A MODEL TO PREDICT THE MECHANICAL STRENGTH OF A GREEN CERAMIC BODY

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SUMMARY

Apparent green density measurements, flexural strength and Young's modulus were performed with the purpose of investigating the pressure of compactation and humidity content effects on the mechanical strength of a porcelainized stoneware green body. From the experimental data collected, a model to predict the mechanical strength was also proposed. The results of the density measurements show that the apparent density increases linearly as the pressure (range from 22 to 67 MPa) of compactation and humidity (range from 3.5 to 7.5 %) content increase. The flexural strength behavior was the same as observed for the apparent density i.e., it increases linearly as the pressure of compactation and humidity content increase. On the other hand, the Young's modulus was affected strongly by the pressure of compactation and to a lesser extent by the humidity content. Meanwhile, a maximum Young's modulus was obtained for humidity contents from 5.5 to 4.5 %. The predictive model proposed in this work is shown to be suitable for designing the mechanical strength of this particular green ceramic body since the calculated and experimental data are in good agreement, as well as being a guide line for application to other ceramic green bodies.

1 - INTRODUCTION

The modulus of elasticity (Young's modulus-E) and the flexural strength (modulus of rupture-MOR), are properties which are related with inherent characteristics of materials and also their processing. Young's modulus expresses the capacity of a material to be deformed without presenting plastic deformation which means, elastic rigidity, while the flexural strength represents the level of stress required to fracture a material [1]. The relationship between both properties provides suitable information about the real load capacity, to which a material is subjected during work.

Solid materials like ceramic tiles after firing process present a suitable elastic rigidity and mechanical resistance, which allow the normal operations of transportation, manipulation and packing. However, a ceramic tile in the green state, especially of large size, during the operations as above mentioned usually shows material loss, deformation, fractures and cracks, as a consequence of its low mechanical strength, such that in the production of "biscuits" 1 to 2% of the products are lost when the flexural strength is about 0.6 to 1.0 MPa [2]. It is generally accepted that the Young's modulus and the flexural strength increase as the density of sintered powder materials increase [3,4]. Meanwhile, little or even nothing is known about the effect of the apparent green density on Young's modulus and flexural strength of ceramic tile materials. On the other hand, the apparent green density is particularly affected (considering all the other parameters constant) by the pressure of compactation and humidity content. In fact, Navarro [6] studying the pressure of compactation and humidity content effects on the apparent green density of a green ceramic body, demonstrated that the apparent green density increases as the pressure of compactation and humidity content increase, since the maximum apparent green density was obtained to a critical humidity content, which depends on the applied pressure. However, attention should be given to the fact that a higher or a lower green density as a function of the processing parameters which causes a variation of the mechanical strength, can also cause deffects in the green and the final products. Consequently, it is also necessary to find the relationship between the mechanical strength and processing parameters to obtain final ceramic products with optimized properties, which is not the goal of this study. This study has the aim of evaluating the influence of the apparent green density as a function of the pressure of compactation and humidity content on the Young's modulus and flexural strength, and also to propose a pratical model to calculate the apparent density, flexural strength and Young's modulus from experimental data, so that the data collected will enable a guide line to be formed for obtaining green ceramic bodies with optimized mechanical properties.

2 - EXPERIMENTAL PROCEDURE

For this study an industrial atomized ceramic powder used in the production of porcelainized stoneware "gres porcellanato" tiles was chosen. With the aim of verifying the influence of the processing variables (pressure of compactation and humidity) on the green apparent density (d), flexural strength (MOR) and Young's modulus (E), samples presenting nominal dimentions of 100 x 50 x 8 mm and with humidity contents from 3.5 to 7.5 %, were obtained by compactation at three different pressures (22, 45 and 67 MPa respectively-typical pressure values used in ceramic tile manufacturing) by means of an automatic press machine.

For the density measurements of the green ceramic samples, Archimedesí principle with mercury immersion method was employed.

Flexural strength tests were conducted in a flexural tester machine according to standard test method EN 100 in the so called three-point bending configuration.

Young's modulus measurements were performed on a "Grindo Sonic MK5" instrument, applying the impulse excitation resonant frequency method for the flexural mode. Figure 1 shows the schematic diagram of the instrumentation that was used.



Figure 1 - Schematic diagram of the instrumentation used for the impulse excitation resonant frequency testing [3, 8].

