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The measurement and interpretation of dentifrice abrasiveness

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Synopsis—A radioactive tracer technique has been used to assess the abrasiveness of dentifrices with the specific object of acquiring knowledge for the establishment of a standard dentifrice test procedure. A detailed study has been made of the relationships that exist between the wear resistance of dental tissues and their indentation hardness when using abrasives of different hardnesses. The influence on the wear rate of initial surface preparation of the tissues has been investigated together with the effect of decalcification brought about by exposure to lactic acid. It has been shown theoretically that the relationship between wear rate, dW/dS, and dentifrice concentration, C, is of the form $dW/dS=\alpha(1-e^{-\beta c})$, where α represents the saturation wear rate obtained when every brush fibre has trapped one or more abrasive particles, and $(1-e^{-\beta c})$ is the trapping probability. The experimental values of the ratio of volume of the 'region of influence' surrounding each fibre tip to that of an abrasive particle, β, are in good agreement with the theoretical values based upon spherically ended fibre tip geometry. In comparative testing, the need for an integrated wear curve is stressed in order to take account of variations in dentifrice abrasiveness arising from changes in concentration during oral brushing.

Introduction

The basic requirements of an abrasive type dentifrice are, firstly, that the abrasives present shall assist in cleansing the teeth without causing injury to them or irritation to the mucous membrane of the mouth, and, secondly, that the dentifrice shall be free of particulate matter that might impart an unpleasant feeling in the mouth. Although the latter refers mainly to gritty particles which might scratch the surfaces of exposed dental

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tissues and perhaps impair the cosmetic properties of the product, it also includes lumps of material which the sensory tissues of the mouth distinguish as discrete matter and which destroy the sensation of smoothness when the dentifrice is in use.

A satisfactory test for dentifrice abrasiveness must therefore assess the overall wear likely to be caused to the main dental tissues and at the same time sense the presence of 'gritty' matter. The former is simply a quantitative test of the total amount of wear, whereas the latter is a form of quality control and rejects the dentifrice unless it achieves the measured level of wear without recourse to visible scratching of the surface. Two tests are therefore required. First the product should be screened to find whether it contains gritty particles, and second, if it does not contain those, its overall abrasiveness towards specific dental tissues should be measured, either absolutely, or by comparison with some reference dentifrice.

The problem of grittiness is not discussed further in this paper, since the well known metal disc (coin)/glass-slide technique (1) provides a sufficiently precise assessment of its more damaging aspects. Such a test can readily detect coarse gritty matter in concentrations of 0.1 % w/w, although the presence of such particles may have little effect on the overall wear. On the other hand, it is also perfectly feasible to formulate a dentifrice that will pass the glass-slide test, but exhibit a very high degree of tissue wear. Despite this possibility many National Standards Specifications still only specify the glass-slide test as the abrasiveness control.

The establishment of acceptable limits of overall abrasiveness presents many problems since the actual cleansing requirements will vary widely from one individual to another. Kitchen and Robinson (2) made an attempt to assess this variability by visually examining a large number of students' teeth which were cleaned regularly with certain proprietary dentifrices. After close examination it was found that the tooth enamel of 20% of the students was susceptible to heavy staining, but that these stains could be eliminated in 95% of this heavy-staining group by the regular use of a dentifrice capable of removing 1 mm of dentine in 100,000 brush strokes. Unfortunately, the equivalent enamel wear was not measured but judging from the type of dentifrices employed one can assume that it would be about one hundredth part of the dentine wear. It follows that during the course of a daily brushing of 15 strokes with a dentifrice of the above abrasiveness, the maximum amount of material worn away would be 1.5×10^{-6} mm of enamel or 1.5×10^{-4}mm of dentine. Although the dentine wear is rather high, 1.5×10^{-6}mm of enamel only corresponds to about four

atom layers of the tissue and can be considered a perfectly safe level of wear. However, the fact that most commercially available dentifrices seem to be capable of removing 0.5–1.0 mm of dentine in 100,000 brush strokes implies that these dentifrices have been designed to cope with the heavy staining proportion of the population. If this is so, then the degree of dentine abrasion is possibly too high for many people, especially those showing advanced cervical exposure. The increasing use of motorized toothbrushes, although beneficial to gingival health, necessitates greater care in selecting a dentifrice which avoids excessive wear of dental tissues.

The measurement of dentifrice abrasiveness has always proved difficult in practice, especially with enamel surfaces for which prolonged brushing is often necessary to obtain an accurately measurable amount of wear. This is aggravated by the fact that dental tissues absorb and release water so rapidly that precise weight loss measurements are not easy to make. In addition the tissues vary considerably in composition, not only from one tooth to another, but also over the surface of a single tooth. For these reasons numerous attempts have been made to find alternative test materials (3), but the results have not proved encouraging. Some experiments, made by the present authors with glass surfaces of similar hardness (370 HV 2.5) to enamel, indicated that some proprietary brands of dentifrice chemically attacked the glass and yielded results which correlated badly with those obtained with human enamel. The particular glass employed was a medium barium crown glass containing 6% Tm_2O_3 for radiotracer purposes.

Dentine has usually been the popular choice of test material, but its use in isolation can, as will be seen later, lead to gross errors in the assessment of the abrasiveness of the dentifrice to harder tissues. For the same reason, ivory has little to recommend it, even though it can be obtained in large and convenient pieces.

Most investigators prefer a brushing test for evaluation purposes, since this clearly reproduces the normal oral cleaning action, but others have utilized polishing machines, based upon a wax lapping plate to retain the dentifrice (4). The latter method was favoured some years ago as it avoided the variability experienced with natural fibre brushes. However, the introduction of synthetic fibres has largely overcome this objection, and lapping plate machines are now rarely employed.

Tissue wear may be recorded by measuring either weight loss or changes in dimensions. In spite of the problem presented by a non-uniformly

wearing surface, many workers have preferred to measure dimensional changes, either directly with the aid of a micrometer or by contour measurement. For example, the examination of a dentine/enamel junction profile can give a quick assessment of dentine wear if one assumes that the enamel wear is negligible by comparison.

Any test of abrasiveness should be made on human dental tissue in view of possible chemical interaction between the testpiece and the dentifrice. Also a brushing action should be employed to reproduce those changes in dentifrice concentration normally associated with oral tooth cleansing.The ideal measurement technique avoids the problem of water absorption, but is sufficiently sensitive for a test to be completed after a short period of brushing the natural surface of an extracted tooth. A high wear sensitivity is particularly important as it permits an accurate comparison of two dentifrices to be made without altering the surface geometry of the specimen. This approach is nearly always more desirable than the employment of an accelerated test.

A radiotracer technique appears specially attractive in studying the wear of dental tissues, since phosphorus is available as one of the elements of hydroxyapatite (5). The radionuclide P^{32} is a high energy β emitter having a half life of 14.3 days, whilst its large neutron cross-section enables one to obtain a convenient level of radioactivity after a few hours irradiation in the reactor. A specific activity of about 1 millicurie per gram (mCi/g) of tissue can be obtained for instance after five hours' irradiation in a neutron flux of 10^{12} neutrons/cm^2/s. Using a mica end-window Geiger-Müller counter for which the background estimated activity of a dried P^{32} source is 10^{-4} μCi, it is possible to determine 10^{-7} g of worn dental tissue. In the case of dentine, this is approximately the amount worn away by one stroke of a brush loaded with a conventional dentifrice.

The wear experiments described in this paper have employed this radiotracer technique in conjunction with a laboratory test rig that closely simulates an oral brushing action. To solve specific problems in the accurate measurement of dentifrice abrasiveness the investigation was divided into two parts: (i) A study of the relationships that exist between tissue wear and the hardness of the abrasive, and (ii) the effect of changes in dentifrice concentration on the wear rate produced by brushing.

An understanding of (i) is essential if a standard test material is to be chosen, whilst a knowledge of (ii) will assist in the selection of test conditions for standardizing purposes.

