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Abstract. Artificial teeth with a low solubility and with properties similar to natural teeth were produced for implantation tests. The teeth were made from $MgAl_2O_4$ spinel and $Ca_3(PO_4)_2$ Whitlockite to achieve low solubility and non-toxic reaction products. The method of manufacture is described.

This research was conducted to develop a material with characteristics much like natural dental porcelain which can be used as a permanent replacement for a natural tooth. The final product is seen as either a complete tooth which has one end of high porosity and one of low, or as an anchor in which a hole can be drilled for a tooth cap to be placed. The difference in porosity throughout the pellet is necessary to enable the jaw bone to grow into the pores and form a permanent anchor while the upper less porous area serves as the tooth or the crown support.

Such an implant must fulfill a number of functions:

1) transmit load in chewing to the jaw bone
2) resist wear and stresses of chewing
3) resist chemical solution of mouth acids and ingested chemicals such as citric acid
4) insulate the mouth when in contact with hot or cold foods
5) insulate the mouth from electrical currents set up by electro-chemical potentials
6) cause no harmful reactions to the hard or soft tissues with which it will be in contact.

To fulfill these functions the material must be a hard, impervious, strong insulator of minimum solubility. Any products of hydration must be those which will not cause harmful reactions to the tissues. Natural teeth are composed of fluorohydroxyapatite and collagen. For this research a mixture of Whitlockite, $Ca_3(PO_4)_2$ and spinel, $MgAl_2O_4$, was chosen to give a minimum solubility. Additions of boric oxide, cryolite and rutile were made to facilitate manufacture.

PROCEDURE

Two compositions were investigated. Both contained equimolar ratios of Whitlockite, $Ca_3(PO_4)_2$, and spinel, $MgAl_2O_4$. Batch number one contained 5 weight percent TiO_2, 1 weight percent Na_3AlF_6 and 5 weight percent B_2O_3 to aid firing to a dense structure. Batch number two did not contain B_2O_3. The source of the various constitutents are shown in Table 1.

Five hundred gram lots of each batch were weighed and then ball milled with water for 14 hours. After milling the material was dried on a plaster bat and then broken into chunks for storage in

Table 1

Constituent	Wt. Percent in Batch #1	Source
MgO	8.41%	$MgCO_3$, reagent grade
Al_2O_3	21.26%	Norton 38-900 Alumina
$Ca_3(PO_4)_2$	59.33%	$Ca_3(PO_4)_2$, reagent grade
B_2O_3	5.0%	H_3BO_3
Na_3AlF_6	1.0%	Na_3AlF_6, reagent grade
TiO_2	5.0%	TiO_2, reagent grade

The compositions of both batches are shown in Table 2.

Table 2 (Batch Compositions)

	Batch #1	Batch #2
$MgCO_3$	88.10 gm	92.73 gm
Al_2O_3	106.30	111.89
$Ca_3(PO_4)_2$	296.65	312.25
Na_3AlF_6	5.00	5.26
TiO_2	25.00	26.32
H_3BO_3	71.54	—

containers. The materials were pressed into two-gram pellets in a ½″ die using a Carver Model B laboratory press. A 2000 lb load was applied when pressing creating a pressure of 10,000 psi on the pellet surface. These mixtures were difficult to press. Several organic binders were added to improve pressing. A few drops of a 5% polyvinyl alcohol were found to improve pressing. The specimens were isostatically pressed to improve their homogeneity.

Samples from both batches were fired on platinum foil in a thermal gradiant furnace. Temperatures along the thermal gradiant were measured with platinum, platinum 10% rhodium thermocouples and a millivolt potentiometer. The optimum firing temperature was determined from porosity and density measurements.

The porosity and density of the pellets were determined by the Archimedes method using kerosene as the liquid. A correction for the kerosene density was applied.

Results

The thermal gradiant results are shown in Table 3.
Using these temperatures new samples were made and fired in a small Globar-furnace for extended times—8 to 12 hours after reaching temperature. Experiments to control the porosity of a part of the pellet were also done at this time using naphthalene powder and egg white foam mixed with the sample and binder.

Table 3. Results of Thermal Gradient Firing

Batch 1		Porosity	Bulk Density	Optimum Temp.
	1155°C	1.49%	2.746 gm/cm^3	
	1145°C	1.23%	2.769	
	1080°C	1.85%	2.901	1145°C
	970°C	21.90%	2.438	
Batch 2	1350°C	1.9506%	3.011	
	1250°C	5.040%	3.062	
	1210°C	11.500%	2.91	1350°C
	1205°C	14.300%	2.86	

Above 1350°C density values began to decrease.

Naphthalene works well for increasing the porosity and can be controlled easily.

The difference in binders did not cause a significant change in the porosity or density of the pellets.

Overall Firing Results. Tests of porosity and density showed that overall the pellets made with the boric oxide had lower porosity, were less dense and required a lower temperature to reach maturity than the pellets without boric oxide.

Crushing Strength Tests. One inch high pellets from both batches which had been fired at their optimum temperatures (1145° and 1350°) were crushed using a Carver Model B laboratory press. The maximum load was recorded and the crushing strength was calculated.

	Maximum Load	Crushing Strength
Batch #1 (with boric oxide)	1500 lbs.	7600 psi
Batch #2 (without boric oxide)	1000 lbs.	5100 psi

X-ray Analysis. Sample pellets were ground for 10 minutes in a Spex impact mill. The ground powder was placed in sample holders and x-rayed under at 2 °/minute using copper radiation and a nickel filter. The x-ray diffraction peaks were compared with those of the ASTM card file (Table 4).