According to the figure the measurement of the Young's modulus, applying the resonant frequency method, consists of exciting the test object by means of light external mechanical impulse and analysis of the transient natural vibration during the subsequent free relaxation. For this particular test method, the specimen was set to resonate by applying a light tap which induces a mechanical impulse on the surface of the specimen. To capture the resulting vibration and convert it into electrical signals, a small hand-held piezo-electric vibration detector was used. In this case, the piezo-electric probe was located in contact with the test bar (close to the node) in order to pick-up the vibration. Subsequently, the identified fundamental resonant frequency was analysed and the Young's modulus for a rectangular test bar (100 x 50 x 8 mm) was calculated according to the representative formula given by Spinner and Telfft [7] as follows:

$$E = 0.9465 \text{ K}_{c} f_{c}^{2} L^{4} \rho/t^{2} . 10^{-10}$$

Where,

L = Length of the beam (cm); t = Thickness of the beam (cm); ρ = Specific density (g/cm³); f = Frequency of the fundamental vibration (Hz); K₁ = Size factor given below by equation (2); E = Young's modulus (GPa)

$$K_{z} = 1 + t^{2}/L^{2} (7.32 - 11 a^{2}/t^{2})$$
 (2)

Accuracy of the resonant frequency measurements is vital for Young's modulus calculation, since E-modulus is a function of several independent variables, which are associated with an error propagation [7]. Although the fractional error of the measured E-value, which is related to the fractional error of the other variables, can change the absolute E-value, its real tendency is maintained.

For each measurement of density, flexural strength and Young's modulus, five different samples for each condition of pressure and humidity were tested.

3 - RESULTS AND DISCUSSION

3.1 - Effect of the pressure of compactation and humidity content on the apparent green density

Figure 2 shows the pressure of compactation and humidity content effects on the apparent density of the green ceramic body. As shown in the figure, the apparent density increases when the pressure of compactation and humidity content increase. This good linear relationship between apparent density and humidity was expected and is typical of green ceramic bodies, as reported by Navarro [6].



Figure 2 - Apparent green density (d) plotted against humidity content (h) for three pressures of compactation ($\blacklozenge p_1 = 22 \text{ MPa}, \blacksquare p_2 = 45 \text{ MPa}, \blacktriangle p_3 = 67 \text{ MPa}$). Correlation factors (r): $r_1 = 0.97$, $r_2 = 0.99$, $r_3 = 0.99$.

(1)

3.2 - Effect of the pressure of compactation and humidity content on the flexural strength and Young's modulus

Figures 3 and 4 show the pressure of compactation and humidity content effects on the flexural strength (MOR) and Young's modulus (E) respectively. It can be seen from figure 3 that the flexural strength is dependent on the pressure of compactation and the humidity content, i.e. it increases linearly as the pressure of compactation and humidity content increase.



Figure 3 - Flexural strength (MOR) plotted against humidity content (h) for three pressures of compactation (\blacklozenge p₁ = 22 MPa, \blacksquare p₂ = 45 MPa, \blacktriangle p₃ = 67 MPa). Correlation factors (r): r₁ = 0.98, r₂ = 0.96, r₃ = 0.96.



Figure 4 - Young's modulus (E) plotted against humidity content (h) for three pressures of compactation ($\blacklozenge p_1 = 22 \text{ MPa}, \blacksquare p_2 = 45 \text{ MPa}, \blacktriangle p_3 = 67 \text{ MPa}$).

This behavior was expected and is also understandable if we consider that a densification state produces a more packed body and consequently a more resistant material. On the other hand, as shown in figure 4, the Young's modulus practically depends on the pressure of compactation, i.e. it increases as the pressure of compactation increases, since the humidity content has a small influence on it. Meanwhile, as can be seen, for humidity contents of 5.5 and 4.5 % the Young's modulus reaches a maximum. To justify this behavior we can suppose that up to the maximum Young's modulus value the material response to the mechanical solicitation is elastic since it was observed to increase, while for humidity contents higher than 5.5 and 4.5 % respectively, the material response to the mechanical solicitation should be plastic since clay materials unlike metalic materials are easily deformed in the plastic state which explain the decrease of Young's modulus. This plastic state is obtained as a result of the densification caused by the applied pressure but especially by the lubricating action of water. In fact, the last curve regards the pressure of compactation of 67 MPa. The plastification process starts before the other curves.

Besides the Young's modulus, the flexural strength always increases. This continuous increase of the flexural strength can in this case, be related to the fact that for this interval of humidities the behavior of lubricating of water is effective, i.e. promotes a high packed body without destroying it. Probably, for humidity contents higher than 7.5 % the flexural strength will also decrease as a consequence of the body destruction caused by the water excess.

3.3 - Predictive model

By combining all the experimental data as a function of the product of the pressure of compactation (p) and humidity content (h) and associating them with each property (densityd, flexural strength-MOR and Young's modulus-E), linear equations were obtained. Table I also presents all the experimental data and those calculated by the respective linear equations with the relative error associated with each measurement.