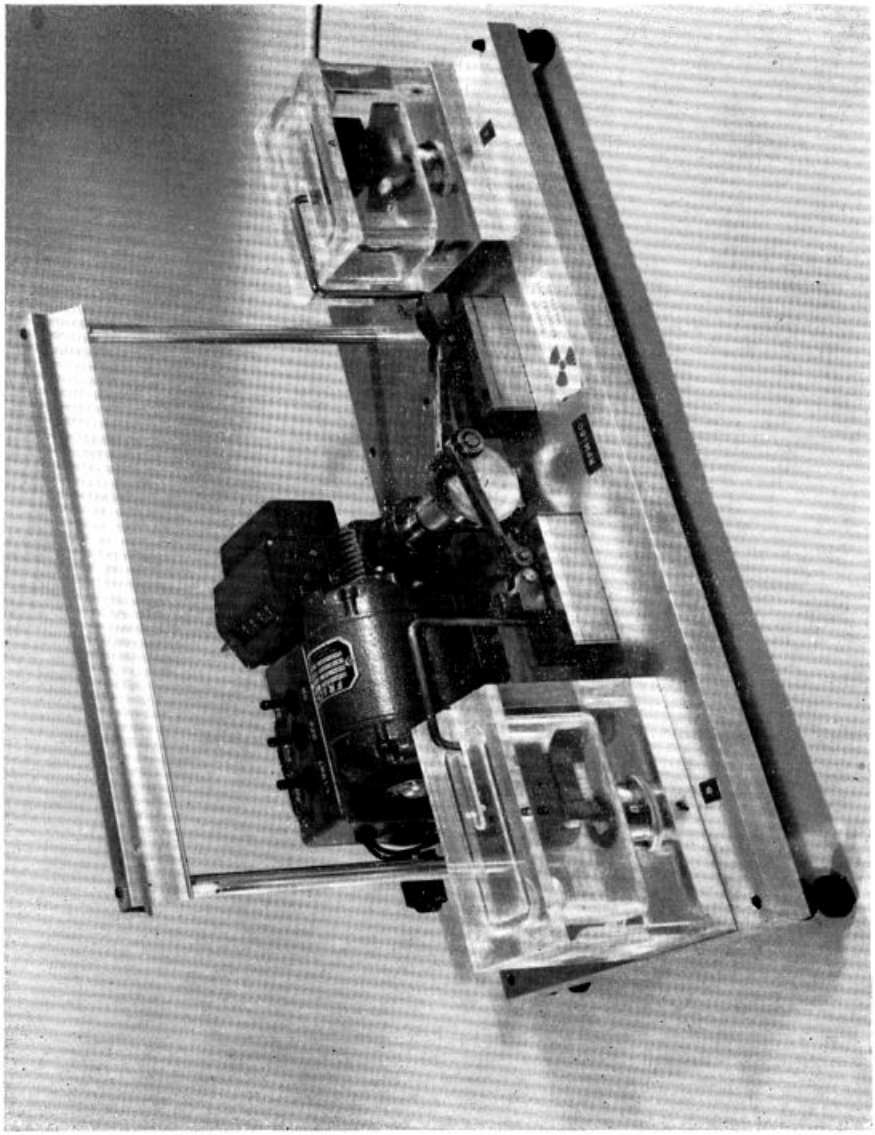

Figure 2
Dentifrice testing rig

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Experimental procedure

A diagram and a photograph of the experimental rig employed in these investigations are given in *Figs. 1* and *2*, respectively. Its construction allows a loaded brush head to reciprocate across a dental tissue mounted in the base of a *Perspex* trough used to retain the dentifrice. With a stroke of 3.8 cm and a frequency of oscillation of 2.5 c/s, the action closely simulates an oral brushing technique. The test specimen mount is cast from *Araldite MY 753* so shaped that it can readily be withdrawn from the base of the

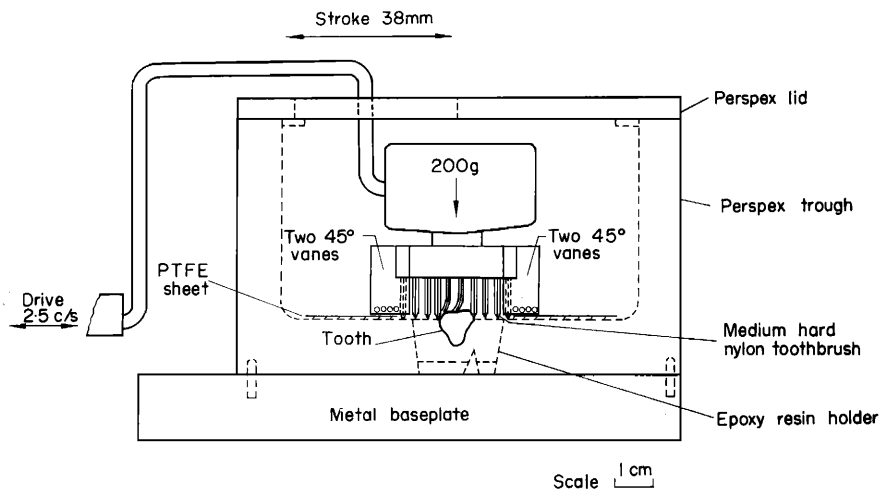


Figure 1
Diagram of dentifrice testing rig

trough for irradiation purposes. By using an epoxy resin as the mounting medium, difficulties arising from the radioactivity of the holder can be avoided. The release agent for the *Araldite* is polyvinyl alcohol.

The dental tissue, which is arranged with its upper surface projecting about 1 mm from the surface of the mount, is cut to expose approximately 0.5–1.0 cm^2 of surface to the brushing action. In the case of an enamel specimen, the labial surface of a frontal incisor is employed in an orientation that permits the brush to pass across the tooth as in normal oral brushing. This geometry ensures that the natural curvature of the tooth assists the brush to pass smoothly over the surface.

Dentine specimens are cut from Bicuspids normally oriented in the same direction as the enamel specimens. The leading edges of the specimens are

slightly rounded to prevent the brush fibres encountering abrupt changes in height.

The brush head of the machine is equipped with four perforated vanes conforming closely to the internal dimensions of the trough and so orientated as to force the slurry to pass through the brush fibres when the head is in motion. The presence of these vanes also assists in preventing sedimentation of the dentifrice abrasives.

The specimens, after mounting, are thoroughly scrubbed with a dentifrice slurry and distilled water, and then placed in a polythene capsule which is filled with distilled water and a trace of bacterial antiseptic 1% *Hibitane.* The capsule and contents are irradiated for a period of five hours. After removal from the reactor, any Na^{24} activity is allowed to decay before the specimens are inserted in the rig, but the level of this γ activity can be reduced by avoiding direct handling of the specimens whilst cleaning.

To perform a typical brushing test, a known volume of dentifrice slurry is placed in the trough and at the end of the prescribed brushing period, 1 cm^3 samples are withdrawn with the aid of a pipette fitted with a microfiller. The samples are transferred to 2.5 cm diameter aluminium planchets and dried beneath an ir lamp at a surface temperature of 105°C for one hour. In cases where excessive frothing of the slurry has occurred, it is preferable to pour the contents of the trough into a beaker and break the foam with a drop of ether. After careful restirring of the slurry a non-aerated sample may be withdrawn.

The level of β activity of the dried samples is measured with a 5 cm diameter mica end-window Geiger-Muller counter with the specimens placed within a millimetre or so of the window. With this geometry a high counting sensitivity can be obtained and errors arising from a non-uniform deposit eliminated. This arrangement also reduces the small back scattering errors which can occur if a comparison is made between dentifrices containing elements of widely different atomic number (6).

The dentifrice samples are usually taken from slurries having a dentifrice concentration of 20% or less and thus containing only 100–200 mg of solid matter when dried. At such mass thicknesses (20–40 mg/cm^2), β absorption errors can be neglected. In other tests where a high concentration of the dentifrice is employed the slurry must be diluted before taking a sample. The measured figures for the β activity are corrected in the usual way for counter 'dead time' followed by background activity, and the final result multiplied by the volume of slurry originally added to the trough. The latter may vary slightly from one dentifrice to another if weight concentrations

are used in making up the slurries. Corrections for the decay in β activity of the source may be necessary if a long time elapses between the measurements of different samples.

The dried samples are rarely active enough to give rise to any health hazard, but the irradiated tooth specimens should be handled in accordance with the approved recommendations for a high energy β source of 1 or more mCi activity. Satisfactory shielding is obtained with 1 cm of water or *Perspex*, whilst tongs of at least 25 cm length and rubber gloves are recommended for handling the irradiated samples. *Perspex* goggles should be used to protect the eyes.

By employing a water-moderated reactor, such as exists at the Scottish Research Reactor Centre, East Kilbride, for the specimen irradiation, it is possible to irradiate the specimens at temperatures below that of the human body and to keep them immersed in water for the whole irradiation period. Under such conditions of irradiation and low neutron dose, major changes in the physical properties of a tissue can be avoided.

