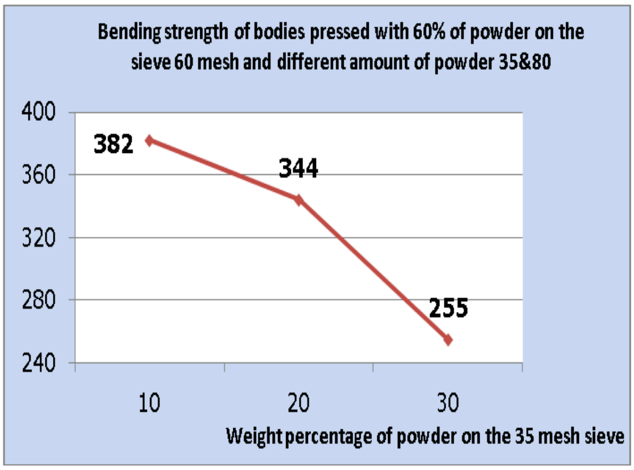
5.3. MANUFACTURING BODY WITH MIXED POWDERS REMAINED ON SIEVES 35, 60, 80

Curve 4 indicates that the level of bending strength is at a maximum regarding the percentage of powder retained on an 80 sieve within the application range of 20-30 and the percentage of powder retained on a 60 sieve within the application range of approximately 70-80. As for the binary system in Curve 5, the highest level of bending strength can be observed regarding the percentage of powder retained on a 35 sieve within the application range of 20-30 and the percentage of powder retained on a 60 sieve within the application range of approximately 70-80.

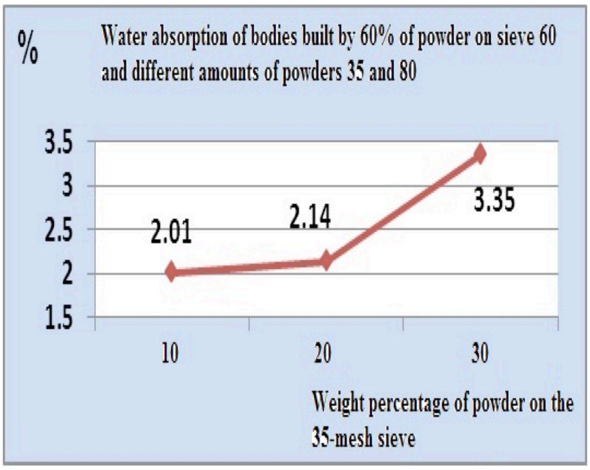
In accordance with the results, this experiment was done in a dual system by keeping constant the amount of powder on sieve 60 in two states:

1) In the first state, the amount of powder on the sieve 60 was considered equal to 60 weight percentage and the changes in the bending strength and water absorption were investigated as well as the changes in the ratios of the two powders (curves 6 and 7).

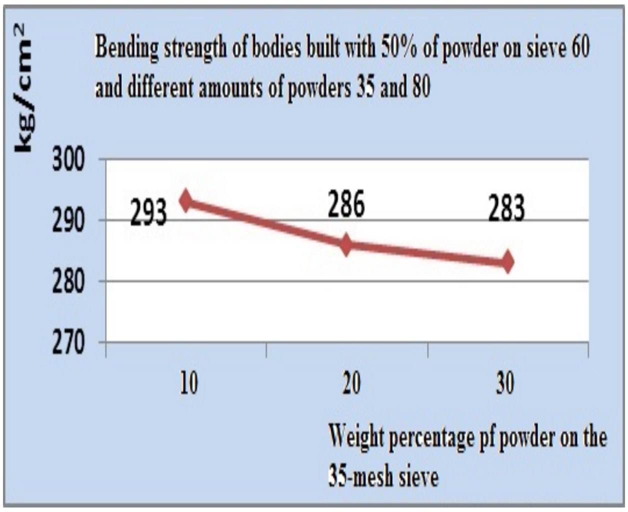
2) In the second mode, the amount of powder retained on a 60 sieve was determined to be 50 weight percentage and the condition of the last experiment was repeated (Curve 8 and 9).



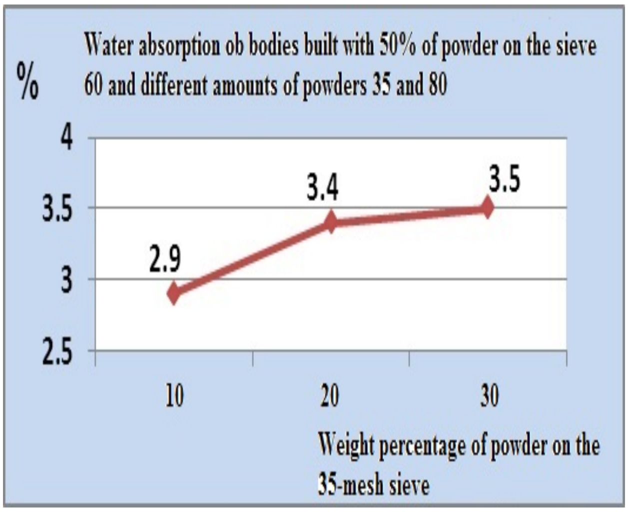
(curve 6)



(curve 7)



