ORIGINAL ARTICLE

Thermal and Mechanical Properties Analysis of Age-Based Human Tooth Enamel

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ABSTRACT

Introduction: As the important part of tooth, enamel must be treated well in order to keep its function. The purpose of study is provide tooth composition for various condition and at different ages. **Methods:** Children (1-2 years), adults (13-40 years), and parents (41-70 years) tooth, aquabidest, abrasive paper, tooth bor, caliper, trimmer, Machines for Hardness test, compressive test and XRD. Each test sample will be cut, mashed, washed and soaked in aquabidest in refrigerator and then to be examined. **Results:** Micro test results were showed that the main components of the teeth are Hydroxy apatite, Fluorine apatite, and Calcium phosphate hydrate. Transition glass occurs in the age range 1-12 years old at 340^{o}C and in the age range 13-40 years old at a temperature of 400^{o}C , however at the age of 41-70 years old at temperatures of 360^{o}C based on the thermal test results, melting point and boiling point. Mechanical test was showed the level of violence has increased from the age of 1-40 years old then later has decreased, as well as for compressive strength. Hydroxy apatite, Fluorine apatite increase, and Calcium phosphate hydrate decreases in line with age. Tooth enamel also tends to be able to withstand heat in line with age. **Conclusion:** The level of hardness and tooth strength tends to increase temporarily in older people's teeth as the calcium content decreases, thus its mechanical strength decreases.

Keywords: Tooth enamel, Thermal properties, Mechanical properties

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INTRODUCTION

Teeth are hard parts found in the mouths of living things that have varied structures to perform their main functions, such as cutting, tearing, and chewing food. Children's teeth are usually prone to porous or rampant before changing into permanent teeth. Adult teeth often experience tooth loss which causes pain (1).

The most dominant factor causing damage to teeth is caused by an imbalance between the consumption of foods that have the potential to damage teeth and good dental care. The rest of the food attached to the side-lines of the teeth will accumulate within a certain period thus the acidity becomes high and triggers the emergence of microorganisms. These foods usually contain high acidity, sugar content, and food conditions that are too hot or cold.

Dental materials have a hard tissue structure of enamel, dentin, and cementum. Enamel contains 90% -92% inorganic material (hydroxyl apatite) and 6% organic matter and water. The composition of dentin consists of 75% inorganic material (hydroxyl apatite), 20% organic material, 5% water, and other ingredients. However, cementum contains inorganic ingredients (hydroxyl apatite) 45% -50% and 50% - 55% organic matter and water in weight. From the description above, it appears that the main composition of dental materials is hydroxyl apatite.

According to Ajay et al (2), heating of enamel and dentin causes physical-chemical changes such as teeth turning pale white when they get heat stimulation at a certain temperature. Meanwhile, according to Boskey et al (3), 2000 dentin containing low crystalline apatite, begins to turn into whitlockite after being heated at 800^{o}C (4). When the teeth get heat stimulation, the heat will be conditioned and heat distribution is regulated by thermal diffusivity, such as the ability of heat to spread through a material. Enamel will reach temperatures faster than dentin because teeth experience sudden changes in heat. While the temperature is low, the enamel does not contract because it is held by dentin, causing thermal tensile stress in the middle of the tooth. However, if the heat changes occur quite large (800^{o}C), the enamel can experience cracks (4). In the formation of a material,

the influence of thermal properties will produce different characteristics. This happens because of differences in temperature which can result in the atom's chance to organize itself, thus the density, porosity, and crystal structure of the teeth making constituents will change. If the structure of the tooth structure changes, it will affect the level of endurance of the tooth (5). From these problems, this study is expected to provide a description of the composition of dental material for various conditions and at different ages. Through this description, it can be used to analyse the function of each tooth-forming element in humans and obtain information about the relationship between material compositions.

