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Characterization of some proprietary soft lining materials

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The physical and mechanical properties of a series of commercial denture soft lining materials have been investigated. The materials were selected to provide a representative sample of the materials widely used for this application. A total of seven products were evaluated including established and widely used acrylic and silicone materials as well as newer polyphosphazine and fluoroelastomer materials. The objective of the study was to determine minimally acceptable and desirable levels for each property which could be used as criteria or standards for the selection of proposed new materials. The results identified several areas requiring attention and future work. In particular, the importance of using fully-water-equilibrated specimens was emphasized for acrylic polymers where large property changes occurred between wet and dry conditions, and in the case of the polyphosphazine materials where the long time required for full equilibration may lead to underestimates of the changes involved. Based upon the properties as measured, recommendations for appropriate standard level for each property are offered.

1. Introduction

Dental soft lining materials are widely used as aids for the treatment and prevention of localized areas of painful tissue irritation under dentures. Although materials of this general type have been available for many years, there has been a continuing clinical demand for improved materials. Many products have been offered for this use, most of which have been either silicone elastomers or plasticized soft acrylics. Neither of these two types of materials have proven fully satisfactory [1–3].

The silicone materials have the reputation of having low tear strength and high notch sensitivity [4]. It has been difficult to finish and recontour these materials, or to remove mould marks and other processing defects without leaving them susceptible to cracking or tearing. The plasticized acrylic soft liners, in contrast, are considered to be too hard and inelastic and have problems with gradual loss of plasticizer resulting in further increases in hardness [5, 6].

The limitations of the available silicones and acrylics have led to continuing efforts to modify and improve these materials, and to frequent introduction of new and experimental materials for this use. Among recent introductions, products based upon polyphosphazine polymers [7] and fluoroelastomers [8] have gained some popularity. The properties of these materials have not yet been extensively investigated although some possible problems with excessive water sorption by polyphosphazine have been reported [9].

This research was undertaken as part of a programme dedicated to the identification and development of improved new materials for use as soft denture liners. It was felt that the accumulated experience in the clinical use of existing commercial products with the associated appraisals of the adequacy or deficiency of particular properties, could be combined with a careful laboratory evaluation of these characteristics to provide minimum standards for each property to be used as criteria for the evaluation and acceptance of proposed new materials. This paper reports the physical property test results for a representative selection of current commercial products.

2. Experimental procedures

A series of commercial soft lining materials were selected as representative of the types in widespread current use. These materials were subjected to a variety of tests considered relevant to clinical use. The results of the dynamic mechanical analysis for these materials have been reported previously [9]. Here we report the findings of other properties.

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2.1. Materials

The materials chosen for evaluation were selected to include representatives of each major type of proprietary material plus a limited number of potentially interesting newer types. The products tested are listed in Table I, which is limited to materials intended for use as permanent soft liners. Thus functional impression materials and reparative materials such as those used primarily for intra-oral relining procedures have been omitted.

2.2. Methods

The characteristics evaluated were water sorption, solubility, indentation hardness, tensile strength and elongation, and tear and peel strength. The methods chosen in each instance were standard methods previously utilized in the evaluation of such materials to facilitate comparison with previously obtained data [9–16]. In each instance the soft lining materials were polymerized in accordance with the manufacturers directions. For all tests except the peel test the materials were polymerized between glass plates in the form of sheets approximately 13 cm square and 1–1.5 mm thick. Specimens of appropriate size and shape for each test were then cut from these sheets. For the peel tests 3 mm thick layers of the soft lining material were cured against previously polymerized pieces of denture base acrylic 4 mm thick.

2.2.1. Water sorption

Specimens 20 × 20 mm were cut from previously polymerized sheets, and preconditioned by storage at 37 °C over Drierite® in a dessicator until constant weight was attained. The specimens were then placed in distilled water at 37 °C, removed periodically, blotted and weighed. This process was repeated until a constant weight was obtained. The initial intervals between removals were short but subsequently increased for materials which were slow to reach equilibrium. The specimens were then again dehydrated with periodic weighings to a constant weight. Such measurements permit the determination of solubility, equilibrium water uptake, and, by application of conventional solutions of Fick's equations, the diffusion constants for the materials. In the initial sorption run, the weight increases due to water sorption may be partially offset by solubility, so that the best estimates for these values are obtained from the initial specimen weights and the results of subsequent desorption and sorption runs.

2.2.2. Tensile properties

Tensile strength and elongation to break for the materials were determined using a Polymer Labs Minimat materials tester with computer control. Specimens were cut from polymerized sheet using a dumbell die producing a 15.0 mm gauge length and a 3 mm specimen width. Dry specimens were prepared by dessication over Drierite® while wet specimens were prepared by storage at 37 °C in distilled water to saturation. Although preconditioned at 37 °C, all samples were tested at 20 °C with a head speed of 100 mm min^{-1} (equivalent to a strain rate of about 5 mm/mm/min).

2.2.3. Indentation hardness

The indentation hardness of the materials was determined at 20 °C using a Shore Type A2 hardness tester. The small sample size required and the nondestructive nature of the test permitted the determinations to be made on portions of the same polymer sheets from which the tensile specimens were cut.