Curve 8



Curve 9

6. RESULT AND DISCUSSION

Investigating Diagrams 2 and 3 indicates that the bodies, which are made of powder that remained on the 35-mesh sieve, have more porosity and open space than two other sizes of powder according to the shape and size of granules, and this increases the water absorption (water entry into the open porosities) and decreases the bending strength (due to the reduced body density). The lower density of 35-mesh powder than the 60 and 80-mesh powders is the reason of low density of bodies formed from this powder. The reduced water absorption and increased bending strength in the bodies formed from powder on the 60-mesh sieve according to the increased application range of powder grains (from 35 to 60 mesh) indicates the increased density in these bodies. The high density of powder in this size of granule confirms this fact. The reduced application range of powder grains while using the powder that remained on sieve 80 (from 60 to 80 meshes) reduces the body density and thus the increased water absorption and reduced bending strength with respect to mesh 60. In this regard, the lower powder density than the 60-mesh powder confirms this claim. In fact, these changes are due to the application of a larger area of the powder grains, which are the mixture of small and large grains, and in fact they play an important role in increased density. The penetration of smaller particles into the space of large particles in the bodies, which apply the wider range of granules, is due to the increased density in these types of bodies.

Investigating the changes in Diagram 4 indicates that as the result of adding the 80-mesh powder gradually into the 60-mesh powder, the small particles will lead to the increased density of body structure by penetrating into the larger particles and this will continue until an increase of about 20% of 80-mesh powder; and the decreased density in the body will take place by its increase and reduction in the average size of particles (da) in the mixture. The strength of granule against grinding is another reason for reducing density by increasing it. As shown in Equation (2), as the smaller particles of powder have greater grinding strength (compared to larger particles), density is decreased in the body.

In curve 5, the entrance of large sizes of 35-mesh powder into the texture of 60-mesh powder leads to an increased average size of particles (da) in the mixture and this increases the body density to the range of 20% 35-mesh powder. The less strength of this granule than the 60-mesh powder also increases the density of body. The higher application of this range of powder due to the increased large particles and enhanced space among the particles decrease the body density and thus decreased bending strength.

Investigating the triple systems according to what may be observed in Diagrams 6 and 7 indicates that adding the 35-mesh powder, decreases the body density and thus decreased bending strength and increased water absorption. This can be observed while adding the 35-mesh powder to the triple system with 50% of 60-mesh powder in Diagrams 8 and 9 and this is due to the increased porosity and reduced body density.

An overview of the shape of powder in different sizes indicates that, the larger particles are made from a combination of smaller particles or sticking of the smaller particles to larger ones, and the more we go towards the smaller sizes of powder, the more the particles appear independently. In fact, the 35-mesh powder, as shown in Figure 3(A), has different particles with different sizes, while the adhesion of powders in 60 and 80-mesh powders is decreasing, respectively.

The question worth bringing up is why higher compaction cannot be achieved in a body considering the fact that large particles are formed by the accumulation of small particles in different sizes? Is it not true that they have been formed by different particles capable of enhancing the fragment density? The answer can be put this way: 1) comparison of powder density in 35, 60 and 80-mesh indicates that density of 35-mesh powder formed by a variety of powders is lower than the other two powder sizes, which is due to particle shapes. As may be seen in Figure 3-A, particles stick together randomly in a powder particle while the empty spaces between particles plays a key role in reducing powder density. 2) Usage of various particle sizes is effective in reducing porosity and enhancing fragment density when the particles are able to move around under the applied pressure and diffuse in intergranular empty spaces. Since large powders such as 35-mesh are conglomerated and bound together, it would not take place easily. 3) Even though compression leads to crushing of large particles, it can help separate and transport small conglomerated particles into empty spaces. Due to the systematic compression applied, however, it takes place quite slowly as network compaction intensifies, particle relocation in the fragment decreases (in Equation 1, a systematic compression is negatively correlated with level of particle penetration into the fragment interior).

Now it can be asked, which range of particle size distribution can be most effective for improvement of compaction in a tile body? As it can be seen in Equation 1, as granulation size grows, the particle penetration into the network in the empty spaces increases, ultimately contributing to enhanced body compactness ($D_{AB} \approx d_A$). In Equation 2, the effect of granule size on crushing resistance indicates that the level of crushing resistance is negatively correlated with granule size ($\mathbf{D^{-2}_G = \delta_{fG}}$). Hence, the larger granules size in a body cause the higher compaction at lower compression. Therefore, adjustment of slip and spray dryer conditions aimed at enhancing granule size can improve compaction and at the same time eliminate porosities, which in turn leads to increased bending strength and diminished water absorption.

7. CONCLUSION

Throughout the tile production process, particle size distribution is a key factor contributing to the identification of several output properties including bending strength and water absorption. Considering the fact that increased particle size distribution leads to more mass conduction, producing greater compactness in the formed body. The application of various powder sizes at different ratios can play an important role in creating the desired characteristics in the final product. Since different powder sizes show unique behaviours throughout the production process, it should be noted, regarding the application of powder with a variety of granulation, that the amount of small grain powders should be limited such as to fill up empty spaces between larger particles, which in turn would unnecessarily enhance porosity and weaken compaction. Hence, body compaction needs to rise in order to yield a high-density body such as porcelain tiles (i.e. water absorption as low as 0.05 and high bending strength), which can be obtained by using a variety of powder granulations and specifying the optimized curve for the equation. As a result, it is essential to determine the optimized range of percentages for each powder size in order to achieve appropriate body compaction.

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