MATERIALS AND METHODS

Dental materials based on age were classified, children (1-2 years), adults (13-40 years), and elderly (41-70 years), each taken 5 (five) core. The teeth were taken from unused tooth (healthy and intact tooth which extracted due to the requirement of orthodontics treatment) were obtained from Department of Dental Surgery Faculty of Dentistry Universitas Airlangga. Aquabides, abrasive paper from coarse-to-fine no. 2000. Tools used for sample preparation include refrigerator, diamond dental bur, cutters, drill bits, callipers, and trimmer machines. Provision of sample temperatures used Differential Thermal Analysis.

Sample Preparation

Preparing test samples in the form of dental material according to age (children (1-12 years), adults (13-40 years), and elderly (41-70 years) with the criteria of no caries and abnormalities in tooth enamel. Each test sample will be cut into 3 parts by using edentate tooth cutting tools.

Every part of the tooth material that has been cut will be tested. Teeth at the age of children who have been cut 1 part micro test, 1 part for the thermal test, and 1 part for the mechanical test. Likewise for teeth in adulthood and parents get the same treatment.

Making Test Samples

After extraction, enamel plates are made by cutting the occlusal surfaces of teeth. Moreover, the enamel was cut with a thickness of 2 mm, a diameter of 1 cm perpendicular to the dental axis, and the surface of the enamel was smoothed then measured length, width, and thickness in accordance with American Standard for Testing and Material (ASTM) E 384 (6). Samples are washed and soaked in aquabides in refrigerators at a temperature of 4oC - 6^{o}C to avoid damage.

Micro Test

The microstructure of the sample was used by X-ray Diffractometer, the result of X-ray diffraction in the form of spectrum with the detected sample intensity. From the intensity distribution recorded in the spectrum of the X-RD test results, it can be analysed quantitatively. The most dominant composition is hydroxyl apatite so Joint Committee on Powder Diffraction Standards (JCPDS) table of apatite hydroxyl crystal was used to match the experimental data.

Thermal Test

Characterization of Differential Thermal Analysis for temperature-190^{o}C - 1600^{o}C with speed settings ranging from 1-50 minutes /^{o}C. The sample (tooth enamel) was placed in an upright position. Administration of temperature for 2 hours aimed to knows the melting point of the teeth or cracks in the teeth were known when getting heat stimulation. This procedure is in accordance ASTM E 1356 (7). Tooth samples that have been treated with temperature were marked, named, and stored in a sterile place.

Mechanical Test

Hardness measurement used Micro-hardness Vicker's Test Future Tech FM 7. The smooth surface of the sample was pressed into the surface of the sample by using a pyramid shaped diamond with a tilt angle of 1 36^{o}. The measurement of the level of hardness was performed at different places on the sample surface. Measurement of compressive strength was conducted by cutting the material in accordance with needs. Install tool accessories for pressure testing and weight selection. After that, put the test material in its place, adjusting the maximum distance needed in accordance with ASTM C773-88 (8). Set the loading speed (choose a low speed) and choose the measured load range (force). Lower the load cell slowly and note the magnitude of the force and strain.

RESULTS

Micro Test

Characterization of micro tests obtained by the diffraction pattern in the form of a continuous spectrum that describes the angles of diffraction occurring at the material atoms (2d) and the intensity result (I / Io) shown in Fig. 1. Each compound that is on international conversion table (JCPDS) has a standard value of the value d, intensity (I / I0), and certain miller index. Determination of the forming compounds was obtained by matching the appearance of the peak position of the chopped results coinciding with the standard peak where the standard. Peak was included in a computer file supporting XRD (9).

According to the identification of tooth-forming minerals, the main material compositions obtained in tooth enamel are hydroxy apatite,fluorine apatite and calcium phosphate hydrate.