In view of the fact that human dentine and enamel contain different proportions of hydroxyapatite, the specific activity for a given radiation dose will not be the same for the two tissues. For comparative measurements on the same tissue this is unimportant, but for experiments comparing the wear behaviour of different tissues any difference in specific activity must be allowed for. The ratio of specific activities for dentine and enamel is about 1:2, but it is usually desirable to include small fragments of the actual tissues with the main specimens so that the ratio can be accurately determined. The size of these fragments must be kept very small if the β particle absorption error is to be minimized. A mass of less than 0.5 mg is recommended.

Relationship between abrasive wear resistance of a dental tissue and its hardness

The technical problem of cleaning and polishing a surface by mechanical means presents many difficulties if it is composed of a number of elements of varying hardness. Thus the abrasives incorporated into dentifrices must assist not only in removing the relatively soft bacterial plaque and any other pellicle present, but also assist in repolishing the tooth enamel together with any cement or dentine exposed at the cervical margin.

The most efficient manner of achieving the required cleansing action

without causing harm to the main dental tissues has yet to be established. Unfortunately many of the published clinical trials (7) have employed dentifrices formulated from abrasives of very similar hardness and it has not been possible to examine the significance of the discriminating power of the dentifrice to tissues of different hardness. Thus it is not known whether a very hard abrasive compound which is likely to show a small wear rate variation with tissue hardness, or a softer compound which will accentuate the wear of the softer regions in the surface, should be employed. It might be argued that the overlying plaque and perhaps the acquired staining pellicle are relatively soft compared to enamel and therefore a soft, highly discriminating dentifrice is required; but if the surface films and the action of food and drink cause some superficial damage to the enamel, then it might be preferable to use a harder less discriminating dentifrice to repolish the surface without causing excessive wear to the softer tissues. Present-day dentifrices tend towards the first of these formulations and whilst this invariably leads to low enamel wear rates, one might ask if it is the correct formulation to use if there is any cervical exposure of dentine.

Although there are a few isolated references in the literature to the dentine/enamel wear ratio, no serious attempt has been made to study it in a systematic manner. This is largely because of the difficulty in measuring enamel wear accurately. However, the abrasive wear properties of metals and certain ionic and covalent solids, abraded with very hard compounds, are such that simple proportional relationships are found to exist between the wear resistance (1/wear volume) and hardness (8). Under these circumstances the coefficient of proportionality varies from one class of material to another.

Major deviations from these simple relationships naturally occur whenever the abrasive compound has a hardness comparable to that of the abraded solid and, in these situations, the wear-resistance/hardness plots curve sharply upwards as the hardness of the solid approaches that of the abrasive. Unfortunately, despite its technical importance, the exact form of the characteristic so obtained has never been studied in great detail.

Although it has never been investigated, it is probable that dental tissues show similar wear/hardness characteristics to metals and other solids. The very high dentine/enamel wear ratio reported in the literature for many proprietary dentifrices, is almost certainly due to a strongly curving wear resistance/hardness characteristic, arising from a limited abrasive hardness. If dental tissues fall into the same category as the other solids, then the use of a very hard abrasive compound in a dentifrice should

cause the wear resistance/hardness plot to reduce to the proportional relationship characteristic of many other materials. The distortion of the linear characteristic is a measure of the discriminating power of the dentifrice to abrade tissues of different hardness and is clearly a factor to consider in selecting an abrasive for dentifrice purposes.

In order to study this problem in greater detail, a number of test slurries were prepared from a range of abrasives of different hardness. A particle size of approximately 10μ diameter was chosen for the abrasive and the slurries were made up with a water-glycerine mixture to which a small amount of carboxymethylcellulose (CMC) was added to give a consistency similar to that of a diluted commercially available dentifrice.

Testpieces of three dental tissues were prepared: Ivory, human dentine and human enamel. The ivory and human dentine specimens were given identical geometry, but the shape of the enamel testpiece differed from the others as it was considered advantageous to preserve the natural tooth contours.

The possibility of a proportional relationship existing between the wear resistance and hardness of the dental tissues was first studied with the aid of a dilute slurry of SiC particles (5% w/w). The results of these experiments indicated a very slight upward curvature to the wear-resistance/hardness plots when suitable corrections for specific activity and nominal area of the specimens were taken into account. This curvature could not have arisen from any lack of hardness in the abrasive, but it could quite easily have arisen from the slight differences in specimen geometry. To investigate this point, additional experiments were made with a standard pin-on-disc machine employing a fixed abrasive cloth lubricated with water. The specimens consisted of small 2.5 mm pins machined from dental tissues and mounted so that the ends traversed the wet abrasive cloth in a spiral path. The change in length of the pins after passing over 3 m of cloth was measured with a micrometer and used to obtain the wear resistance of the tissue. With this machine, which is normally used for studying the abrasive wear of metals, the wear rates are higher than those usually recorded with a loose abrasive and brush loading. However, the load applied to the pins was adjusted so that the load on any abrasive particle was commensurate with that normally experienced with fibre loading. The results obtained from these experiments are shown in *Table I*.

It will be seen that slight variations in the wear resistance of ivory arise with different orientations of the tissue, but if one accepts the mean value obtained for this material, then the wear resistances of the three tissues are

Table I
Wear resistance and hardness of dental tissues

Dental tissue	Enamel	Dentine	Ivory		
			Grain parallel to track	Grain perpendicular to track	End grain
Relative wear resistance with respect to dentine	4.25	1.00	0.67	0.71 Mean 0.61	0.45
Hardness, HV2.5	280	57		28	

closely proportional to their indentation hardnesses. As there was no reason to suppose that the employment of a brush was the cause of the curvature shown by the first set of results, it was decided to correct all subsequent brushing experiments so that the wear resistances of the tissues, when using SiC as an abrasive, were strictly proportional to their hardnesses. The wear resistance values obtained with the other abrasives, Al_2O_3, SiO_2 and $CaHPO_4.2H_2O$ are shown in *Fig. 3* after correction in this manner. Although the test dentifrices have all been shown with a dentine rating of unity, the actual wear figures varied widely, being dependent upon such additional factors as particle size and concentration. However, by reducing all the data to a common dentine rating, it is possible to observe the differences in discriminating power more clearly. Of particular significance is the strong discriminating power of the $CaHPO_4.2H_2O$ abrasive, which in these experiments recorded an enamel wear resistance rating 40 times that of the much harder SiC abrasive. The inherent dangers in routine dentifrice testing with dentine specimens are immediately apparent and this practice cannot be justified if any major changes are contemplated in the composition of a product.

Owing to the limited number of dental tissues available, it is not possible to describe the exact shape of the wear-resistance/hardness curve for the dicalcium phosphate (DCP) type dentifrice, but it is clear that slight variations in the hardness of an enamel specimen will have a pronounced effect on the wear resistance recorded. The curves, given in *Fig. 3*, suggest that a 10% change in hardness of enamel might cause its wear resistance to change by as much as 50%, whereas for the harder abrasives, such as SiC and Al_2O_3, a hardness variation of this magnitude would produce only a 10% change in wear resistance. A comparison of two dentifrices of very different discriminating power in a range of hardnesses which includes that of the test tissue, will thus be highly sensitive to the actual hardness of the

