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ABSTRACT

RELATIVE DENTIFRICE ABRASIVITY ON DENTIN AND ENAMEL

**by
Laura Marchetti**

The abrasive characteristics of six (6) brands of toothpastes on Enamel and Dentin material were assessed. Human teeth were sliced to expose the dentin surface and then polished using $3\ \mu\text{m}$ and $0.25\ \mu\text{m}$ diamond paste. The outside surface of the tooth (after slicing) was used "as is" for the enamel surface testing. The surface smoothness was evaluated prior to testing using a surface profiler and averaging four (4) to five (5) readings across the surface for each sample.

The tooth slices were then embedded into the acrylic plate surface by routing out an area with a dental drill and using polymethylmethacrylate (PMMA) to adhere the teeth to the plate.

An acrylic abrasion machine was used to brush each sample for 3000 strokes with each of the toothpastes being tested. At least three (3) samples of each material were used for each paste. The same type of medium stiffness brush was used for each test. The surface smoothness was then re-assessed using the surface profiler again averaging four (4) to five (5) readings across the surface. The data was statistically analyzed and ranked by abrasiveness.

RELATIVE DENTIFRICE ABRASIVITY ON DENTIN AND ENAMEL

by
Laura Marchetti

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Master of Science in Biomedical Engineering

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RELATIVE DENTIFRICE ABRASIVENESS ON DENTIN AND ENAMEL

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This thesis is dedicated to the memory of my mother.
Always the inspiration for my continued education.

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CHAPTER 1

INTRODUCTION

1.1 History of Dentifrices

Evidence of toothbrushing dates back to Egyptian and Roman times but did not become widespread until the late 18th and early 19th century when the modern toothbrush was invented.¹ In 10 BC, dentifrices consisted of honey, perfume and ground shells or animal bone.¹ Modern paste dentifrices contain the following ingredients: 1 to 5% binders (e.g. methylcellulose) to prevent separation, 10 to 30% humectants (e.g. sorbitol or glycerin) to prevent water loss, 25 to 60% abrasives, 1 to 2% detergents, 20 to 30% water for bulk, flavoring, therapeutic substances such as fluoride and miscellaneous ingredients such as colors and stabilizers to prevent hardening.^{1,2}

As was understood even back in 10 BC, a certain amount of abrasiveness is needed in order to properly clean teeth to effectively prevent caries (cavities). What is understood now after decades of experiments is that there should be a limit to the amount of dentifrice abrasion. Too much abrasiveness can cause the erosion of tooth enamel which can lead to lesions and areas where cavities can form. Some people also have exposed dentin and cementum surfaces or restorative materials which are softer and more susceptible to abrasion and need to be especially careful in choosing a dentifrice since it has been found that dentin and cementum wear 25 and 35 times,

respectively, more than enamel.^{2,4} Due to the similarity in all dentifrice preparations, abrasiveness is one of the few means used to choose a dentifrice.

1.2 Tooth Anatomy

A normal tooth is composed of a center pulp area which contains the nerves and blood supply. Surrounding the pulp cavity is the dentin which makes up the bulk of the tooth. Dentin is harder than compact bone and is composed of 72% inorganic salts.³ Surrounding the dentin above the gum line, is a layer of enamel thinner than the dentin. Enamel is extremely hard and is composed of 96% inorganic salts.³ Cementum covers the dentin of the root (area below the gumline) and serves to attach the tooth. Cementum is composed of collagen in a calcified matrix, similar to bone.³

1.3 Experimental Review

It is nearly impossible to extrapolate the *in vitro* data from this or any other experiment to what would occur *in vivo*. Considering the differences between individuals in brushing practices and the forces of mastication, it is impossible to predict how each dentifrice will act in actual use. Even in standardized *in vivo* studies, several factors were found to be significant enough to consider when designing an *in vitro* experiment. Some important factors to consider are: temperature range, from 28° to 38° C depending on the rinse water temperature; the dentifrice is initially diluted to 22% with saliva; after 30 seconds, 59% of the paste has been spit out; also saliva has a significant buffering effect.⁴

Various experimental methods have been used to evaluate the abrasive characteristics of dentifrices. The most common methods used are the radiotracer and surface profile methods. The radiotracer method provides a measure of "average wear" whereas the surface profile method provides information on "overall wear".⁵ The radiotracer method provides an indirect measurement of abrasion and determines average wear over the brushed surface; for example, it is assumed that the P³² measured in the supernatant is entirely due to wear and not to isotope exchange.⁶ The tracing of a surface profile shows detailed changes in surface configuration after brushing in addition to providing the information needed to estimate overall wear through direct measurement.⁵ The various methods are explained in greater detail further on in this section.

