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Self-synthesize and flexural strength test porcelain from Indonesian natural sand

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Abstract. Dental porcelain is usually used for aesthetic indirect restoration. Porcelain powder is still imported, although Indonesia is rich with natural sand as porcelain raw material. Flexural strength is a mechanical property which is related to porcelain's ability to resist occlusal force. The objective of this study is to synthesize porcelain from the mixture of Sumateran and Javanese natural sand and evaluate its flexural strength. This experiment used explorative descriptive method. Feldspar Pangaribuan (65% wt), silica Belitung (25%wt), kaolinite Sukabumi (5%wt), and potassium salt (5%wt) was mixed with wet-ball mill and dissolved by fritting to produce porcelain powder. Five samples were made by liquid- phase method in size 3mm x 2cm x 7cm. XRD test was used to analyse crystalline formed and flexural strength of these samples was measured by Netzch Universal Testing Machine with 7.5kg initial load and calculated by flexural strength formula. The result was porcelain powder which has been sintered on 1150 ^{O}C contain leucite (potassium aluminium silicate) crystalline on XRF test with average flexural strength 26.678 MPa. Leucite crystalline is the main indicator for synthesizing dental porcelain because leucite is not usually found on other ceramic. Leucite crystalline is known for increasing strength, preventing crack propagation, and resisting pyroplastic flow. Ten percent of leucite on X-Ray Fluoroscence shows the percentage is less than other dental porcelain. The conclusion of this research is that porcelain succeeded to be self-synthesized from the mixture of Sumateran and Javanese natural sand and having flexural strength 26.678 MPa.

1. Introduction

Porcelain is a type of ceramic which contains glass matrix phase and at least one crystalline phase. Porcelain was used as one of indirect restoration material in dentistry [1, 2]. For its esthetic and mechanical properties, porcelain is fit to restore massive damaged teeth structure. Mechanical property, flexural strength, is really important on this biomaterial as this is related to porcelain's ability to resist occlusal force [1, 3].

On the other hand, production price of dental porcelain is expensive. This might be caused by dental porcelain materials still imported from abroad. In fact, all raw materials of dental porcelain can be obtained in Indoneisa. Feldspar, as the main constituent, can be found at its best at Aceh and North Sumatera. Silica with least impurities can be found at Belitong and kaolinite can be found at any region in Indonesia. [4, 5]

This study aims to synthesize porcelain from the mixture of Sumateran and Javanese natural sand and evaluate its flexural strength. Hypothesis of this experiment is dental porcelain can be synthesized from the mixture of Sumateran and Javanese natural sand (indicator: leucite crystalline on XRD & XRF analysis) and have average flexural strength similar with marketed porcelain's.

2. Materials and methods

This experiment was done in several steps: mixing and fritting raw materials, sintering, X-Ray Fluorescence testing, and flexural strength test.

On this experiment, porcelain powder was made with composition of 65%wt Pangaribuan feldspar, 25%wt Belitung silica, 5%wt Sukabumi kaolinite, and 5%wt potassium salt. Initially, all the raw materials was milled by wet ball mill with 97% ethanol for 4 hours. Milling output was dried with drying cabinet on 50^{O}C for 24 hours. Then, dry mixtures were molded to aluminous crucible for fritting. Fritting was done on the furnace with firing graph as below (figure 1) [6, 7].