Table 4. X-ray Diffraction Results

Batch #1
- $MgAl_2O_4$ Spinel
- Whitlockite, β calcium orthophosphate, $Ca_3(PO_4)_2$
- Traces of $Ca_5(PO_4)_3(OH)$ basic calcium phosphate

Batch #2
- $MgAl_2O_4$ (higher intensity that Batch #1)
- Whitlockite

X-ray analysis also showed that the proportion of $Ca_3(PO_4)_2$ to $MgAl_2O_4$ (magnesium spinel) remained the same as before firing.

Solubility Tests. The object of this test was to determine which batch of material would have minimum solubility in distilled water.

Pellets were crushed very finely using a hammer covered with plastic wrap and the Model B laboratory press. After crushing in this manner they were placed in an agate morter and crushed to pass a #40 sieve. The resulting product was placed in a 250 ml Erlenmeyer flask and washed 6 times with acetone using a clean 30 ml of acetone for each washing and swirling the sample for 30 seconds each washing. The samples were then placed in the drying oven until all acetone had evaporated—approximately thirty minutes.

After drying, 10.000 gms of each sample was weighed into a clean Erlenmeyer flask to which 50 ml of distilled water was added. A flask containing distilled water served as a blank. The three flasks were heated in a boiling water bath for 3 hours, stood overnight, were heated for 8 hours, stood overnight, and were then heated for 3 more hours. The resulting mixture was filtered through #40 filter paper, washed 4 times with 15 ml portions of distilled water, dried and weighed. Weight loss was <0.001 gms. The filtrate was titrated using 0.02n H_2SO_4 with 5 drops of methyl red as an indicator. The total volume of solution being titrated for each batch was 110 ml. The end point with the methyl red solution is PH5.6.

Blank —required 3 drops acid

Batch 1—required 41 drops acid (2 ml)—blank=38 drops

Batch 2—required 7 drops acid—blank=4 drops

The products of hydration could be MgOH, $Al(OH)_3$, $Ca(OH)_2$ and various phosphate ions. Because the spinel is so insoluble the most probable solution is the Whitlockite[1]

$$Ca_3(PO_4)_2 \rightleftharpoons 3\,Ca^{++} + 2\,PO_4^{-3}$$

$$\downarrow\uparrow\ H_2O$$

$$OH^- + [H\,PO_4]^{-2}$$

$$\downarrow\uparrow\ H_2O$$

$$OH^- + [H_2\,PO_4]^{-1}$$

Assuming a simple calcium ion solution, batch two has negligible solubility and batch one represents 0.014% of the sample.

[1] Cit. Dana (to C. S. 686). Gmelin. p. 288 P[A].
Cit. Lange Handbook of Chemistry.

Microscopic Analysis. Samples for reflected light microscopy were mounted in Bakelite. They were polished successively with 220 grit silicon carbide paper, 600 weight silicon carbide paper, 6μ diamond paste, and 1μ diamond paste. Final polishing with 0.05μ alumina using a syntron polishing lap produced a mirror finish.

Microscopic analysis showed that, although specimens from batch number one has the lowest apparent porosity, small gas bubbles and voids are distributed throughout. Those specimens polish to a high glossy finish more like a natural tooth in appearance than do specimens from batch 2. Dr. Phillip Pearson, College of Veterinary Medicines, implanted both compositions below the skin of a canine animal. He indicated slightly more reaction to Composition #1 than Composition #2. Although the reactions were greater than that of Vitallium or Teflon, Dr. Pearson thought they were not harmful. Slight reaction might be beneficial.

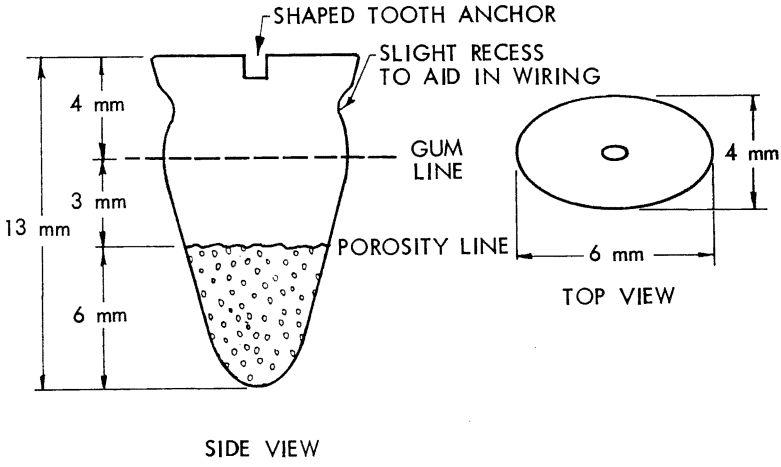

Fig. 1. Shape Of The Finished Pellets.

Artificial teeth were made with one end of high density and one of low density. To achieve this for each pellet 1.3 gm of the unaltered powder was used for the dense end and 1.3 gms of the powder which contained 40% naphthalene crystals was used for the low density end. These materials were placed in layers in a ½" die and then pressed until they held their shape using the Carver Model B laboratory press. No binder was needed using this method. Once the shape was formed the pellets were pressed isostatically to a pressure of 25,000 psi. This prevented the middle section of the pellet from shrinking more than the ends during firing. When the pressing was finished the pellet was tapered and ground to the required shape to fit the tooth socket into which it would be

placed. An allowance for shrinkage during firing was made in the shaping. The shaped pellets were then fired.

Batch #1—with boric oxide at 1145°C

Batch #2—without boric oxide at 1350°C

The pellets can also be shaped after firing by grinding. These implants are now being tested.

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