In these studies, several factors were found to affect the results of the study. The brush characteristics (number and length of tufts and hardness), dentifrice concentration, diluent and test temperature were all found to be significant contributory factors.⁷ One study published in 1975, even found that the detergent ingredient used in most dentifrices (sodium lauryl sulfate) causes etching of enamel surfaces.⁸

In an effort to standardize the tests and minimize the variables, a definitive method was described by the Laboratory Abrasion Committee of the American Dental Association (ADA) Dentifrice Program in 1976.⁹ The British Standards Institute (BSI) also came up with a method very similar to the ADA method. Both of these methods use radioassay marking of the dentin to measure abrasiveness. This method works by exposing the dentin and enamel

to P^{32} neutrons for one week. After brushing, the radioactivity of the slurry is measured and the amount of dentin or enamel abrasion is calculated. Other methods used are gravimetric; they measure the amount of material eroded away by weight-loss. Surface profile or shadowgraph methods are also used. The weight-loss method works by weighing each sample before and after brushing to determine the amount of material eroded away. The surface profile method uses a stylus which traverses across the sample surface and measures the oscillatory movements. This is then electronically transferred to a recorder which produces profile curves. A shadowgraph uses light contrast and magnification, similar to a microscope, to show the surface in great detail so that changes in surface configuration can be distinguished. Even with all these methods, the actual abrasive effect of dentifrices during actual use in the mouth is pure conjecture. These methods are only a means to rank the relative abrasiveness of various dentifrices. A 1984 international study headed by the person who developed the ADA method compared the most widely used gravimetric (surface profile) method to the ADA and BSI radioassay methods and found the radioassay methods to be equivalent to each other and more accurate than the surface profile method.¹⁰ It was pointed out, however, that brush characteristics and brushing speed may have been the cause of the poorer results obtained by the surface profile method. A 1984 Swedish study was also performed comparing radiotracer, laser light reflection, laser diffusometer and surface profile methods.¹¹ The laser diffusometer method is as follows: a laser beam is passed through transparent replicas while opaque disks block out the direct beam; the light is then converged on a photometer.

This study found that a combination of both the quantitative (radiotracer) and surface descriptive (profile) methods should be used to more thoroughly evaluate the abrasive properties of dentifrices. A critical comparison of the ADA radiotracer method and a weight comparison gravimetric method was performed in 1992 after different dentifrice rankings were issued using each method.¹² This study found errors in both methods. The most significant problems with the gravimetric method include moisture retention or loss, accumulation of abrasion particles within the dentin tubules and extrapolation accuracy to natural dentin after the drying-wetting-drying cycle of the test method. The radiotracer problems include (1) coloring agents in the dentifrice which affect the recordings, and (2) due to precipitation times the accuracy of the decay rate may be dependent on the time of the recording. Another study has also found that radiation of the dentin specimens alters their properties so that they are more susceptible to abrasion thereby questioning the validity of the radiotracer method.¹³ This study found that the wear resistance of dentin was reduced by 25% and the wear rate was increased by 36%. From all these studies, it can be seen that improvements are needed to both the gravimetric and radiotracer methods in order to make the results comparable. A summary of test methods in 1987, found that the following factors influence *in vitro* abrasion scores: types of abrasion test and standard used, type and concentration of the abrasive, physical characteristics of the abrasive, binders and their concentration, pH of the dentifrices, hardness of the toothbrush bristles, pressure applied during brushing, frequency and length of the brushing strokes and duration of brushing.¹⁴ The limited number of *in vivo* studies have shown

that the following factors are most influential for abrasion rates: number of daily brushings, individual duration of brushing, individual pressure applied during brushing, bristle quality, brushing technique, dilution of the dentifrice by oral fluids, uniformity of composition of the dentifrices and the amount of pellicle, plaque and stain on the teeth.¹¹ The number of methods described here alone attest to the fact that what appears to be a simple problem is really extremely complex.