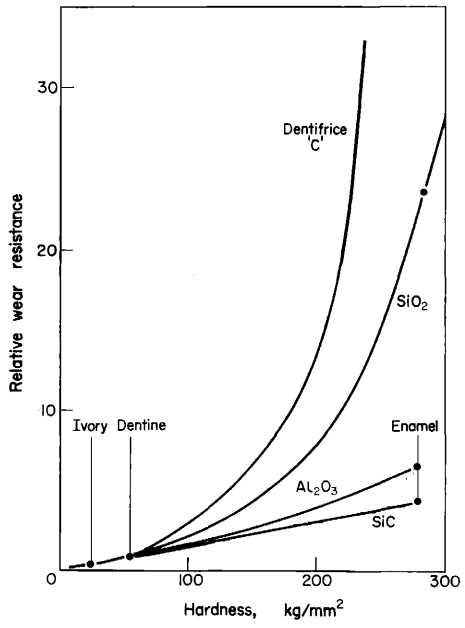


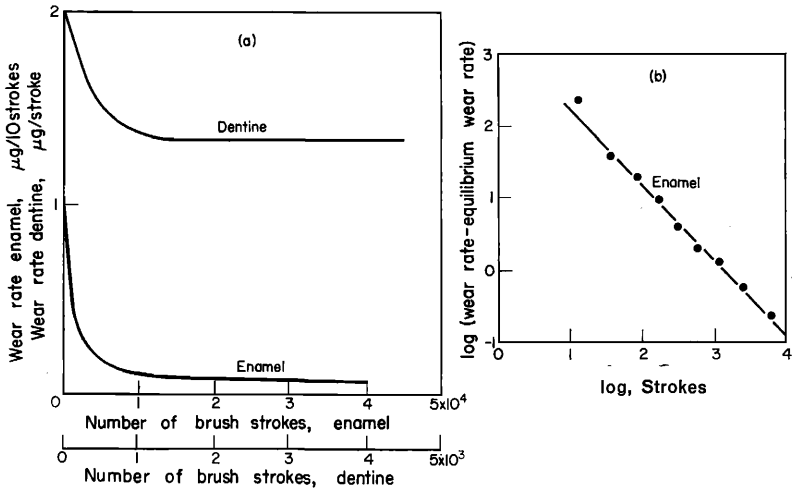
Figure 3
Relationship between wear resistance and hardness of dental tissues for different abrasives

test specimen. As it is not easy to control the hardness of dental tissues, such comparisons should always mention the actual hardness of the tissue used. Fortunately, many of the present-day dentifrices are largely based either upon calcium carbonate or on calcium phosphate and, since these two compounds do not differ greatly in hardness, slight variations in tissue hardness will not have a pronounced effect on the recorded comparative wear rates. However, the possible introduction of new dentifrice formulations, especially if their discriminating power varies with concentration, necessitates an awareness of the effect of tissue hardness. If the new dentifrice is to contain a mixture of abrasives having different hardnesses, the effect of concentration level must also be considered.

Effect of previous history on the wear rate of a dental tissue

To avoid variations in conditions during the course of a test, the test period should be kept as short as possible, by employing a highly sensitive method of wear measurement. In this respect the radiotracer technique is greatly superior to many of the other methods employed. Full advantage of

this short test period cannot always be taken, however, because the initial detailed surface condition of a test specimen can have a very large effect on the recorded wear rate. In effect the tissues have to be 'run in' until a surface texture is created which is characteristic of the particular dentifrice under investigation. A particularly marked effect is observed, for instance, if one changes from a coarse to a fine abrasive dentifrice, since it is necessary to obliterate the highly roughened surface before one can establish the true intrinsic wear rate of the finer compound. For this reason it is recommended that all wear test measurements be preceded by a fairly lengthy conditioning period often exceeding the actual duration of the test measurement run. Conditioning in this way not only allows a physical equilibrium to be established between the dentifrice and the surface it is creating, but also assists the surface to attain chemical equilibrium with its environment. The latter may be important in tracer techniques, since ion exchange between the slurry and the tissue is a possibility.

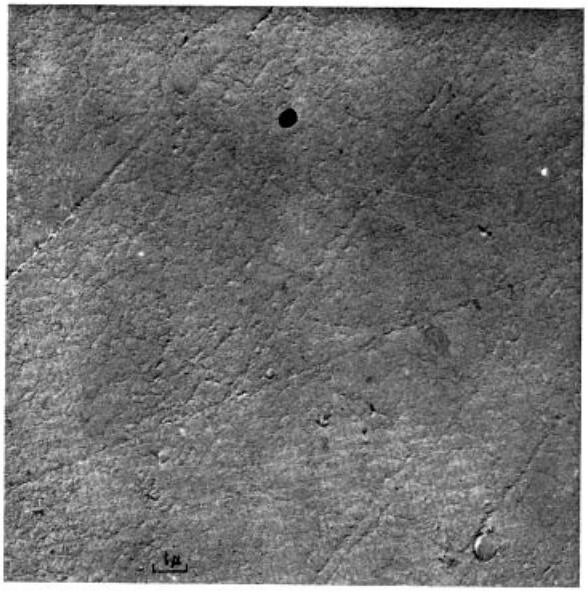


(a) Dentine and enamel brushed with 'foreign' dentifrice

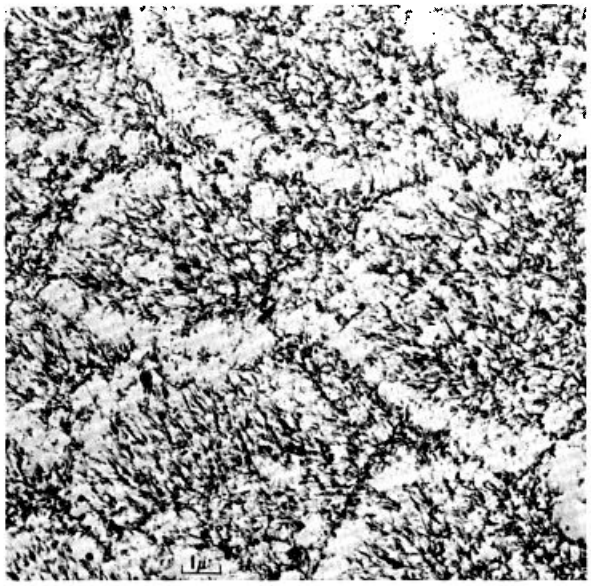
(b) Logarithmic curve for enamel after decalcification in lactic acid

Figure 4
Conditioning curves for specimen

Examples of two 'running in' tests are shown in *Fig. 4(a)* for dentine and enamel specimens. It will be seen that in these cases it was necessary to remove at least 50 μ of dentine tissue and 8 μ of enamel tissue, before a constant equilibrium wear rate was obtained. The exact amount of material and time taken to remove it will obviously depend on the initial state of



(a) before etching



(b) after etching for 10 seconds in 2.5% lactic acid

Figure 5

Electron micrograph of enamel specimen after polishing with ¼ micron diamond paste followed by brushing in water for 5,000 double brush strokes

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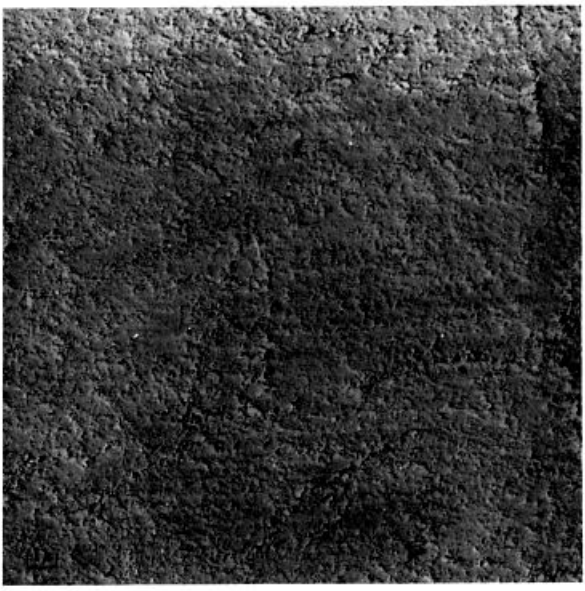
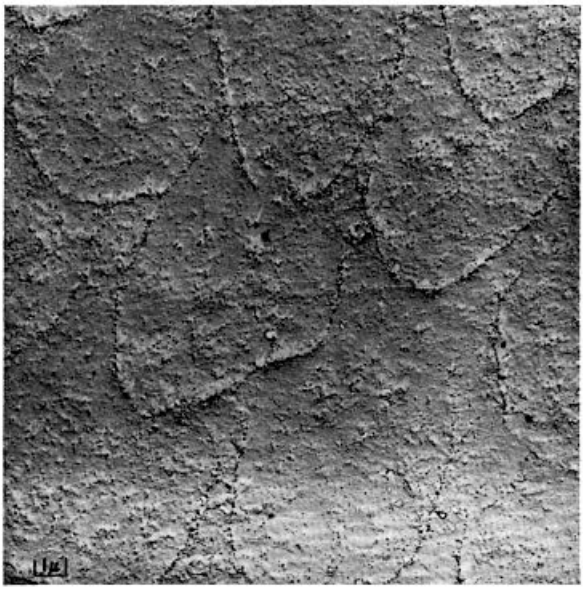


(a) surface etched with 2.5% lactic acid and brushed for 5,000 double strokes with dentifrice 'M'



(b) surface etched with 2.5% lactic acid for 10 seconds and brushed for 5,000 double strokes with dentifrice 'C'

Figure 6

Effect of dentifrice on etched enamel surfaces

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the surface and the dentifrice used, but the values of 1,000 and 10,000 brush strokes, for dentine and enamel respectively, can be regarded as fairly extreme values, and are satisfactory conditioning periods for most dentifrice testing. A detailed examination of the shape of the 'running in' curves suggests that the enamel wear rate, dW/dS, is given by an expression of the form $dW/dS=a/(S+B)+c$, where S is the number of brush strokes, c the equilibrium wear rate and a and b constants for a given tissue surface condition and dentifrice. Dentine seems to show a slightly different form of conditioning curve, the wear rate decreasing inversely with the square of the number of brush strokes, but more work is required to establish whether or not this is always true. In this respect it should be remembered that the tracer technique yields information only on the volume of inorganic tissue removed and that, in certain cases, it will be difficult to relate this quantity to layer thickness.