Table I - Measured and calculated	properties as function	of the processing variables
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	Humidity contents (%)				
Properties	3.5	4.5	5.5	6.5	7.5
	$p_1 = 22$ MPa (pressure of compactation)				
d (g/cm³)	1.58	1.60	1.65	1.66	1.73
d1 (g/cm³)	1.57 (0.63)	1.61 (0.62)	1.64 (0.61)	1.68 (1.20)	1.72 (0.58)
MOR (MPa)	0.410	0.450	0.510	0.540	0.640
MOR1 (MPa)	0.400 (2.40)	0.456 (1.30)	0.510 (0.00)	0.566 (4.80)	0.620 (3.1)
E (MPa)	1094	1107	1220	1110	1100
E1 (MPa)		-	1198 (1.80)		-
E2 (MPa)		-	1202 (1.50) 0.3*		-

Properties	$p_2 = 45$ MPa (pressure of compactation)				
d (g/cm³)	1.66	1.69	1.73	1.74	1.78
d2 (g/cm ³)	1.66 (0.00)	1.69 (0.00)	1.72 (0.58)	1.75 (0.58)	1.78 (0.00)
MOR (MPa)	0.660	0.740	0.800	0.800	0.850
MOR2 (MPa)	0.682 (3.30)	0.726 (1.90)	0.770 (3.80)	0.814 (1.80)	0.858 (0.90)
E (MPa)	1496	1517	1560	1540	1480
E1 (MPa)		-	1632 (4.60)		-
E2 (MPa)	_	-	1610 (3.20) 1.3	*	-
Properties	Properties p ₂ = 67 MPa (pressure of compactation)				
				1	
d (g/cm³)	1.71	1.74	1.78	1.79	1.82
d (g/cm ³) d3 (g/cm ³)	1.71 1.71 (0.00)	1.74 1.74 (0.00)	1.78 1.77 (0.58)	1.79 1.79 (0.00)	1.82 1.82 (0.00)
d (g/cm ³) d3 (g/cm ³) MOR (MPa)	1.71 1.71 (0.00) 0.900	1.74 1.74 (0.00) 0.960	1.78 1.77 (0.58) 1.020	1.79 1.79 (0.00) 1.020	1.82 1.82 (0.00) 1.060
d (g/cm ³) d3 (g/cm ³) MOR (MPa) MOR3 (MPa)	1.71 1.71 (0.00) 0.900 0.916 (1.80)	1.74 1.74 (0.00) 0.960 0.954 (0.60)	1.78 1.77 (0.58) 1.020 0.992 (2.70)	1.79 1.79 (0.00) 1.020 1.030 (1.00)	1.82 1.82 (0.00) 1.060 1.070 (0.90)
d (g/cm ³) d3 (g/cm ³) MOR (MPa) MOR3 (MPa) E (MPa)	1.71 1.71 (0.00) 0.900 0.916 (1.80) 1814	1.74 1.74 (0.00) 0.960 0.954 (0.60) 1868	1.781.77 (0.58)1.0200.992 (2.70)1840	1.79 1.79 (0.00) 1.020 1.030 (1.00) 1800	1.82 1.82 (0.00) 1.060 1.070 (0.90) 1700
d (g/cm ³) d3 (g/cm ³) MOR (MPa) MOR3 (MPa) E (MPa) E1 (MPa)	1.71 1.71 (0.00) 0.900 0.916 (1.80) 1814 -	1.74 1.74 (0.00) 0.960 0.954 (0.60) 1868 1818 (2.70)	1.78 1.77 (0.58) 1.020 0.992 (2.70) 1840 —	1.79 1.79 (0.00) 1.020 1.030 (1.00) 1800 -	1.82 1.82 (0.00) 1.060 1.070 (0.90) 1700

d, MOR, E : Are the measured values

(): Are the relative errors (%)

* : Are the relative errors (%) between E_1 and E_2

3.3.1 - Density prediction

Based on the data collected, as shown in table I, three linear equations to calculate the apparent green density from the experimental data as a function of the processing variables are written as follows:

$d_1 = 0.00164 p_1 h + 1.446$	(3)
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$$d_2 = 0.000644 p_2 h + 1.560$$
 (4)

 $d_3 = 0.000403p_3h + 1.620$ (5)

Where:

 d_1 , d_2 and d_3 = Are the calculated apparent green densities (g/cm³); p_1 , p_2 and p_3 = Are the pressures of compactation (MPa); h = Humidity content (%).

