CHAPTER 3 EXPERIMENTAL PROCEDURE

This chapter describes all the experimental procedures employed in this work to fabricate and characterize dental porcelains materials with their physical properties tailored by method of ceramic nanocomposites approaches.

3.1 Sample Preparation

Preparation of dental porcelain powders and fabrication of ceramic nanocomposites have been employed as follow:

3.1.1 Powder Preparation

The as-received commercial dental porcelain powders are used as starting materials (Vita-VMK95, Vita Zahnfabrik, Bad Säckingen, Germany), which had particle size range of ~1.5-2.5 μ m. Porcelain powders were mixed with 0, 5, 10, 15, 20, 25, 35, 45, 55 and 65 wt% of zirconia powders (Sigma-Aldrich, purity > 99%) by using a rapid vibro-milling for 30 min [112, 142] (Micronisingmill McCrone Scientific Ltd., England) and left to dry for 6 hours at 120°C in atmospheric furnace (Electrical furnace, Somsak supply, Thailand).

3.1.2 Preparation of the specimens

Green samples were obtained by mixing powders with polyvinyl alcohol binder (PVA) via a slip-casting technique as recommended by the manufacturer [143], and then poured into a standard stainless steel mould with a normal-sized cavity of 30 mm x 6 mm x 2 mm, reproducing the desired dimensions and shapes (Fig. 3.1 and 3.2) [112, 142]. After molding, the ceramics were fabricated by employing different firing schemes with heating rate of 25 °C/min in a vacuum furnace (Multimat Touch & Press, Germany) (Fig. 3.3), as detail demonstrated in Table 3.1. During heating, the temperature was maintained at 500°C for 1 hour to burn out the PVA binder. The dental porcelain was sintered at 980°C for 5 minutes as recommended by the

manufacturer. Samples of dental porcelain ceramics modified with 5, 10, 15, 20, 25, 35, 45, 55 and 65 wt% ZrO_2 additive were sintered by employing the two-step sintering process, sintered at 1040°C for 5 min followed with tempering at 940°C for 90 min, and then quenched into room temperature. However, all of them were sintered in the same drying time (5 min), preheating time (5 min), heat rate (25°C/min), and vacuum level at 0.05 atm. After firing, all specimens were serially ground and wet polished with 280, 400, 800 and 1,200 grade silicon carbide paper mounted on a metallographic lapping machine (Abramin, Struers A/S, Copenhagen, Denmark) to produce surface like a mirror. Finally, the specimens were cleaned using an ultrasonic bath with acetone at room temperature for 15 min (Fig. 3.4).

Group	n	ZrO ₂ addition	Sintering temperature	Sintering time	Tempering temperature	Tempering time
D0	30	0	980	5	-	N-'
D1	30	5	1040	5	940	90
D2	30	10	1040	5	940	90
D3	30	15	1040	5	940	90
D4	30	20	1040	-5	940	90*
D5	30	25	1040	5	940	90
D6	30	35	1040	5	940	90
D7	30	45	1040	5	940	90
D8	30	55	1040	5	940	90
D9	30	65	1040	5	940	90

Table 3.1 The firing schemes employed for the production of samples.

* condition form work of Sanitnapapong et al. [7] All samples were quenched except control group (D0)



Fig. 3.1 Flow chart of green sample preparations.



Fig. 3.2 Industrial shaping process of dental porcelain by slip casting technique: (1) pouring slip into the metal mold, (2) excess moisture removing, (3) surface flattening, (4) unpacking, (5) green specimen, and (6) sintered specimens.



Fig. 3.3 Vacuum furnace (for reducing sample porosity after sintering process).

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★ Details in Table 1.

Fig. 3.4 Flow chart of the specimen preparations.

3.2 Sample Characterization

The following section is intended to address the main characterization techniques used to investigate phase formation, content, size, morphology, distribution of leucite and the density.

3.2.1 Phase analysis

X-ray diffraction (XRD) (X'pert MPD, Philips Corp, Japan) (Fig. 3.5) analyses were conducted to determine the crystalline phase formation and quantitative amount of the crystalline phase within the porcelain composite materials [81]. All 10 selected samples were ground into powders, placed in the holder of a diffractometer and scanned with Cu K_a X-ray, 0.154056 nm λ at 40 kV and 45 mA.

Lattice parameters of the leucite phase were determined from the *d* spacings for the (400) and (004) peaks for the tetragonal phase [3, 4]. The leucite crystallite content was calculated by using the diffraction peak (400) of the leucite pattern and the diffraction peak ($\overline{111}$) of the ZrO₂ pattern [144] as follows:

Leucite content =
$$\frac{I_{(400)}}{I_{(400)} + I_{(\bar{1}11)}}$$
 (3.1)

(3.2)

where

 $I_{(400)}$ is the investigated intensity from leucite (400). $I_{(\bar{1}11)}$ is the investigated intensity from ZrO₂ ($\bar{1}11$).

The mean leucite crystallite size was determined using the diffraction peak (400) of the leucite pattern by using Scherrer equation [144] as follows:

$$t = \frac{0.9 \,\lambda}{B \cos \theta_B}$$

where

B

 θ_{R}

is average crystallite size.

is the wavelength of the X-ray.

is the full width at half maximum intensity of the peak.

is Bragg's diffraction angle.

3.2.2 Microstructural analysis

The 30 polished specimens per groups were section with carborundum disc in to specimen 2 x 2 x 2 mm in size. The specimens were etched with 2 vol% of hydrofluoric acid for 1 min, and platinum-coated (20 nm) for observe microstructure with a field emission SEM (JSM 6335 F, Jeol, Tokyo, Japan) (Fig. 3.6). The chemical composition of the phase formed was also elucidated by an energy dispersive X-ray (EDX) analyzer with an ultra-thin window. EDX spectra were quantified with the virtual standard peaks supplied with the Oxford Instruments eXL software.

3.2.3 Size measurement

Using Image J version 1.46 to measure the particle sizes in control and experimental groups from SEM micrograph.

3.2.4 Density analysis

Densities of the final sintered products were determined by the Archimedes principle using xylene as immersion liquid at room temperature [145]. The density (ρ_c) was calculated according to the equation as follows:

$$\rho_c = \left(\frac{w_1}{w_2 - w_3}\right) \times \rho_{xylene}$$
(3.3)

where

 $\rho_c \text{ and } \rho_{xylene}$ is the density of ceramic and xylene in room temperature ($\rho_{xylene} = 0.863 \text{ g/cm}^3$).

 W_1 , W_2 and W_3 is the dry weight of specimen, wet weight of specimen and weight of the specimen in xylene, measured using a digital scale (Fig 3.7).

3.3 Statistical Analysis

The particle sizes were analyzed by statistical technique of one-way ANOVA (with Scheffé's pairwise multiple comparisons were used to assess whether there was any statistical difference among groups and to identify which pairs of groups were different) at a significance level (*p*) of 0.05 by SPSS version 17.0 (SPSS Science, Chicago, III).



Fig. 3.5 X-ray diffractometer.



Fig. 3.6 Scanning electron microscope, equipped with EDX analyzer.



Fig. 3.7 Digital scale was used for measuring density by the Archimedes principle.

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