Modulus Of Rupture And Hardness Of Opaque Dental Porcelain

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A R T I C L E   I N F O

A B S T R A C T

Opaque dental porcelain was produced from the combination of 13 different raw materials. The mixture was mixed for 2 hours before melted at 1350°C for 4 hours, followed by quenching in cold water to form frit. The frit was crushed using fast mill machine, and sieved to pass 75 µm. The powder was compacted to form pellets at 60 MPa and sintered at 980 °C for 1 minute using a dental furnace. A comparison between the prepared and commercially available opaque dental porcelain was made. Hardness for opaque dental porcelain is higher than the commercial opaque dental porcelain, while the Young's modulus of opaque dental porcelain is lower than commercial opaque dental porcelain. It is obvious that this opaque dental porcelain can be further improved in order to match the commercial opaque dental porcelain.

INTRODUCTION

Porcelain is composed of a crystalline phase (leucite) dispersed in a glassy (amorphous) matrix. Its chemical composition includes silica (SiO_2), alumina (Al_2O_3), sodium oxide (Na_2O), and potassium oxide (K_2O). Opacifiers (TiO_2, ZrO_2, SnO_2), various heat-stable coloring oxides and small amounts of fluorescing oxides (CeO_2) are added to match the appearance of the dentin/enamel complex structure. The presence of a large amount of glassy phase in dental porcelains (80-90 vol %) permits a translucency similar to that of enamel (Sakaguchi, R.L. and J.M. Powers, 2012). The most critical property is a subjective one-aesthetics. An implant, bridge or crown should be comparable to its neighbouring teeth. Thus, in visible use the ability to produce colour, translucency and fluorescence becomes the key acceptance criteria (Ibsen *et al.,* 1991). A successful colour match is an important aspect of any aesthetic restoration. However, a perfectly matched aesthetic, tooth-coloured restoration cannot be ensured, despite the improved colour and translucency of layered ceramic restorations (Sahin, V., *et al.*, 2010). Another problem is that, during sintering the density of the porcelain greatly increases and is associated with volume shrinkage of between 30 and 40% (Denry, I.L., 1996; Santander, S.A., *et al.*, 2007; Shenoy, A. and N. Shenoy, 2010). In order to overcome the problem of lack of strength and toughness of dental porcelains, there are two possible solutions to the problem. One solution is to provide the dental porcelain with support from a stronger substructure. The other option is to produce ceramics, which are stronger and tougher (Noort, R.V., 2007). The aim of this study is to produce opaque dental porcelain its properties as close as possible to the properties of commercially available opaque dental porcelain.

MATERIALS AND METHODS

Preparation of the opaque dental porcelain powder begins with the characterisation of the raw materials. This will be formulated by preparing the best composition to produce the chosen colour of opaque dental porcelain after sintering. Three different additives i.e. zirconia, cerium and vanadium oxides are selected for this task. The experimental work will be concluded by various testing study of the prepared samples.

Analysis of Raw Materials:
The composition of the opaque dental porcelain in this study was based on the work by Ibsen (1990). From this chemical composition, raw materials as given in (Table 1) were selected. The raw materials used in this

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study were supplied by R & M Chemicals (UK) Limited except K-feldspar and Na-feldspar, which are bought from a local supplier. The quality of the raw materials was analysed using XRD, XRF and particle size analysis.

Table 1: Raw materials for preparation of opaque dental porcelain base frit.

Ingredient	Weight %
$Na_2O_3.Al_2O_3.6SiO_2$	27.91
$K_2O.Al_2O_3.6SiO_2$	51.46
$Al_2O_3.2SiO_2.2H_2O$	0.70
SiO_2	11.00
Al_2O_3	1.06
Na_2CO_3	1.38
KC_2O_3	2.95
$MgCO_3$	0.28
$CaCO_3$	1.29
$SrCO_3$	0.08
$BaCO_3$	0.28
TiO_2	0.115
Li_2CO_3	1.50

X-Ray Diffraction (XRD):

XRD method was applied to confirm the phases present in the starting materials and the sintered pellets. Diffractometer (Phillips, Model PW1820) with a copper anode ($\lambda = 1.54056Å$) was used. Powder from samples was pressed into flat compacted layer in the aluminium based sample holder, whereas sintered pellets were placed directly into the sample holder. The sample placed into the stage of diffractometer, monochromatic X-rays are directed at the sample and the diffracted X-rays at various angles with respect to the primary beam processed by the detector.