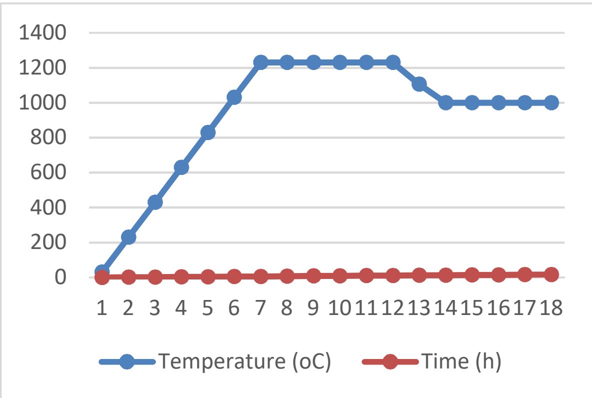

Figure 1. Fritting graph

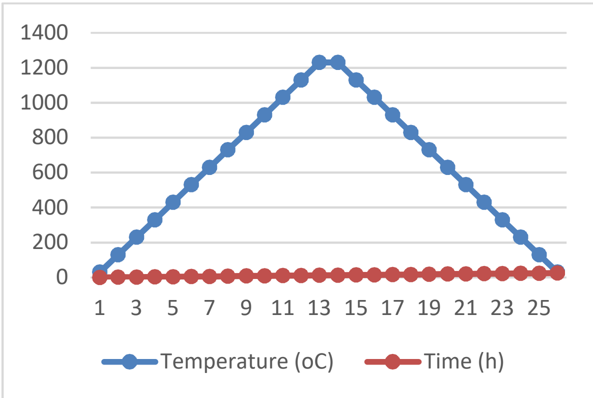

Figure 2. Sintering graph.

On the temperature of 1000^{O}C, crucible was taken out and quenched in iced water until water's temperature is equal with crucible's. Fritting product was then grounded until 200 mesh sized. This powder, called as porcelain powder, manipulated with little aquades for making bar samples as flexural strength test needed. Five samples with size of 70mm x 20mm x 3 mm was sintered (figure 2) on phosphate-bonded gypsum impression [7, 8].

Five samples were tested by Netzsch universal testing machine with 50mm jig range, 1, 6mm lever diameter, and 7.5 kg smallest load. This machine went on the intercalation load of 0.25kg/second. Calculation of flexural strength then performed on the data obtained by the following equation (1) [5, 7].

$$FS = \frac{1.5FL}{bt^2} \quad (1)$$

Where FS: flexural strength; F:biggest load before sample fractures; L: sample length; b: sample width; t: sample thickness.

Broken samples after flexural strength testing were then prepared by milling to 200 mesh sized and tested by Philips Analytical X-Ray BV Lelyweg 1 type 9430 01830401 to wit the crystalline produced in the porcelain.

3. Results and discussion

Dental porcelain on this experiment was synthesized by using 65% wt Pangaribuan feldspar, 25% wt Belitung silica, 5% wt Sukabumi kaolinite, and 5% wt potassium salt. Feldspar was used as glass matrix former, silica as filler and framework, kaolinite as adhesive, and potassium salt increase potassium component. Sintered porcelain shown in white colour and had fairly good translucency (figure 1). [1, 8] Color and translucency are related to particle size and porcelain maturity. Suitable particle size will provide good transparency along with the mechanical properties. The smaller particle size, the closer and stronger bond between particles which means less translucency but greater mechanical properties. While less firing temperature can reduce translucency and mechanical properties [9, 10].

Figure 3. Sintered porcelain.

XRD test results show 2θ most intense are on 27.13; 25.97; and 26.59. The analysis was then performed in accordance with ICDD 15-0047: 3 highest peak of crystalline leucite was at 27.34; 25.93; and 16.52. This indicates the presence of quartz and crystalline leucite in porcelain composition.

X-Ray Fluorescence presented approximately 10% crystalline leucite was on this porcelain. Approximate 10% leucite crystalline found from stoichiometry of the percentage of SiO_2 (58.60%), Al_2O_3 (20.52%), K_2O (10.38%) (table 1). This shows that dental porcelain has been succeeded to be synthesized. On the other hand, leucite crystalline concentration on this dental porcelain is lower than the marketed dental porcelain. This crystalline was formed from the mixing of materials, especially silicate, alumunium oxide, and potassium oxide, which greatly affect the formation of triaxial pottasium alumino silicate. Ratio between pottasium oxide and sodium oxide on the raw materials alse affect the leucite production, the more potassium the more leucite will be formed. Leucite crystalline need to be more considerable as it improves mechanical properties, including strength and hardness [2, 3, 10].