CHAPTER 2

ABRASIVITY

2.1 Summary of Abrasive Systems

The purpose of abrasives in dentifrices is to remove stain, debris and plaque from the teeth. The abrasives must be compatible with the therapeutic (drug) substances and should cause only minimal loss of tooth material.¹⁵ Some factors affecting the abrasiveness are particle size, pressure exerted on the particles, the speed at which the particle passes over the dental surface and the hardness of the abrasive.¹² Detailed formulations of abrasive systems are closely guarded by manufacturers but the most commonly used abrasives worldwide are carbonates, phosphates, silicas and aluminas.

The following is a brief outline of each abrasive category.¹²

1) Carbonates: Calcium Carbonate in its synthetic form is most commonly used. The synthetic form is not compatible with ionized fluoride but is compatible with sodium monofluorophosphate (MFP). The natural form of calcium carbonate is contaminated with more abrasive oxides and silicas.

2) Phosphates: Calcium Phosphate in its synthetic form is used and is grouped into four major types; calcium phosphate monobasic, calcium phosphate dibasic (either anhydrous or dihydrate), calcium phosphate tribasic and pyrophosphate. As with the carbonates, the natural form of phosphates is more abrasive due to contaminants. Furthermore, depending upon the manufacturing method, the

Phosphates may or may not be compatible with sodium fluoride. Sodium Phosphates in several forms are also sometimes used. For example, an insoluble metaphosphate (IMP) is more commonly used in Europe.

3) Silicas: Silicas are the most common abrasive used in the United States, and are also rarely used in their natural form. Various silicas such as pyrogenic, precipitated and gelatinous can be used; they differ in their particle size, pH and their abrasive properties.

4) Aluminas: These are used in their natural or synthetic forms. They are usually used only when the dentifrice components are incompatible with calcium carbonates or phosphates. The natural form is anhydrous aluminum oxide. The synthetic aluminas come in three forms as aluminum hydroxide, aluminum oxide-trihydrate and aluminum phosphate.

Some more abrasive additives, such as zirconium silicate, flour of pumice and diatomaceous earth are rarely used in dentifrices but are used in prophylaxis pastes. Zirconium silicate has a large particle size and is very abrasive at first but wears rapidly and becomes a good polishing agent. Pumice is a silicate mixture of volcanic origin; and diatomaceous earth is a noncrystalline form of silica.

2.2 Abrasive Systems Evaluated

Six (6) different dentifrices available commercially were evaluated for this paper. The abrasive systems are as follows: Colgate - dibasic calcium phosphate dihydrate; Crest - hydrated silica and trisodium phosphate; Aquafresh - calcium carbonate and hydrated silica; Tom's - calcium carbonate;

Sensodyne - sodium bicarbonate and hydrated silica and Caffree - diatomaceous earth and aluminum silicate. All the pastes contained MFP except the Crest and Sensodyne which contained sodium fluoride. The percentages and particle size of the abrasive systems are controlled by the manufacturers and are not available but some general conclusions can be drawn as to the expected results and this is detailed in the Discussion section of this paper.

CHAPTER 3

EXPERIMENTAL DESIGN

3.1 Materials and Methods

Human teeth obtained from dental extractions were thinly sliced laterally, from the grinding surface up through the root, using an Isomet low speed, diamond blade saw. Most of the teeth used were healthy, third molar extractions; approximately eight to twelve months old. Third molars (aka. wisdom teeth) are normally extracted from individuals in their late teens or early twenties. Slices of either side [buccal (e.g. cheek) or lingual (e.g. tongue)] of the normally exposed tooth surface were used for enamel and dentin surfaces depending on how they were mounted for testing. Prior to mounting, the samples were stored in a water and formalin solution. After mounting, the samples were stored in a closed container with a moist towel to avoid drying. The enamel surfaces were used "as is" but the dentin samples were sanded by hand using 600 grit sand paper then polished by hand using 3 μm and 0.25 μm grit diamond paste. The samples were all mounted on acrylic plates by first routing out an area in the plate with a dental drill and then filling the void with polymethylmethacrylate (PMMA) and setting the sample into the PMMA and allowing it to set. The dentin samples were then as flush to the acrylic surface as possible. The enamel samples, having a rounded surface, were higher than the acrylic surface. The dentin and enamel samples were embedded to avoid dislodging

during brushing, brush "dragging" effects caused by the samples being too far above the surface and the possibility of carrying over PMMA particles from a surface mounting. The surface smoothness was measured using a Mitutoyo SurfTest 401 surface profiler prior to brushing by taking four (4) to five (5) readings across the surface and averaging the results to minimize errors.