Based on Table I and Fig. 2 it can be seen that the main mineral composition in % is hydroxy apatite at the age

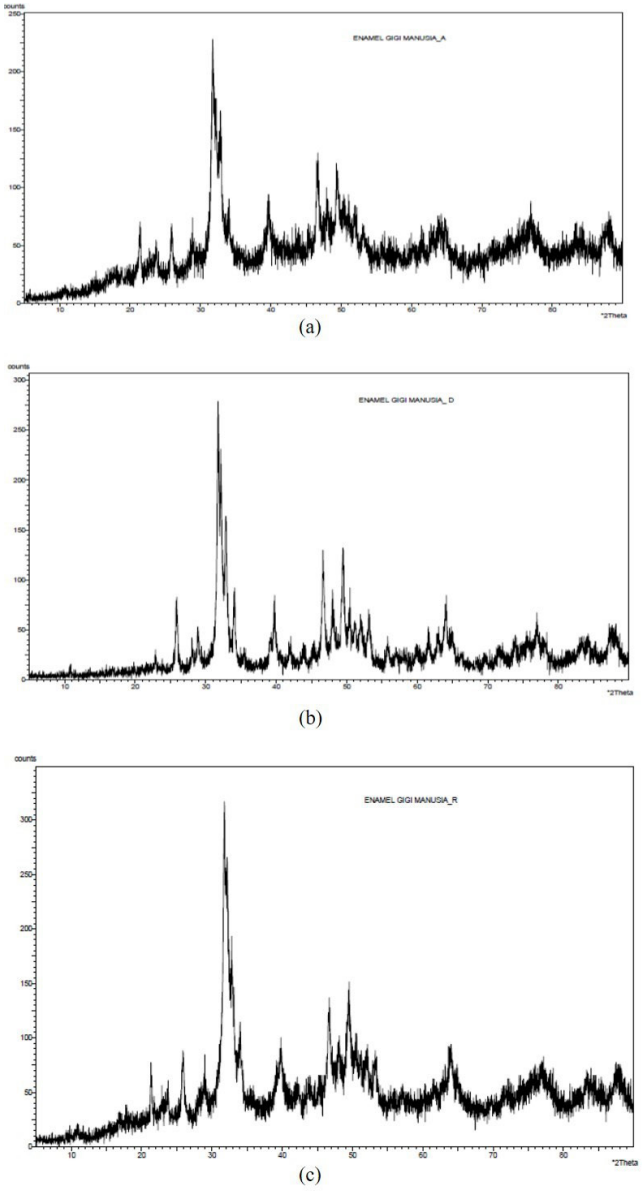

Figure 1: a) XRD Result of Children Teeth, b) XRD Result of Adult Teeth, c) XRD Result of Elderly Teeth. The hydroxyapatite volume fraction of child teeth was 33.00%, the hydroxyapatite volume fraction of adult teeth was 54.41% and the hydroxyapatite volume fraction of elderly teeth was 58.98%.

Table I: Volume Fraction Results in Dental Enamel

SI No	Teeth Material	Hydroxy apatite $[Ca_5(PO_4)_3(OH)]$ (%)	Fluorine apatite $[Ca_5F(-PO_4)_3]$(%)	Calcium phosphate hydrate $[Ca_3(PO_4)_2H_2O]$ (%)
1	Children	32.99	25.04	41.97
2	Adult	54.41	30.76	14.83
3	Elderly	59.23	33.02	7.75

of the child reaching 32.99, at the age range 13-40 years old reaching 54.41 and hydroxy apatite tends to increase at the age of 40-70 years old. Increased fluorine also occurs with age, however as age increases calcium phosphate hydrate tends to decrease starting from the age of 1-12 years the percentage of calcium phosphate

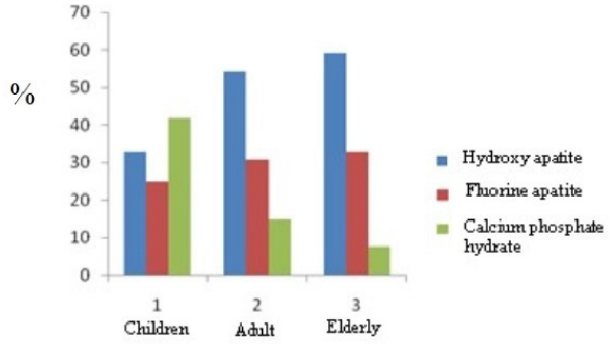

Figure 2: Mineral Percentage Diagram. Hydroxy apatite in child (age range 1- 12 years old) is 32.99%, in adult (age range 13-40 years old) is 54.41%, in elderly (age range 41-70 years old) is 59,23%; Fluorine apatite in child (age range 1- 12 years old) is 25.04 %, in adult (age range 13-40 years old) is 30.76 %, in elderly (age range 41-70 years old) is 33.02 % and calcium phosphate hydrate in child (age range 1- 12 years old) is 41.97 %, in adult (age range 13-40 years old) is 14.83 %, in elderly (age range 41-70 years old) is 7.75%