X-Ray Fluorescence (XRF):

Chemical analysis of each raw material carried out using an XRF machine (RigakuSpectometer, Model RIX 3000). Sample prepared from glass disc. About 0.6 g powdered sample mixed with 6 g flux (50 % lithium tetraborate and 50 % lithium metaborate) in a platinum crucible and fired to form a glass disc. XRF analysis carried out using this glass. The value of L.O.I. (loss on ignition) of each sample has been done using electrical furnace (Carbolite HTF 18/3).

Particle Size Analysis:

Particle size distribution analysis was carried out using Malvern Master Size E version 1.2. The powder was dispersed in the deionised water tank using ultrasonic stirrer. A drop of defflocculant (2 % Calgon) will be applied to help the dispersion of the powder. The dispersed particles pass through the parallel laser beams of the sensor. The diffraction pattern is collected on a highly sensitive semi-circular multi-element detector.

Preparation of The Opaque Dental Porcelain Powders:

The composition of the basic opaque dental porcelain is given in (Table 1). Each powder of the raw materials was weighted using 4 decimal points balance (Precisa, Model XT220A). The composition was dry mixed using a plastic container with 10 zirconia balls. The mixing was carried out for 2 hours using a mixer (Multi-Drive). The mixed powder was transferred into an alumina crucible before subjected to the melting process at 1350°C for 4 hours using Lenton glass melting furnace. The molten product took out from the furnace and immediately quenched in cold water to form frit. The fruit was dried before crushed using a fast mill machine to produce below 75 µm powders. This is the base-opaque dental porcelain powder. The powder was pressed at 60 MPa to form 13mm diameter pellets. Each pellet was sintered using dental furnace for three minutes. The soaking time at the maximum temperature 980 °C is 1 minute according to the instructions of the manufacturer (Vision).

Comparison between the Opaque Dental Porcelain with the Commercially Available Opaque Dental Porcelain:

Both powders were prepared as a pellet and sintered at the same temperature. Both pellets were subjected to the same testing procedures.

Hardness:

Hardness test was carried out according to ASTM C-1327. In this test, a small pyramidal diamond is pressed into the material being tested. The test applied using Vickers Hardness Tester (FUTUER-TECH, Model FV-7). The Vickers Hardness number (HV) is the ratio of the load applied to the surface area of the indention. The indenter is made of diamond, and is in the form of a square-based pyramid having an angle of 136 degrees between faces. The faces are highly-polished, free from surface imperfections, and the point is sharp. Three

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samples used in this test, for each sample five indentions at different places. The load applied was 5 kg for 2 seconds.

The Vickers hardness number is computed from the equation 1:

HV = 1.8544 (P/d2)   (1)

where:
P = load, kgf.
d = average length of two diagonals of indentation mm.
d = (d1 + d2) / 2

Modulus of Rupture (MOR):

Ten samples of both materials; opaque dental porcelain and commercial opaque dental porcelain were used. The test applied by Instron 3366. This test was conducted by placing a sample of known width and thickness onto two parallel bars with the bottom of the sample in contact with the bars. By slowly lowering two additional parallel bars with a force gauge attached, the bar was deflected until it breaks. By multiplying factors for the width and thickness of the sample as well as the force load at the point of breakage, then modulus of rupture can be calculated using equation 2.

$$\sigma = \frac{3FL}{2bd^2}$$

(2)

Where:
σ = stress.
F = applied force.
L = distance between the supports.
b = width of the specimen.
d = depth of the specimen.

RESULTS AND DISCUSSION

Analysis of Raw Materials:

The quality and purity of the raw materials used in this study were analysed using XRD, XRF and particle size analysis. Due to certain constraints only major raw materials were analysed using XRF.