Table 1. The X-Ray fluorescence data of self-synthesized porcelain.

Element	Percentage (%)	Element	Percentage (%)
SiO_2	58.60	Al_2O_3	20.52
K_2O	10.38	Na_2O	7.58
CaO	1.16	PbO	0.59
MgO	<0.001		

The result of flexural strength test on self-synthesized dental porcelain was 26.678MPa (table 1), while marketed dental porcelain's is 65-120MPa. Differences on testing standard (ANSI/ADA 69) were occured on sample size and testing machine requirement. Bigger and thicker sample size on this experiment induced smaller flexural strength. This occured as thicker materials has less strain and easier to crack on flexural strength test [7, 11]. Testing machine requirement on ANSI/ADA 69 is smallest load 5.4kg and intercalation load speed 0.25kg/second. On other hand, testing machine used here has heavier initial load and faster intercalation load speed. This mattered on the more easily samples fractured and the calculation become less accurate. The force loaded on samples becomes dinamic force that strength tested not only flexural but also impact strength [5, 7, 12].

Table 2. Flexural strength test data of self-synthesized porcelain.

Sample Number	F (kg)	L (cm)	B (cm)	T (cm)	Flexural Strength Test (kg/cm^2)
1	11.25	5.0	1.628	0.424	288.289
2	12.50	5.0	1.653	0.432	303.900
3	12.50	5.0	1.621	0.497	234.140
4	12.00	5.0	1.644	0.451	269.146
5	11.00	5.0	1.704	0.424	269.310
				AVERAGE	272.957

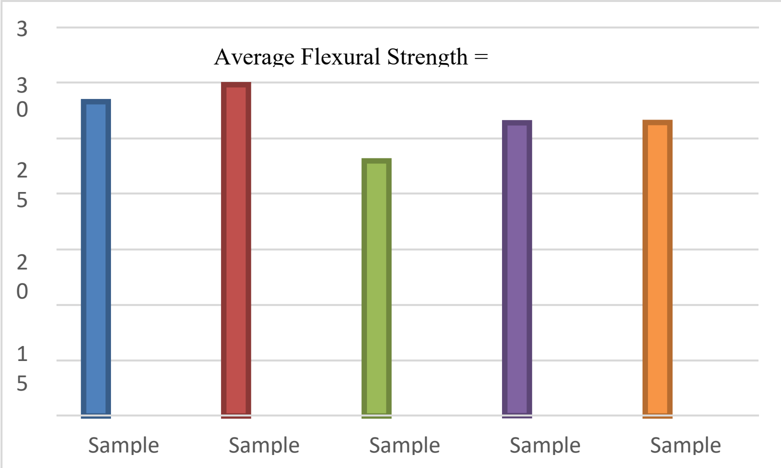

Figure 4. Flexural strength data (MPa).

On the other hand, suitable sintering temperature and time are taking effect on flexural strength. On pre-experiment procedure, that was found that sintering temperature and time is 1150^{o}C in an hour. However, on this experiment with larger size of sample, sintering temperature and time should be higher because of the unmatured porcelain. The calculation of sintering temperature should be made correctly as this affect the maturity and pores production. The unmature and overheating porcelain that will lead to pores production are decreasing dental porcelain flexural strength. [10, 13]

4. Conclusion

From this study, it is shown that the synthesized porcelain contains leucite crystalline which means this porcelain can be used as dental porcelain. The flexural strength of this porcelain is 26.678 MPa and this should be tested further by the method that is suitable with dental porcelain standard (ANSI/ADA) so that can be compared with marketed dental porcelain. Visualization of synthesized porcelain which is probably opaque can indicate this porcelain as body layer, however there is a probability to produce translucent porcelain by manipulate the composition.

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