Although in the physical testing of dentifrices it is desirable to measure intrinsic abrasiveness, in practice their properties will depend both on previous history and environment. Apart from the aspects already discussed the teeth will come into contact with acid products of the mouth and some decalcification may take place. This type of exposure can be expected to increase the initial wear rate not only by virtue of the surface roughening that occurs, but also by the damage done to deeper tissues.

An example of a highly polished enamel surface, both before and after exposure to $2\frac{1}{2}\%$ lactic acid, is shown in the electron micrographs of *Figs. 5(a)* and *5(b)*. This same decalcified surface after brushing for 5,000 strokes with two different dentifrices is depicted in *Figs. 6(a)* and *(b)*. It will be seen that this amount of brushing has eliminated most of the surface detail brought out by the attack, but the prism structure of the enamel is still evident after brushing with the milder of the two dentifrices. The equilibrium structure for this dentifrice, which is based upon dicalcium phosphate, is shown in *Fig. 6(c)*.

These changes in surface detail are accompanied by a progressive decrease in the wear rate as was also the case for the mechanically prepared tissues. However, decalcification can lead to extremely high wear rates; thus a 10 min exposure to 5% lactic acid can result in the initial wear rate being 2,500 times greater than the final equilibrium value. This high level of wear was recorded during the first 25 brush strokes and indicates that the top surface layer is in a highly friable condition. The conditioning characteristic for this tissue when polished with the $CaHPO_4.2H_2O$ type dentifrice is shown as a double logarithmic plot in *Fig. 4(b)*. The form of the curve is

similar to the conditioning curve obtained for a mechanically prepared enamel surface. As many soft drinks contain citric acid, the damage caused by excessive consumption of this form of refreshment requires further study.

Not all 'running in' phenomena experienced during the testing of dentifrices can be attributed to such factors as mechanical or chemical damage to the surface, since a very similar effect can be observed with carelessly prepared enamel specimens. As many dentifrices are highly discriminating towards tissues of different hardness, even a small percentage of dentine remaining on a prepared enamel specimen can greatly increase the initial wear rate; for example 1% could quite easily double it. This effect is greatly accentuated by the use of a brush, since this permits the softer regions of the surface to be indented below its mean level before the fibre loading in these regions is sufficiently lowered to give the same wear rate as the rest of the surface. The use of a rigid flat lapping plate to load the abrasive will clearly give a much reduced 'running in' effect, since the load distribution will rapidly readjust itself to give a uniform wear rate over the whole surface. The measured wear rate of the surface in this equilibrium condition will be given by the additivity rule

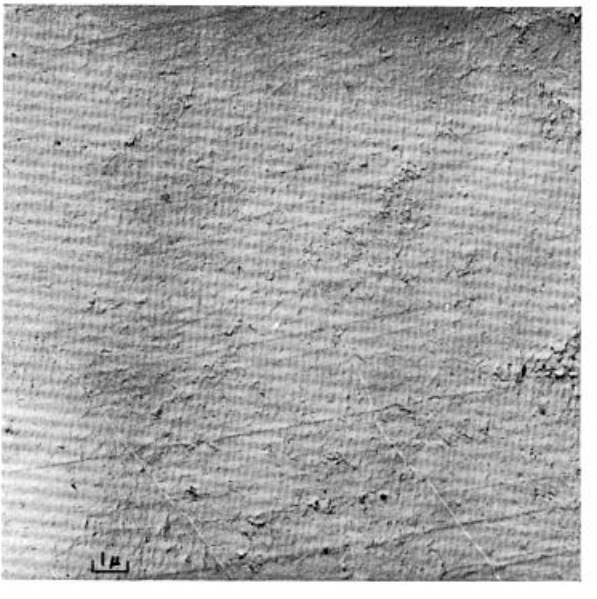
$$\varepsilon = \sum_{1}^{n} \varepsilon_n v_n,$$

where ε_n and v_n are the wear resistance and volume concentration of the n^{th} component of a heterogeneous surface (8). The presence of a small percentage of dentine will now have very little effect, even with a highly discriminating dentifrice.

The same selective removal of the softer tissues will apply even to sound enamel as there will be slight variations in its surface hardness. In the case of a highly discriminating dentifrice this will be recorded by a pronounced roughening of the surface and lack of lustre. A very hard abrasive compound such as alumina, will reduce this degree of mechanical 'etching' of the surface and yield a higher lustre value than abrasives such as chalk or dicalcium phosphate dihydrate, which are commonly used.

The influence of abrasive concentration on tooth wear

For highly angular abrasive particles and relatively small depths of surface indentation, the overall wear rate of two sliding surfaces separated by a layer of the particles will be, to a large extent, independent of particle



(c) equilibrium enamel surface for dentifrice 'C'

Figure 6
Effect of dentifrice on etched enamel surfaces

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concentration. Under these circumstances each particle's contribution to the total wear is directly proportional to the fraction of the load it carries.

If one of the sliding surfaces is replaced by a brush, the individual fibres will transmit a constant fraction of the load which is independent of the presence of any trapped abrasive material between the opposing surfaces.

Assuming that the fibres themselves cause little or no wear to the opposite surface, the wear rate will become sensitive to particle concentration and will depend on the probability of trapping one or more particles within some 'region of influence' surrounding each fibre tip.

The density of particles in a dentifrice slurry is extremely high, but because of the small diameter of the fibre the trapping probability is low and may be derived from the Poisson approximation to the binomial theorem. The general term of this approximation is

$$\frac{e^{-nv}n^r v^r}{r!},$$

where n=population density of the abrasive particles,
v=volume of the 'region of influence', and
r=number of particles trapped by a fibre.

If one makes the further simplification that the wear rate will be independent of the number of abrasive particles trapped by each abrasively loaded fibre, then the wear rate will be determined by an expression of the form $wN(1 - e^{-nv})$, where e^{-nv} represents the first term of the Poisson approximation, namely the probability of trapping no particles (r=0), w the intrinsic wear rate of an abrasively loaded fibre and N the total number of brush fibres. The assumption that the wear rate of an abrasively loaded fibre will be largely independent of the number of abrasive particles trapped is justified on the basis of the load-sharing property of an individual fibre.

As the population density of the abrasive particles will be proportional to the dentifrice concentration (vol/vol), C, the relationship between wear rate, dW/dS, and dentifrice concentration can be expressed in the form $dW/dS=\alpha(1 - e^{-\beta C})$ where α represents the maximum wear rate possible with all the fibres loaded in an abrasive capacity and β a measure of the ratio of the trapped volume of slurry at the fibre tip and the volume of an abrasive particle. Although the trapped volume will not remain perfectly constant throughout a brush stroke, an approximate value is given by the volume of slurry entrained by a fibre and the tooth surface when the two are in contact. The boundary of the trapped volume will be defined by the

position where the separation of the two surfaces is just sufficiently wide to accept an abrasive particle. In the case of a spherically ended fibre, this volume is approximately equal to $10/9\pi Rd^2$, if $R \gg d$, so that $\beta \simeq 7R/d$, where R is the radius of the fibre section and d the diameter of the particle.

Comparisons of some experimentally derived values of β and those obtained theoretically from the known geometry of the fibre and particles are given in *Table II*. The calculated values have been corrected to allow for the small amount of elastic deformation of the fibre-tooth contact and the embedding of the abrasive particles. The former extends the boundary of the active zone, whereas the latter tends to diminish it. This correction is quite large for the finer abrasives and causes the β values for dentine to be somewhat greater than those for enamel. This trend is followed in the experimental results obtained with Al_2O_3.