X-Ray Diffraction (XRD):

XRD analysis indicated that all the raw materials used for the preparation of opaque dental porcelain containing the desired compounds. It is clear that all the major raw materials (i.e. K-feldspar, Na-feldspar, alumina, calcium carbonate and silica) containing the correct minerals. The same observation can be seen for other minor components such as kaolin, alumina, sodium carbonate, potassium carbonate and magnesium carbonate. Therefore, this XRD analysis is able to confirm all the minerals required in the raw materials for the preparation of the opaque dental porcelain. The result of the analysis was shown in (Table 2).

Table 2: XRD results for raw materials used in the specimens

Materials	ICDD No	Name of compound
$Na_2O_3.Al_2O_3.6SiO_2$	09-466	Na-feldspar
$K_2O.Al_2O_3.6SiO_2$	2-675	K-feldspar
$Al_2O_3.2SiO_2.2H_2O$	6-221	Kaolin
SiO_2	3-444	Silica
Al_2O_3	10-173	Alumina
Na_2CO_3	19-1130	Sodium carbonate
KC_2O_3	16-820	Potassium carbonate
MgO_3	8-479	Magnesium carbonate hydroxide hydrate
$CaCO_3$	5-586	Calcium carbonate
$SrCO_3$	5-418	Strontium carbonate
$BaCO_3$	5-378	Barium carbonate
TiO_2	21-1276	Titanium oxide
Li_2CO_3	2-1141	Lithium carbonate

X-Ray Fluorescence (XRF):

Only 5 major raw materials were analysed using XRF system. The result of XRF analysis for Na-feldspar, K- feldspar, alumina, limestone and silica was shown in (Table 3). The analysis indicated that these major raw

materials are relatively pure. The amount of Fe_2O_3 in each compound is considered very minimum to affect the bulk colour of the opaque dental porcelain.

Table 3: XRF results for 5 major raw materials, wt % of the elements in their oxide form

Wt % of elements (Metal Oxide)	K-feldspar	Na-feldspar	Alumina	Calcium Carbonate	Silica
Na_2O	2.30	5.40	-	-	-
Al_2O_3	19.00	19.25	99.766	0.017	0.2
SiO_2	67.00	71.00	0.088	0.23	99
P_2O_5	0.17	0.26	-	0.023	-
SO_3	0.027	0.028	0.034	0.048	-
K_2O	11.00	2.2	-	-	0.026
CaO	0.11	1.6	0.039	99.651	0.015
Fe_2O_3	0.12	0.18	0.055	0.013	0.054
NiO	0.025	0.014	0.011	-	0.016
Rb_2O	0.27	0.04	-	-	-
ZrO_2	-	0.028	-	-	Trace
CuO	-	-	-	-	Trace

Particle Size Analysis:
     The powders dispersed in water and stirred continuously to disperse fine particles. The average particle size of powders is given in (Table 4).

Table 4: Average particle size of the raw materials

Raw Materials	Average Particle Size (μm)
K-feldspar	2.48
Na-feldspar	2.33
Silica	2.58
Calcium carbonate	2.77
Lithium carbonate	8.95
Magnesium carbonate hydroxide hydrate	2.04
Titanium oxide	0.46
Barium carbonate	1.13
Sodium carbonate	30.96
Alumina	4.64
Kaolin	1.30
Strontium carbonate	2.11
Potassium carbonate	18.64