hydrate by 41.97% then at the age of 13-40 years old by 14.83% and the rate of decline is very sharp. The percentage of calcium phosphate hydrate is 7.75% when in the age range 41-70 years old.

Thermal Test

Thermal analysis uses a Differential Thermal Analysis (DTA) is conducted to determine the differences that occur in tooth enamel on temperature changes. The data obtained in the form of a curve that shows the relationship between exothermic processes and endothermal processes on temperature rise. From the sample characterization results can be made the relationship between the types of samples to the thermal properties shown in Fig. 3.It can be seen through the process of transition glass in the age range 1-12 years old of 340°C and in the age range of 13-40 years old,

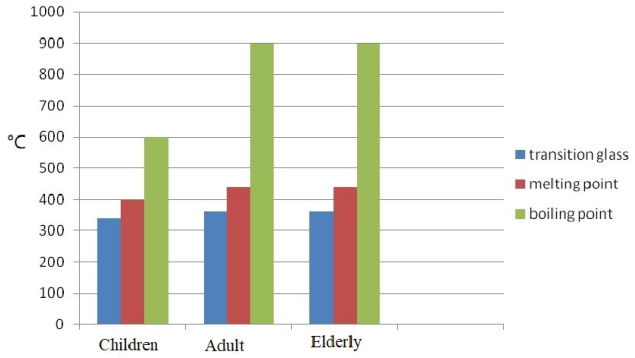

Figure 3: Sample Types of Thermal Properties. Transition glass in child (age range 1-12 years old) is 340°C, in adult (age range 13-40 years old) is 360°C, in elderly(age range 41-70 years old) is 360°C. Melting point in child (age range 1-12 years old) is 400°C, in adult (age range 13-40 years old) is 440°C, in elderly(age range 41-70 years old) is 440°C. Boiling point in child (age range 1-12 years old) is 600°C, in adult (age range 13-40 years old) is >600°C, in elderly(age range 41-70 years old) is >600°C.

the transition glass occurs at temperatures of 400°C, however at the age of 41-70 years old, the transition glass has occurred at 360°C for melting point and boiling point events too.

Mechanical Test

Hardness Test

Hardness value of each sample can be seen at Table II. Based on Table II, it can be seen the relationship between value of teeth hardness.

Table II: Value of Hardness in Teeth Enamel

No	Teeth Enamel Sample	Vickers Hardness Number(VHN)
1	Children	52,02
2	Adult	52,98
3	Elderly	51,49

The age range of 13-40 years old which is 294.2 kgf /mm^2 and the age range 1-12 years old, the value of hardness reaches 298.3 kgf /mm^2, and the age range 41-70 years old, the value of tooth hardness reaches 290.0 kgf /mm^2. It can be seen that the value of hardness tends to rise until adulthood and decrease in the level of hardness in the age range 40-70 years old.

Compressive Strength Test

The compressive test uses the compressive strength values of each sample can be shown in Table III. Based on these data, it can be seen the compressive strength test of tooth enamel by age.

According to the data obtained, Figure 4 shows the compressive strength tends to increase with increasing age, the age range of 1-12 years old the value of compressive strength reaches 46,444 MPa and the age range 13-40 years old reaches 68,999 MPa while in the 41-70 year old age range strength the press has decreased by 57.444 MPa.