The equations (3), (4) and (5) are valid in the follow conditions:

Equation (3) : $p_1 = 22$ MPa and $3.5 \le h \le 7.5 \%$ Equation (4) : $p_2 = 45$ MPa and $3.5 \le h \le 7.5 \%$ Equation (5) : $p_3 = 67$ MPa and $3.5 \le h \le 7.5 \%$ By comparing the caculated apparent green density values in table I with those measured, it can be seen that there is a good agreement between them with an associated maximum relative error of 1.2 %.

3.3.2 - Flexural strength prediction

By the same method as before (item 3.3.1) three linear equations to calculate the flexural strength-MOR (MPa) were written:

$MOR_1 = 0.0025p_1h + 0.208$	(6)
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 $MOR_2 = 0.000978p_2h + 0.528$ (7)

$$MOR_2 = 0.000567 p_2 h + 0.783$$
 (8)

The conditions of validity of the equations (6), (7) and (8) are the same as before (item 3.3.1). Also in this case, the calculated and experimental values are in good agreement with an associated maximum relative error of 4.8 %.

3.3.3 - Young's modulus prediction

Combining the Young's modulus with the product of the pressure of compactation as shown in figure 5, no linear relationship exists, as is also observed in figure 3. However, the points of maximum Young's modulus are the same. As already mentioned previously, Young's modulus values lower or higher than the maximum cause an increase or decrease of the MOR values. Consequently, from the point of view of mechanical strength the best combination between Young's modulus and flexural strength will be the one concerning the maximum Young's modulus. Considering these aspects and the fact that through the points of maximum Young's modulus a straight line can be drawn, it is possible to express the Young's modulus by a linear equation as follows:

$$E_1 = 3.43 \text{ph} + 783.45$$
 (9)

Applying the same procedure as before a plot between Young's modulus and flexural strength was made, as shown in figure 6. In this case the relationship between Young's modulus and flexural strength is that one expresses by the equation (10).

$$E_2 = 1407.69MOR + 484.18$$
 (10)

Where:

 E_1 and E_2 = Are the Young's modulus (MPa); MOR = Flexural strength (MPa)

The equations (9) and (10) are valid in the follow conditions:

Equation (9) : 22≤p≤67 MPa, 4.5≤h≤5.5 % and 121.00≤ph≤301.50 Equation (10):22≤p≤67 MPa, 4.5≤h≤ 5.5 %, 0.510≤MOR≤O.960 MPa and 121.00≤ph≤301.50

Also in this case the Young's modulus values calculated by the equations (9) and (10) are in good agreement with those measured. The maximum relative errors associated with each measurement and relative to the respective equations were 4.6 and 3.2 %. The

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application of equations (9) or (10) will depend of availabity of experimental data and the correlations of interest. Equation (9) is easier to apply since it depends on the pressure of compactation and humidity content, which are normal in-put parameters of processing. On the other hand equation (10) enables green ceramic bodies with optimized mechanical properties to be predicted. Anyway, independently of the application, both equations can be used to predict the Young's modulus with good agreement since the maximum associated relative error between them is 1.3 %.



Figure 5 - Young's modulus (E) plotted against the product of the presure of compactation (p) and humidity content (h). $\blacklozenge p_1 = 22 \text{ MPa}$, $\blacksquare p_2 = 45 \text{ MPa}$, $\blacktriangle p_3 = 67 \text{ MPa}$) for $3.5 \le h \le 7.5\%$ constant. Correlation factor (r): r_1 (E₁)= 0.98.



Figure 6 - Young's modulus (E) plotted against the flexural strength (MOR). $\blacklozenge p_1 = 22$ MPa, $\blacksquare p_2 = 45$ MPa, $\blacktriangle p_3 = 67$ MPa) for $3.5 \le h \le 7.5\%$ constant. Correlation factor (r): $r_1 (E_2) = 0.99$.

4 - CONCLUSIONS

a). The apparent green density and the flexural strength of the porcelainized stoneware green body studied increase linearly when the pressure of compactation and humidity content increase;

b). The Young's modulus increases as the pressure of compactation increases, since the humidity has little influence on it. However, maximum Young's moduli are obtained for humidity contents of 5.5 and 4.5 % respectively;

c). The calculated values from the developed equations are in good agreement with the measured one, which demonstrates that they can be applied to predict and optimize the properties of this particular green body. On the other hand, the predictive model presented in this work can be used as a guide line to determine other particular equations to evaluate and optimize the properties of other green ceramic bodies. Consequently, from the point of view of ceramic materials on the green state, the equations obtained from the predictive model concept can be an useful tool to design green ceramic materials with optimized properties without the necessity of performing subsequent laboratory tests.

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