Table II
Values of β

Abrasive	Manufacturer and product	Particle diameter (diameter corresponding to mean volume, $(\bar{d}^3)^{\frac{1}{3}}$ all particles $>1\,\mu$	Values of β (R=100μ)			
			Experimental		Theorical	
			Enamel	Dentine	Enamel	Dentine
Al_2O_3	Griffin & George (5/20)	3 μ	130	168	290	390
$CaHPO_4.2H_2O$	Albright & Wilson (SM)	12 μ	30	30	59.0	59.5
$CaCO_3$	Gibbs Proprietaries (Waterworks Chalk)	15 μ	8	8	47.5	48.0
SiC	Carborundum Company (700)	(2 – 20 μ)	(400 – 13)		(650 – 35)	

It will be seen that the experimentally derived values for β confirm the predicted dependence on the diameter of the abrasive particle, but show a slightly stronger dependence than that suggested by the spherical tip geometry. Although other forms of fibre tip are conceivable, they usually lead to a reduced dependence on d. Thus a conical tip would yield β values which were independent of d, whilst an inclined cylindrical form in contact with a plane surface would cause β to vary with $d^{-\frac{1}{2}}$. Microscopic examination of the actual fibres shows them to be rounded, but with a surface that

might be better described as an oblate spheroid. As the fibres will be inclined to the tooth surface when brushing, the effective radius of the tip will be slightly less than that of the fibre cross-section. This may explain why the experimental values of β are smaller than the theoretical values. It is also likely that the diverging region of the trapped volume will be less efficient in causing particle abrasion than the converging leading zone and this factor alone could reduce all the theoretical β values by a half (tiddly-wink action). Bearing these factors in mind, the agreement between theory and experiment is remarkably good.

It is interesting to note that over the usual dentifrice concentration range employed in oral brushing, approximately 1–5 particles are trapped by each fibre, assuming particle sizes ranging from 14–10 μ. Thus one sweep of a brush containing 1,500 fibres might be expected to cause the whole surface to be traversed once by abrasive matter and 15 strokes should be adequate to remove a soft overlayer of 10-50 μ thickness.

The above theoretical analysis applies only to a mono-disperse system where all the particles are assumed to be of approximately equal size and abrasiveness. In practice, dentifrices contain abrasive particles of a fairly wide range of diameters and, in some cases, mixtures of compounds of sharply contrasting abrasiveness. The combination of these two factors can lead to a variation of β with concentration level. Consider, for instance, a mixture of finely divided and coarse particles of similar abrasiveness. Initially at low concentrations the finer material will dominate the wear rate/concentration characteristic, but as the dentifrice concentration is increased, the coarser material will prevent the fine abrasive playing a major role and β will decrease. Such an effect is probably occurring with the SiC abrasive used to obtain the result shown in *Fig. 7*. In this case there was a wide range of particle sizes present and a simple exponential function did not adequately describe the results. Initially, the magnitude of β was in accord with the value one might associate with the very finely divided component of the abrasive but this slowly decreased to 13 which is a value more typical of coarse particles of about 14 μ. These results do not take into account the actual concentrations of the various components and thus tend to give rather low values for β.

In practice, advantage could be taken of this ability of a dentifrice to make use of different particle sizes at different concentration levels. Thus a dentifrice, composed of a mixture of a very fine hard abrasive compound and a coarse soft material, would exhibit a discriminating power that varied with the actual concentration of the dentifrice being used. If such a denti-

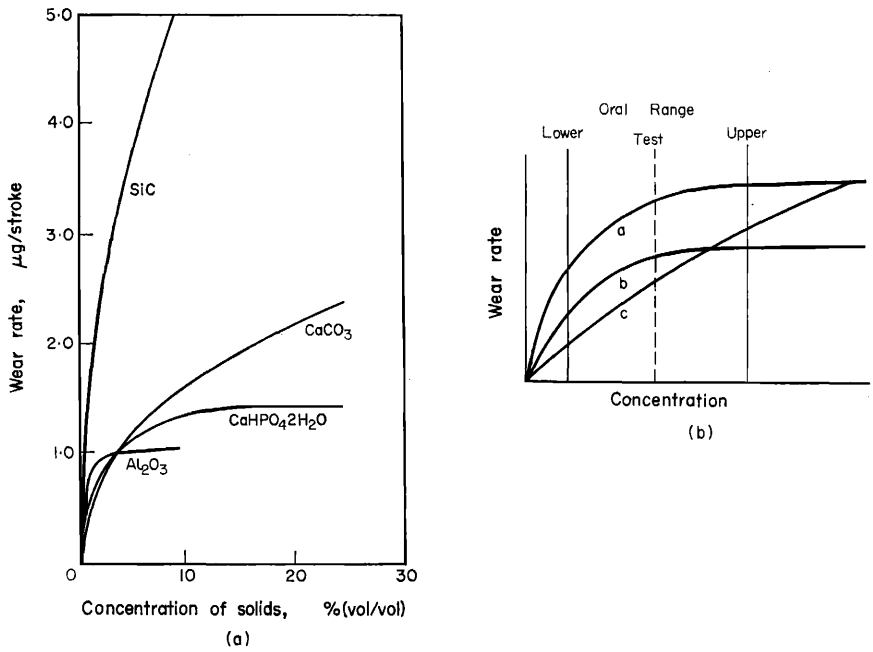

Figure 7
Dental tissue wear rate versus concentration of dentifrice solids
(a) Typical curves
(b) Curves to emphasise danger of selecting an arbitrary level of dentifrice concentration

frice were orally applied, the soft coarse material would come into action first and probably be very effective in removing soft thick overlayers from the teeth surfaces. Later, as the dentifrice was diluted with saliva, the hard finely divided material would still exhibit a high trapping probability and enable the compound to provide a high surface lustre to the teeth and keep the dentine/enamel wear ratio to its minimum value. An alternative and equally satisfactory procedure would be to incorporate the hard finely divided abrasive into the actual brush fibres during manufacture.

All the above arguments are based upon the assumption that the fibre tip geometry allows the brush to differentiate between particles of different diameter. If the geometry were such that β was independent of particle size, then the variable discriminating property described above would be much reduced in magnitude. This sensitivity of dentifrice abrasiveness to fibre geometry is such that some account of it must be taken in dentifrice testing.

It is equally apparent that, in dentifrice testing, proper consideration must be given to the range of concentrations covered during oral usage. The common practice of comparing dentifrices at some arbitrary concentration level cannot be justified in the light of the present experimental data. For example, examination of *Fig. 7(b)* clearly shows that although two dentifrices with characteristics such as 'a' and 'b' are directly comparable at any concentration level, this situation will rarely arise and, in most cases, one will be attempting to compare dentifrices with characteristics such as 'b' and 'c'. Furthermore, although many of the dentifrices investigated show a gradually saturating wear characteristic, some dentifrices were found to exhibit a sharp falling off in abrasiveness at high concentration levels. This effect could have arisen from the interplay of a mixture of hard and soft abrasives, but it is generally felt to be due to the stiff paste-like qualities of these dentifrices preventing full tissue coverage during brushing.

The only satisfactory solution to this problem is to resort to an integrated wear test, which takes full account of the changes in dentifrice abrasiveness over the range of concentrations normally experienced during oral brushing. As the dentifrice concentration is not itself linearly related to the number of brush strokes, this test must take the dentifrice dilution function into account. If one assumes that saliva is produced at a constant rate under the stimulation of the brushing action, then the relationship between concentration, C, and number of brush strokes, S, will be of the form $C=a/(1+bS)$, where a and b are constants. a is determined by the initial concentration of the dentifrice, which is generally less than 100% owing to the habit of wetting the brush before use, whilst b reflects the rate of saliva production.

For practical testing purposes, the integrated dentifrice abrasiveness is best obtained by simply allowing the dentifrice to be continuously diluted in a fashion that reflects the oral condition, but adjusting the rate of dilution to give the required final concentration, usually about 20% (w/w) (9). The initial concentration is similarly adjusted to correspond to a wetted brush.

As the continuous addition of diluent is not easy to arrange in practice, a satisfactory alternative is to add the liquid at a few set times during a test. A suitable liquid, having a similar viscosity to human saliva, is obtained by adding a small quantity of CMC to water (10). The level of radioactivity of the slurry at the end of the test then yields a measure of the integrated dentifrice abrasiveness under oral conditions of usage.