Each sample was brushed on an acrylic abrasion machine with six (6) positions (See Figure 1) .

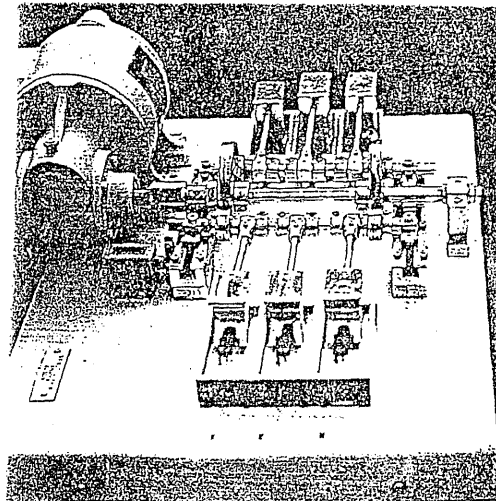


Figure 1 Acrylic Abrasion Machine

An ORAL B 40 regular, medium stiffness brush containing 47 tufts was used to brush each sample and the brush was changed for each test. Approximately 0.5 grams of each paste was placed directly on the sample. The brush was dipped into room temperature tap water for a few seconds just prior to brushing. The samples were brushed for 3000 strokes at a fast speed and a distance of 6 cm with a load of 264 grams. The samples were gently rinsed in tap water and carefully dried. The surface profile was again measured the same

way as before, at right angles to the brush strokes and averaging readings across the surface. Six (6) different dentifrices, commercially available, were tested. The following six (6) pastes and their lot numbers, all purchased in 1997, were evaluated for this test: Colgate Regular lot #8C100496X, Crest Regular lot #6325G, Aquafresh Triple Protection lot #6K11B, Tom's Spearmint lot #7529, Sensodyne with Baking Soda lot #H6874A and Caffree Anti-Stain lot #3023. The test was repeated with three different samples of dentin and enamel for each dentifrice to confirm the results. The measurements were performed at right angles to the direction of the abrasion in five areas of the sample horizontally across the surface. The horizontal X vertical magnification (in centimeters) range used for the dentin samples was 10x3 and for the enamel was 20x3 prior to brushing. After brushing, the range used for all dentin samples was 20x3. A few samples were measured at a range of 20x1 after brushing depending on the characteristics of the brushing site of the machine. This vertical magnification adjustment was made to allow for the area measured laterally by the profiler to be only the area of the dentin surface actually abraded. This was determined by the brush position on the sample during brushing. The enamel samples were measured at a range of 50x3 or 50x1 again allowing for the area actually brushed by the machine.

The data was subjected to statistical analysis of variance (ANOVA). Ten (10) data points before and after brushing were used for each dentifrice. The dentifrices were then ranked by increasing abrasiveness.