Table III.:Values of compressive strength of tooth enamel

No	Sample	Compressive Strength (MPa)
1	Children	46,444
2	Adult	68,999
3	Elderly	57,444

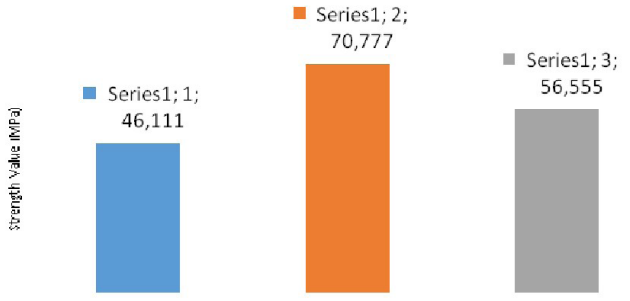

Figure 4: Compressive Strength against Tooth Enamel by Age. The compressive strength in child (age range of 1-12 years old) is 46.11 MPa; in adult (age range 13-40 years old) is 70.77 MPa; in elderly (age range 41-70 years old)is 56.55 MPa.

DISCUSSION

Based on the micro test results obtained mineral compositions namely hydroxy apatite, apatite fluorine, and calcium phosphate hydrate. Hydroxy apatite is a mineral crystal that forms enamel and dentin. Almost all apatite crystals naturally have a small size and less developed thus it will be seen in a picture of sibar x diffraction pattern that is less sharp (10). The properties of apatite crystals are: specific gravity 3.1-3.2 gram /cm^3. Fluorine apatite is derived from the mineral apatite, the most important chemical ion which is expected to be bound a lot by the hydroxy apatite is the fluorine ion (11).

Besides that, hydroxy apatite will turn into apatite fluorine which is more acid resistant. This is because fluoride makes the enamel surface more resistant to caries (12). Calcium phosphate hydrate is a mineral containing molecular compounds together with water molecules in them. Calcium phosphate hydrate is included in amorphous calcium phosphate which is metastable in line with increasing age and will form stable apatite crystals (12). Analysis of thermal properties using DTA is conducted to determine the ability of tooth enamel to temperature. Initially, the temperature of the sample and comparator is the same until there are events that result in temperature changes such as glass transition and melting. From the data obtained, tooth enamel is able to withstand heat long enough when in the age range of 13-40 years old and in the age range of 41-70 years old, the tooth enamel undergoes a glass transition process and melting faster than adult tooth enamel. This is because the hydroxy apatite makes tooth enamel harder and the hydroxy apatite increases as age begins to increase. According to Ajay et al (2), heating of enamel and dentin causes physical-chemical changes that affect the pressure on acids. Whereas, the results of apatite fluorine micro tests tend to increase with age, which can be resistant to acids. Older teeth melt or crack faster. This is due to the low water content in tooth enamel (11).

According to Thompson T4 it takes heat activation energy of 10.2 and 12.5 kcal / mol to remove water and protein degradation. Meanwhile, it is reported that dehydration and the organic matrix of dentine began at heating 60 -100°C and at 250 - 500°C. Decomposition and protein change ends at 600°C (12). Tooth enamel shows better heat stability than dentin especially in the presence of fluorine formation (13). According to the results of mechanical tests both, of the hardness and compressive strength values tend to increase until the age of 40 years old. Judging from the results of micro tests apatite hydroxy mineral content increases with age and also at the age of children to adults have

high calcium, besides that the thickness will increase with age. The elderly of calcium have decreased and the gums will slowly decrease from the enamel to the bottom of the teeth (cementum)the tooth enamel. This study is in accordance with the study of Kunin AA et al (13) which stated that in due to aging process, teeth change both the enamel structure and the level of its ionic processes.

CONCLUSION

The main composition of dental material is composed of hydroxy apatite, apatite fluorine, and calcium charcoal hydrate through micro tests. Thermal differential analysis is showed that tooth enamel tends to be able to retain heat with increasing age due to an increase of fluorine in the dental constituents which makes the enamel more stable in holding heat. Meanwhile, mechanical analysis is showed that the level of hardness and strength of teeth tends to increase due to the increase of apatite hydroxy compounds and apatite fluorine percentage in children and adults teeth towards cementum. This make an impact on mechanical strength which tends to decrease. This study could deliver illustration of dental material composition for various conditions and at different ages. It still need further well characterization with conventional age-specific differences.

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