Conclusions

1. It has been shown that the radiotracer technique can provide an extremely rapid assessment of the abrasiveness of a dentifrice and can be made so sensitive that it is possible to measure transient wear rates arising from changes in surface contour or environment of the dental tissue. If necessary it is possible to measure the wear caused by one or two strokes of a brush on dentine polished with a commercially available dentifrice.

2. The results obtained with different tissues suggest that their wear behaviour is similar to pure metals or other materials in that their abrasive wear resistance is proportional to indentation hardness when very hard abrasive compounds are used. In other cases where the dentifrice abrasive has a hardness comparable to that of the tissues, the wear resistance increases rapidly as the tissue hardness approaches that of the abrasive and the dentifrice exhibits a strong discriminating action towards different tissues. This situation applies to most proprietary dentifrices and accounts for the large dentine/enamel wear ratios and the sensitivity of enamel wear resistance to its hardness.

The possibility of much harder abrasive compounds being used in dentifrice formulation makes it imperative to incorporate into any test specification the requirement that enamel as well as dentine be used as a test material. Failure to do this could lead to the production of dentifrices which were excessively abrasive to enamel.

3. A detailed examination of the tissue wear/dentifrice concentration curves suggests that the form of this relationship is closely associated with the brush fibre tip geometry. In the case of mono-disperse systems the controlling factor appears to be the ratio of the volume of the active collection region of the fibre tip to that of an abrasive particle. However, in most practical cases, the former increases with particle diameter and the sensitivity of the wear/concentration curves to the size of the abrasive particles is reduced. As a result the level of dentifrice concentration at which tissue wear saturation occurs varies but slowly with particle diameter. The effect could be enhanced by a change in tip geometry, but most commonly used geometrical forms tend to minimise it.

This variability stresses the need to standardise brush fibre geometry in dentifrice testing and to employ an integrated test to take account of the wide range of dentifrice concentrations that occur in oral use. The fact that some dentifrices often show a marked falling off in abrasiveness when used at high concentrations strengthens the case for this form of assessment.

The above considerations also clearly show that lapping pad machines should be discouraged, since they cannot reproduce the interaction between the brush fibres and the dentifrice abrasives.

4. It has been well established that in situations where there has been a change in environment or surface contour, it is important to carry out a conditioning run prior to any test evaluation of a tissue/dentifrice combination. This precaution is particularly necessary when a radiotracer technique of wear measurement is employed, since the number of brush strokes required to obtain a measurable amount of wear is small in comparison to other test methods. This high sensitivity of the radiotracer technique makes it particularly valuable for future investigations of the 'running in' phenomenon.

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DISCUSSION

MR. F. R. RIDGWAY: You have described the wear rates of enamel in terms of particle size and hardness, but you have not considered the reactivity of the materials with the enamel itself. You irradiated the teeth producing active phosphorus. It seems to me that all you may have described is the solubility effects of the materials on the phosphorus in the enamel, and that the three curves in *Fig. 7*, excluding that of silicon carbide, just measure the reactivity or solubilising effect of the abrasive on the phosphorus in the enamel. If you are going to carry out a comparison in that way

should you not have used a material of differing particle size and hardness but having the same chemical reactivity to the material you are examining?

DR. WRIGHT: It would certainly have been better to have done this, but the data was acquired as part of a programme to study the abrasiveness of commercially available dentifrices and the theoretical interpretation of the results followed later. The alumina type dentifrice was added primarily to study the relative behaviour of enamel and dentine tissues towards an abrasive of very high hardness.

The question regarding the solubility of the tissue has to be answered in two parts: first, does the reactivity or solubilising effect of the dentifrice play a significant role in determining the wear rate of a dental tissue in the absence of radiophosphorus in its structure, and second, does the presence of radiophosphorus lead to real changes in solubility of the structure or possibly lead to a false indication of the actual wear rate as a result of ion exchange between the P^{32} in the dental tissue and the phosphate ions in the slurry.

In reply to the first of these possibilities, I doubt very much whether solubility effects enter into the wear process with the current dentifrices. None of the dentifrices was acidic in character, in fact they would all be expected to have pH values close to 8.

The second possibility requires a more detailed examination, since the recoil action associated with the trapping of a thermal neutron may very well lead to highly localised changes in tissue structure. However, the total volume of the tissue affected in this way must be very small since the concentration of P^{32} atoms is only about $10^{-7}\%$ of the total number of phosphorus atoms present. One can therefore dismiss the argument that there is an overall *real* change in the solubility of the tissue. More permanent and extensive damage can result from excessive heating of the tissue during irradiation, but this is avoided by using a water moderated reactor.

Although the overall solubility may not be changed, one does have to remember that the wear process is recorded by the release of P^{32} into the dentifrice slurry and hence it is the local wear resistance or solubility of the regions around the radiophosphorus atom that is relevant. On the short time basis, the rate of removal of these localised regions of tissue might very well differ from the rate of removal of the unchanged lattice and, indeed, perhaps exert some control on the latter. However, in the presence of mechanical action, insoluble products will not accumulate on the surface of the tissue and it is reasonable to assume that the solubility (and wear rate) of the unchanged tissue will not be affected, and will be adequately monitored by the release of P^{32} in the slurry.

Again the abrasive wear resistance of the tissue is unlikely to be changed by the presence of small quantities of tissue converted into a different structure by the recoil action. Most of the abrasive particles will engage groups of atoms greatly in excess of the small number associated with the recoil process.

A third possibility arising from the use of P^{32} as a tracer element is that ion exchange between the P^{32} in the tissue and phosphate ions in the slurry will lead to a surface depletion of the radionuclide. The extent of the reduction will depend upon the nature of the slurry and the time of exposure. In order to counteract this possibility we recommended that all tissues be 'conditioned' in the dentifrices slurries prior to the performance of the actual test. In this way one establishes an equilibrium P^{32} distribution in the surface layers of the tissue which is the same at the beginning of the test as at the end. The total amount of P^{32} entering the slurry, partly by

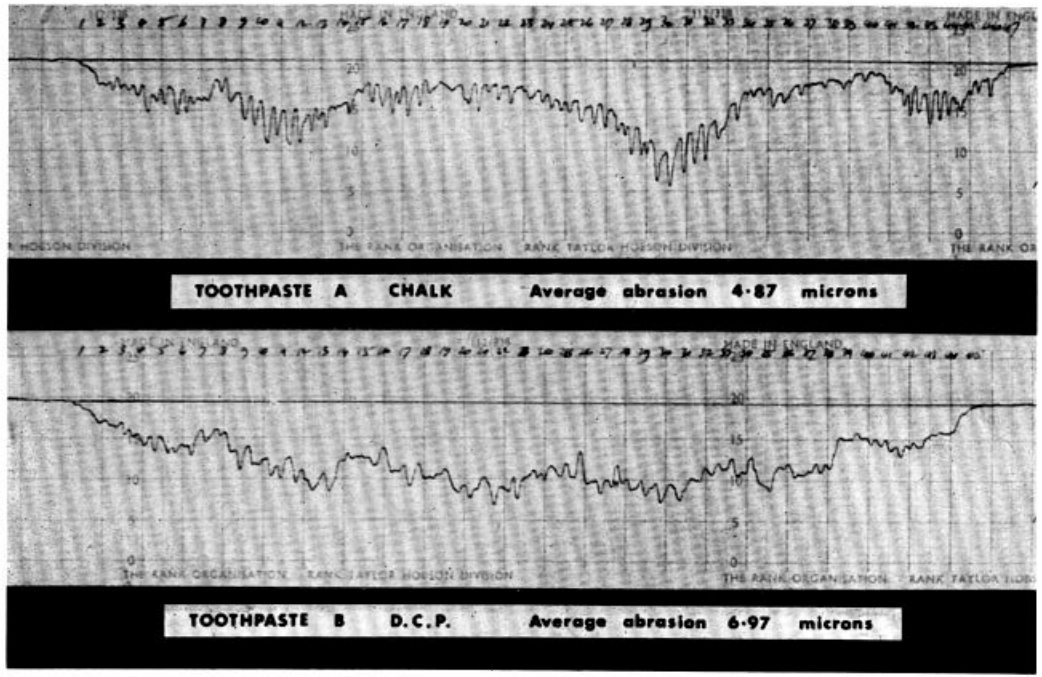

Figure 8
Traces obtained with the Talysurf surface profile instrument showing the effect on dentine.

mechanical action and partly by ion exchange, will always correspond to the amount that would have entered the slurry in the absence of ion exchange processes.