Average microns

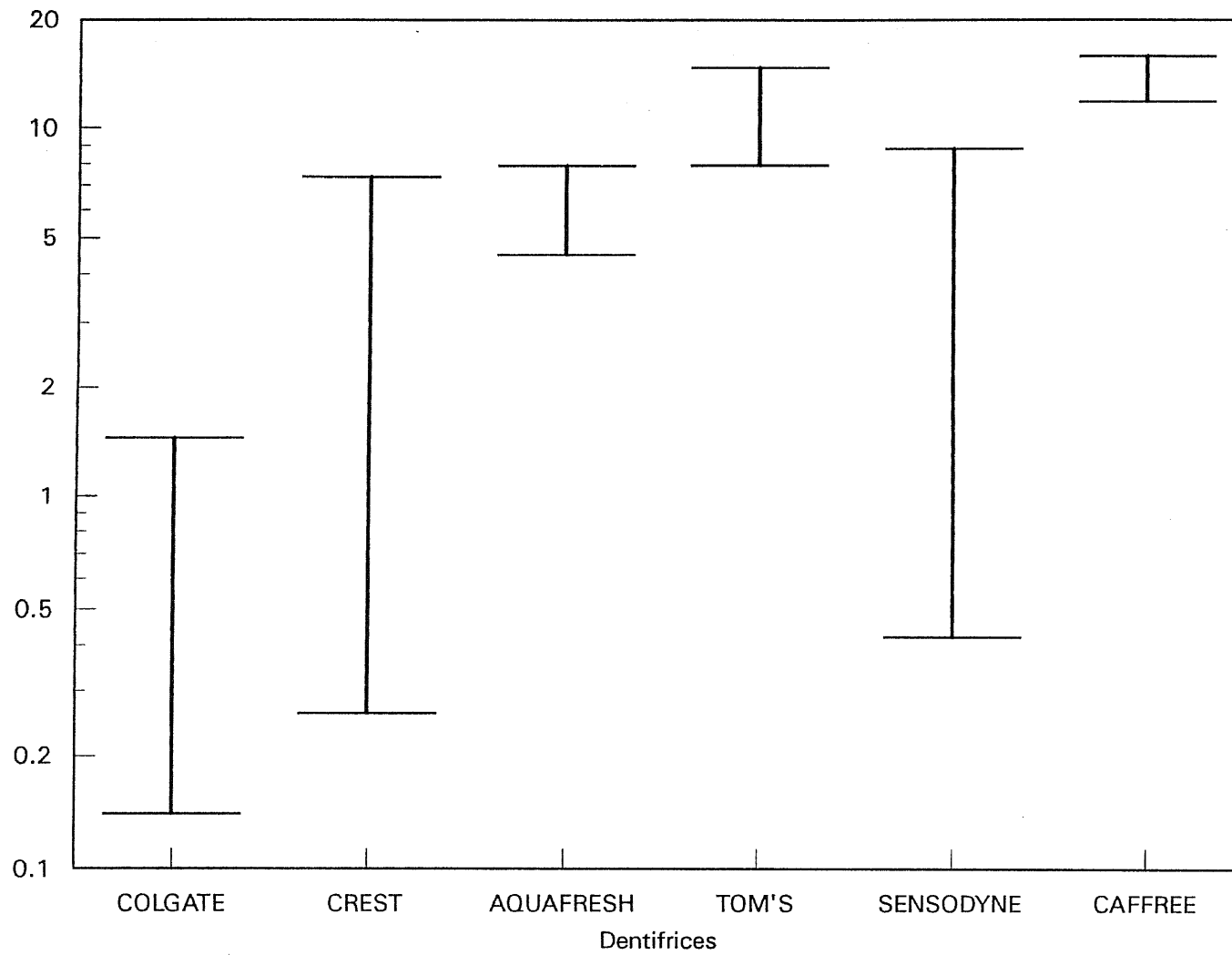


Figure 2 Logarithmic Plot of Table 1 Dentin Data

CHAPTER 4

RESULTS

4.1 Dentin Samples

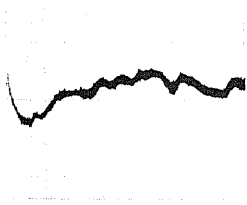
The dentin samples were first ranked by increasing relative abrasiveness by subtracting the average surface profile (in μm) prior to brushing from the average profile after brushing. Table 1 below illustrates the results.

Table 1 Relative Abrasion Results for Dentin Samples

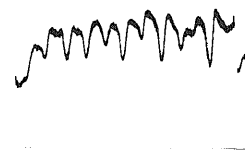
Dentifrice	Sample 1	Sample 2	Sample 3
Colgate	1.44 μm	0.14 μm	0.70 μm
Crest	7.38 μm	0.26 μm	2.92 μm
Aquafresh	5.16 μm	7.92 μm	4.5 μm
Tom's	7.94 μm	10.50 μm	14.76 μm
Sensodyne	4.18 μm	0.42 μm	8.83 μm
Caffree	13.08 μm	15.84 μm	11.86 μm

Facing 14

COLGATE



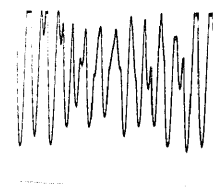
CREST



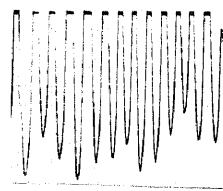
SENSODYNE



AQUAFRESH



CAFFREE



TOM'S

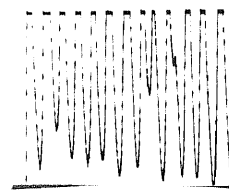


Figure 3 Representative Surface Profile Graphs of Dentin Samples

A logarithmic representation of this data is displayed in Figure 2. As can be seen from this graph, there is a wide margin of data for Crest and Sensodyne. The samples were closely examined and Trial 1 of Crest and Trial 3 of Sensodyne were found to be inconsistent due to the sample being embedded in the acrylic plate at an angle which would affect how the brush abraded the surface. In fact, examination of the results shows that this Crest trial displayed abrasion which varied across the surface from 7 μm to 11 μm ; the Sensodyne trial results ranged in abrasion from 9 μm on the lower end to 16 μm on the higher end. Figure 3 shows a representative illustration of the surface profile graphs. Table 2 below shows the ranking for each trial and an asterisk marks the two trials believed to be anomalous due to the uneven sample placement.