Comparison between the Opaque Dental Porcelain with the Commercially Available Opaque Dental Porcelain:
Hardness:
     The most important observation was that the specimens of opaque dental porcelain have higher values of hardness compared to the commercially available opaque dental porcelain as shown in Figure 1. This result is not accurate due to porosity in both opaque dental porcelains. According to ASTM C-1327 if there is excessive cracking from the indentation tips and sides, or the indentation is asymmetric, the indent shall be rejected for measurement. However, it can be considered that their hardness values are in the same range.
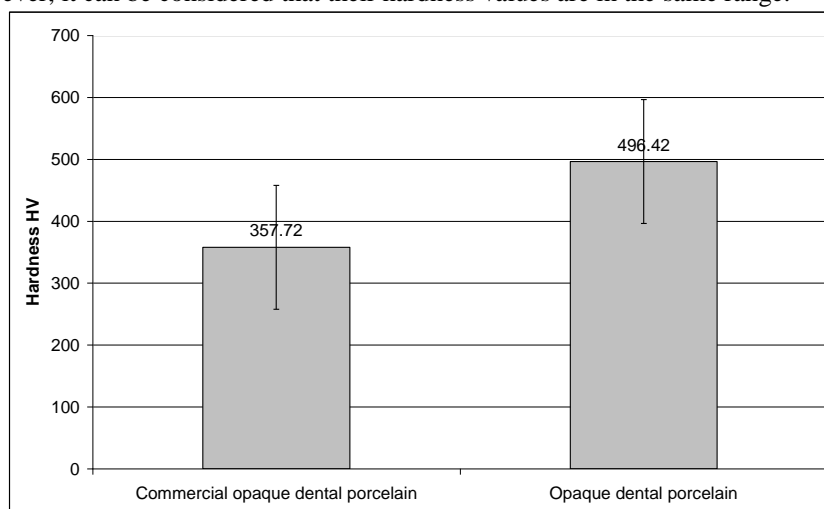

Fig. 1: Vickers hardness for commercially available and opaque dental porcelains.

Modulus of Rupture (MOR):

Figure 2 shows the Young's modulus for two groups of opaque dental porcelain. From diametral test, it is found that the MOR or Young's modulus for commercially available opaque dental porcelain is higher than the opaque dental porcelain. The reason is due to the high porosity in opaque dental porcelain. Increasing in porosity leads to fast decreasing in Young's modulus [8]. Furthermore, there are differences in the composition and sintering temperature for both porcelains (980°C for opaque dental porcelain and 970°C for commercially available opaque dental porcelain) which affects the microstructure (opaque dental porcelain more glassy phase) consequently affects the strength.
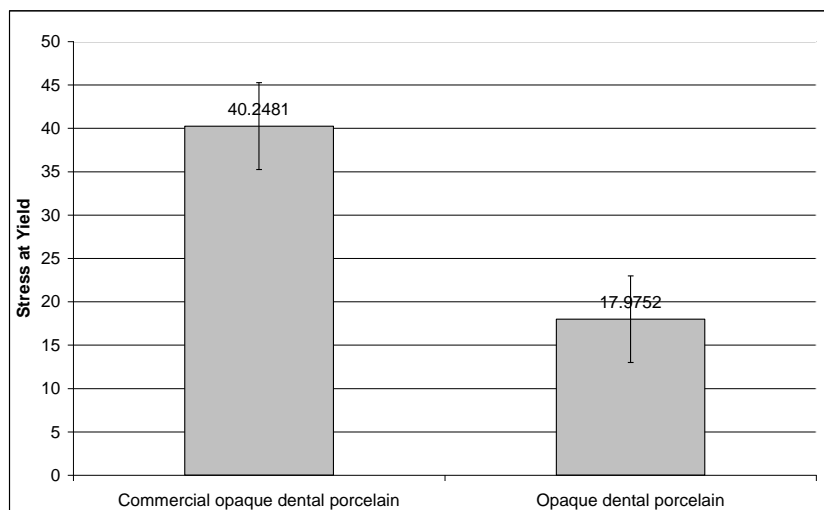

Fig. 2: Young's modulus for commercially available and opaque dental porcelains.

Conclusion:

Opaque dental porcelain was prepared by the mixing of 13 different raw materials for 2 hours then melted at 1350°C for 4 hours, followed by quenching in cold water to form frit which crushed to pass 75 μm. The opaque dental porcelain was subjected to Hardness and Young's modulus tests in form of pellets and compared with commercial opaque dental porcelain. The results showed that Hardness for opaque dental porcelain is higher than the commercial opaque dental porcelain, while the Young's modulus of opaque dental porcelain is lower than commercial opaque dental porcelain. However, more systematic studies are needed to obtain good properties of opaque dental porcelain.

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