MR. A. I. FREW: What is the effect of the viscosity of the toothpaste sample on the chances of trapping particles between brush fibre and tooth surface, and hence the degree of abrasion recorded?

DR. WRIGHT: The viscosity of the dentifrice slurry does probably play some part in determining the form of the wear-concentration curves, especially towards the higher concentration levels. In one case, a commercial dentifrice product was observed to show a marked falling off in the level of abrasiveness when the dentifrice slurry concentration exceeded 80%. This, we believe, was due to the slurry being physically displaced from the dental tissue, thus reducing the concentration of abrasive matter. It is possible that the viscosity of the dentifrice has some effect at lower concentration levels of the dentifrice, but it might be necessary to make use of elastohydrodynamic theory to calculate the magnitude of the effect, since elastic deformation of the soft nylon fibres and the dental tissue cannot be neglected.

MR. N. J. VAN ABBÉ: You have not mentioned the effect of hydration on the brush fibre, which can possibly have quite a marked effect, but on the other hand, you have stressed conditioning of the tooth surface. However, there is no sign that *in vivo* the equilibrium condition of abrasion on the tooth is ever reached; hence it seems to me totally unrealistic to produce the equilibrium surface before testing. I would suggest that it is better to start with an optically flat surface on every brushing run and to resurface between brushings. Another factor that should be taken into account is the aggregate size within the dentifrice, rather than particle size of the polishing agent.

I would like to stress that the tracer method in common with weight loss methods only measures average wear; on the other hand, a profile method shows the detailed pattern of wear. In *Fig. 8* wear is shown by the area under the curve. The average depth of abrasion due to dicalcium phosphate is nearly 50% more than that due to the chalk dentifrice but the primary texture is different and this may be the critical factor with regard to conditioning. If the specimens had been conditioned to equilibrium initially, the relative depths of abrasion might well have been reversed, but this would not necessarily correspond to the natural effects of tooth-brushing.

You have mentioned the grittiness test using the nickel coin and glass slide; I have tried to replicate some of your results but mine have differed considerably from yours; it seems to me that there are numerous problems to solve before this test can be obtained as a grittiness standard.

DR. WRIGHT: We make no great claims for the coin and glass slide technique, although it is a very simple and yet sensitive method for detecting the presence of small concentrations of gritty matter in dentifrices. Despite the fact that it forms the basis of the U.S. Federal Standard Test for Dentifrice Abrasiveness, it is not a satisfactory way to measure the overall abrasiveness of a product.

As a grittiness test it clearly works best whenever the gritty material is present as a few large particles, but as the particles diminish in size, it becomes more difficult to decide whether the glass slide has been scratched or finely polished. By its nature the test will be subjective. The present visual criterion meets the requirement that the

gritty particles shall not disfigure an enamel surface (approx. as hard as soda glass), but not all are agreed upon the levels of disfigurement which are acceptable and, before we can even attempt this, it is necessary to devise some measurement technique whereby one can measure the severity and frequency of the scratch marks. Profilometer type instruments can be used to record the nature of the surface damage, but such traces still require interpretation.

I agree that the radiotracer technique cannot provide any information about the surface texture of the dental tissue, indeed it has never been claimed to do so. Such information can readily be obtained from a supplementary experiment using either a profilometer or optical reflection technique. One assumes that in designing a new dentifrice formulation, the first requirement that must be met is that it does not cause undue damage to the main dental tissues. When this feature has been settled, one can then proceed to examine the quality of the surface texture that it produces. If both of these factors can be examined simultaneously so much the better.

The question of particle aggregation is clearly important and must have some effect on the form of the wear-dentifrice concentration curves. In our theoretical model, the capture probability will now correspond to the aggregated particle size and not to the individual dimensions of the components. This will mean that the wear-concentration curves will tend towards those appropriate to large single particles. On the other hand, the aggregates will break up under the normal and sheer stresses imposed by the loaded fibres, so that the surface finish produced on the dental tissue is likely to be superior to that which would normally be observed with large single particles of abrasive.

When considering the problem of the initial surface preparation of the dental tissues, one has to bear in mind that the range of surfaces likely to be encountered *in vivo* is so wide that one could always argue against the choice of any particular surfacing technique. Whenever one abrades a surface, the topography of which is 'foreign' to that which would be appropriate to the abrasive employed, the wear rate will slowly change until the equilibrium surface texture for that abrasive is obtained. In our paper we refer to this as the conditioning process. If one includes this portion of the wear process in an experimental run, one finds that the average wear is a function of the time of the experiment. This is clearly undesirable unless the wear process closely replicates that which occurs *in vivo* and this would seem impossible to attain at the present moment. It, therefore, appears better to employ an abrasively conditioned surface and to measure the intrinsic abrasiveness of the dentifrice. Such a measurement is independent of the length of test and enables a range of different measurement techniques, perhaps of different sensitivity, to be employed for evaluation purposes. In the case of the tracer technique, this procedure also allows the surface to reach chemical equilibrium with its environment.

DR. B. R. PUGH: We have examined the effect of "vane" size on brushhead and found that vane dimension affects the concentration curves quite markedly. Do you believe that your hypothesis regarding combinations of small and large particles is therefore only theoretical?

DR. WRIGHT: I think you are aware that our equipment is fitted with stirring vanes that closely conform to the internal dimensions of the trough. These stir the dentifrice slurry vigorously and are, we believe, completely effective in preventing sedimentation. We have so far only employed two designs, both of which yielded essentially the same results. It is, however, difficult to assess the true efficacy of any vane design as

this can really only be ascertained by carrying out the test with different periods of brushing and extrapolating the results back to zero brush strokes. If sedimentation is occurring then this will show itself by producing a gradually increasing wear rate as the number of brush strokes diminishes.

I agree that if sedimentation of the abrasive solids in the dentifrice slurry had been occurring in our tests, then the shape of the wear-concentration curves would have been affected. Clearly large particles would tend to settle more rapidly than fine material, especially with the more highly diluted dentifrice slurries, and this would tend to produce differences in the wear-concentration curves of the type we have recorded. However, I repeat that we do not believe that sedimentation plays an important part in our experiments, not only because of our vane design, but because of the brief period of brushing which is necessary when a radiotracer technique is employed.

DR. B. R. PUGH: We found in practice that the curves of all dentifrice type materials which we have examined are identical; we do not get any very large variation such as you have described.

DR. WRIGHT: I think your tests have been confined to the normal range of products currently on the market. We have increased the range of particle sizes considerably in a deliberate attempt to assess the magnitude of the problem, since the object of our experiments was to devise a test procedure that would be capable of assessing almost any type of dentifrice that might be marketed.

DR. B. R. PUGH: Does irradiation affect tooth hardness?

DR. WRIGHT: We do not think that the irradiation dose of 5 hr at a thermal neutron flux level of 10^{12} neutrons/cm^2/s is likely to affect the hardness of the tissue. One must remember that the number of P^{32} atoms produced by such a dose will only amount to about 10^{-7}% of all the phosphorus atoms present. However, it is important to ensure that the temperature of the tissue does not rise, say above 100°C, since in such circumstances there may be a gross change in the structure of the tissue. In our experiments, the test tissues were always irradiated under water in a water moderated reactor, the temperature of which never exceeded 30°C. Hardness tests did not show any increase in hardness following irradiation and this result is in accord with the findings of Grabenstetter (5).

MR. C. PUGH: It can be shown in studying the wear of mixers by toothpaste that the abrasion markedly decreases as the hardness of scraper blades increases from very soft plastic to very hard plastic or metal. It seems likely that this is due to imbedding of particles in the blade, converting it to a sort of sandpaper structure. This may well be relevant to your determination of the sphere of influence of the bristles and also show differences between different bristle materials.

DR. WRIGHT: This is interesting since it clearly represents the two-dimensional case of the more general problem of a fibre tip contact. So long as the saturation levels of dentifrice wear have not been reached, one would certainly expect the softer blades to produce more wear than the harder materials. In practice, this might be accentuated by permanent trapping of abrasive particles in the softer grades of materials, but I rather doubt if the effect would be very large in the case of dentifrice abrasives.