Table 2 Dentifrice Ranking (Increasing Abrasiveness) Prior to Statistical Analysis

Sample 1	Sample 2	Sample 3
Colgate	Colgate	Colgate
Sensodyne	Crest	Crest
Aquafresh	Sensodyne	Aquafresh
Crest *	Aquafresh	Sensodyne *
Tom's	Tom's	Caffree
Caffree	Caffree	Tom's

For this reason, another trial of Sensodyne and Crest was run by polishing the previously used dentin samples. Experiments have shown that dentin samples may be reused, with careful polishing, at least twenty (20) times without showing marked evidence of changes in wear resistance.⁵ This produced an average result of 7.32 μm for Sensodyne and 11.6 μm for Crest. A fifth trial of Crest was run and produced a 5.28 μm reading. No explanation for the wide variation in Crest results was found by sample examination. One explanation could be inconsistency in the paste within the tube.

For each dentifrice, two (2) trials were chosen with the most consistent before and after profile readings for the statistical analysis. Occasionally, individual dentin specimens have exhibited exceptionally high or low readings. Experimentally, results differing by more than 25% from the mean are preferably discarded.⁵ Table 3 shows the dentifrices listed by increasing abrasiveness, standard deviation, comparison mean, Duncan grouping and sample trials used for the analysis.

The dentifrices prior to the statistical analysis, were ranked in the following order of increasing abrasivity: Colgate, Crest, Sensodyne, Aquafresh, Tom's, Caffree. After the statistical analysis, the only difference is Tom's is slightly more abrasive than Caffree.

Table 3 Dentifrice Ranking by Statistical Data

Dentifrice	Standard Deviation	Comparison Mean (μm)	Duncan Grouping	Samples Used
Colgate	0.39	0.42	D	2 & 3
Crest	1.46	4.10	C	3 & 5
Sensodyne	2.18	5.75	B	1 & 4
Aquafresh	2.19	6.21	B	2 & 3
Caffree	3.01	12.47	A	1 & 3
Tom's	2.37	12.63	A	2 & 3

The Duncan multiple range test grouping assigns the same letter to groups in which no statistical difference was found between the comparison means. This test controls the mean comparison error rate and not the experimental error rate. Most statistical data comparison methods need to be selected prior to analysis of the data. This is a problem for researchers since during an experiment there is little idea of what comparisons will be of interest prior to collection and analysis of the data. The Duncan test is one of the methods which can be used after data analysis.

By reviewing Tables 2 and 3, the statistical data is supported by the average micron differences. Colgate is consistently by far the least abrasive,

Crest, Aquafresh and Sensodyne results are similar and Tom's and Caffree are both highly abrasive.

4.2 Enamel Samples

The enamel sample results were calculated by subtracting the average micron readings before and after brushing as was done for the enamel samples prior to the statistical analysis. No statistical analysis was performed on the enamel samples due to the inconsistent results. Table 4 below illustrates the results.

Table 4 Relative Abrasion Results for Enamel Samples

Dentifrice	Sample 1	Sample 2	Sample 3
Colgate	-2.0 μm	-2.15 μm	-1.4 μm
Crest	-6.7 μm	+2.7 μm	-0.5 μm
Aquafresh	-0.8 μm	-1.0 μm	+5.1 μm
Tom's	-4.6 μm	+12.3 μm	-7.7 μm
Sensodyne	+1.85 μm	+0.5 μm	-1.3 μm
Caffree	-0.2 μm	-2.5 μm	+2.98 μm

As can be seen from these results, there is no consistent pattern as there was with the dentin samples. Also many of the results are negative indicating a polishing or smoothing effect on the enamel surface. The samples did appear

to have a glossier or polished appearance after brushing. The positive results indicate some degree of abrasion or frictional erosion of the surface. These results are discussed in more detail in the Discussion section.

CHAPTER 5

DISCUSSION

5.1 Design Justification

This experiment was designed to simulate as realistic a brushing scenario as possible with the available resources. The enamel samples were used as they were cut from the tooth surface without radiation exposure, polishing or undue drying. The dentin samples were subjected to as little drying as possible and not subjected to any foreign agents other than water and formalin. The samples were brushed with a small amount of paste using a wet brush and a realistic brushing load. The 3000 strokes used are approximately equivalent to 3.6 months of twice daily brushing.⁴

It should be pointed out that most teeth are not normally brushed in only one direction. As early as the 1940's, studies were performed which showed that specimens brushed horizontally and vertically showed more wear than specimens brushed in one direction only.⁴ The influences of saliva and plaque also cannot adequately be reproduced in an *in vitro* setting.

5.2 Expected Findings

The overall results of the experiment were as expected. As previously mentioned in this paper, the diatomaceous earth contained in Caffree is highly abrasive. Sodium Bicarbonate found in the Sensodyne, has been shown to be

softer and of low abrasivity.¹⁶ The calcium carbonate found in Tom's as the only abrasive, was found to be highly abrasive to dentin and cementum but not enamel.¹⁷ Tom's is also sold as an "all natural" toothpaste. As pointed out earlier in this paper, the natural form of calcium carbonate is highly abrasive. Studies have shown that the type of calcium carbonate used (e.g. crystalline form and particle size) has considerable effect on the dentifrice abrasivity.⁵ Dibasic calcium phosphate dihydrate found in Colgate is a soft abrasive and when diluted 80%, has been found to decrease its abrasivity by 50%.⁴ This form of calcium phosphate is also five times less abrasive than the anhydrous form.¹³

It is difficult to predict the affect each abrasive system will have without also considering the percentage and particle size of the abrasive. As was discovered in a 1985 study, average particle diameter of the abrasive determines the abrasion rate.¹⁸ Therefore, the silicas and silica combination abrasive systems cannot be estimated prior to testing. Also, as pointed out earlier, the concentration and type of binder affects the abrasion of the dentifrice. The reason for the widely varying Crest trials could be due to non-homogeneity or inconsistency in the paste. This theory cannot be proved without further analysis of the dentifrice.

The enamel samples were not expected to show much wear since enamel is much harder than dentin. Using 3000 strokes is really not enough to show much effect on a substance which has been shown to be harder to abrade than bone.⁴ There also appeared to be large error in the surface profile readings of the enamel before and after brushing due to the curved surface. A study

published of enamel samples in 1976 brushed the samples for 10,000 strokes and used a surface profiler to evaluate the samples.¹⁹ This study also used a complex method of sample preparation involving a silicone rubber mold, epoxy resin and then lapping the specimen flat. This made it easier to control the area brushed and would create more reproducible and precise profile readings. There also may be some polishing effect of the enamel samples. There are little data on this effect but it has been found that the greatest polishing was obtained with an IMP and calcium phosphate mix whereas no polishing effect was found with calcium carbonate.¹⁵ In dentifrices, softer, bigger particles remove film on the tooth surface, whereas small, hard particles polish the surface.⁹ A polished enamel surface is not only cosmetically desirable but also allows for less plaque and debris to adhere to the surface. The samples appear polished after brushing which indicates that there is some polishing effect; however, the primary reason for the inconsistent results is the poor accuracy and reproducibility of the surface profile readings.

General conclusions concerning each brand of dentifrice cannot be made because manufacturers are constantly changing formulations and most have several varieties of dentifrice on the market with various abrasive systems, concentrations and binders. As an example, Caffree is no longer produced.

CHAPTER 6

CONCLUSION

This study has shown that overall reproducible and predictable results on dentifrice abrasion can be obtained by a relatively simple experimental procedure using dentin. Some method improvement is needed to align the samples as flush as possible to the brushing surface in order to eliminate artifacts in the measurement of abrasion. Methodology changes to the enamel samples such as longer brushing time and improved sample preparation are needed to obtain more meaningful results.

This paper also illustrates the extreme complexity of a problem which appears on the surface to be quite simple. This is summed up in the following quote from a 1982 study of restorative material wear; "It is doubtful if a single laboratory test can ever be developed to reproduce the complex wear processes occurring in the mouth, which are mainly masticatory wear and toothbrush/dentifrice abrasion. The relative effects of these different wear processes will vary from one individual to another and will also vary from one location to another within a single mouth."²⁰

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