TABLE I Soft lining products evaluated

Sample	Product name	Type	Manufacturer
1	Molloplast B	Heat-cured silicone (HCS)	Detax/Karl Hubber GmbH & Co., KG Germany
2	Mollosil	Catalysed silicone (CCS)	Detax/Karl Hubber GmbH & Co., Germany
3	Lite line	Visible-light-cured silicone (LCS)	L. D. Caulk Division, Dentsply International, Milford, Delawane USA
4	Evatouch	Catalysed silicone (CCS)	Neo Dental Chemical Products Co., Ltd., Tokyo, Japan
5	Super Soft	Heat-cured acrylic (HCA)	Coe Laboratories, Chicago, Illinois, USA
6	Novus	Heat-cured polyphosphazine (HCP)	Hygenic Corp., Akron, Ohio, USA
7	Kurapeet	Heat-cured fluoroelastomer (HCF)	Kureha Chemical Inc. Co., Tokyo, Japan

2.2.4. Tear strength

Tear strengths were determined by a method similar to that of Wright [17]. Specimens for tear strength determinations were cut from prepolymerized sheets as 10 mm × 50 mm rectangles. An initial cut of 20.0 mm up the midline of the specimen was made to form two legs. These legs were clamped in a Polymer Labs Minimat tester and pulled at 180 degrees to each other at tearing rates of 0.5, 5.0, and 50.0 mm/min. Both wet and dry samples were tested at 20 °C.

2.2.5. Peel strength

Peel strengths (bond strengths) were tested by the method utilized by Wright *et al.* [18]. The specimens were composite structures consisting of a strip of rigid denture base resin (Lucitone 199, L. D., Caulk Div. Dentsply Intl.) to which was cured a layer of the soft lining material under test. The denture base strip was 4 mm thick and the layer of soft liner was 3 mm thick. The two layers were bonded by the manufacturers recommended procedure for approximately one half their length, so that the free end of the soft liner could be doubled back 180 degrees and pulled parallel to the bonding surface. An Instron 1123 computer-automated tester with a 200 lb load cell (approximately equivalent to 890 N) was used for the tests. All tests were performed at 20 °C, at peeling rates approximating 10.0, 1.0, and 0.1 mm/s. Dry specimens were preconditioned over Drierite®, while wet specimens were preconditioned by storage in 37 °C water to constant weight.

3. Results

3.1. Water sorption

The results for water sorption and solubility of the various products are summarized in Table II. These two properties are reported together because the test procedure combines the determination of these two properties with the values being calculated from the weight changes of the alternating wet–dry cycles. Note that the values do not correspond simply to the observed weight changes. For example in the case of Mollosil where the measured solubility exceeds the water sorption, the weight change at the end of the first hydration is negative (approximately − 7.00%) because of the simultaneous occurrence of sorption and solubility. As commonly observed for other materials the rates of desorption are consistently higher than the rates of sorption. As a group the soft lining materials are consistently slower to reach equilibrium than are denture base resins. For most of the materials tested here approximately 3 weeks were needed to attain constant weight in sorption. The main exception was Novus where 20 weeks were required.

3.2. Tensile properties

The measured tensile strengths and elongations of the materials in uniaxial tension are given in Table III. The Shore durometer hardnesses determined on the same sheets from which the tensile specimens were cut appear in the same table. The values reported are predominantly for specimens in the dry condition. They permit a tentative comparison between the characteristics of the different products and an estimate of the relationships between the results of the different test methods. It is recognized that wet values are of greater significance in the clinical situation and should form the basis for eventual standards for product acceptance. However, as a result of the problems noted above in regard to equilibration, results for water-saturated specimens are not yet available. Comparing these results between test methods finds no detectable correlation between tensile strength and either elongation or hardness. An apparent negative correlation does appear to exist between elongation and hardness, so that products exhibiting high hardness values are likely to have low tensile elongation.

3.3. Tear strength

The tear strength of each material was tested in both the wet and dry conditions at the rate of 5.0 mm/min. In addition Molloplast (wet and dry) and Novus (dry) were tested at 50 and 0.5 mm/min. The results of all of the tear tests are shown in Table IV. These results divide the materials tested into two clear groups. All of the silicone materials plus the fluoroelastomer Kurapeet exhibited low tear strengths (highest value of 3.30 kJ/m^2 for any combination). In comparison the results for the acrylic and phosphazine materials indicate tear strengths five to ten times higer. Where measured, the effect of increased tearing rates was as expected to increase the measured strengths, however, the rate of increase was proportionately greater for

TABLE II Water sorption and solubility of soft liners

Product name	Type	Water sorption (%)	Solubility (%)
Molloplast B	HCS	0.40	0.37
Mollosil	CCS	1.17	8.07
Lite Line	LCS	1.51	0.37
Evatouch	CCS	6.21	4.56
Super Soft	HCA	7.35	2.05
Novus	HCP	30.16	0.34
Kurapeet	HCF	3.5	1.28

TABLE III Tensile properties of soft liners (dry)

Product	Tensile strength (MPa)	Elongation (%)	Shore durometer hardness
Molloplast B	4.94	413	51
Mollosil	3.62	433	23
Lite Line	3.28	100	73
Evatouch	3.14	133	50
Super Soft	2.90	330	45
Novus	4.41	200	38
Kurapeet	3.29	253	35

TABLE IV Tear properties of dry and wet soft liners

Product	Tearing rate (mm/min)	Tearing energy (kJ/m^2)
Molloplast B (dry)	50	1.56
Molloplast B (dry)	5.0	1.43
Molloplast B (dry)	0.5	1.28
Molloplast B (wet)	50	1.65
Molloplast B (wet)	5.0	1.40
Molloplast B (wet)	0.5	1.10
Mollosil (dry)	5.0	1.74
Mollosil (wet)	5.0	1.04
Lite Line (dry)	5.0	3.30
Lite Line (wet)	5.0	3.10
Evatouch (dry)	5.0	1.67
Evatouch (wet)	5.0	1.22
Super Soft (dry)	5.0	9.70
Super Soft (wet)	5.0	19.30
Novus (dry)	50	20.59
Novus (dry)	5.0	14.41
Novus (dry)	0.5	7.59
Novus (wet)	5.0	14.41
Kurapeet (dry)	5.0	2.03
Kurapeet (wet)	5.0	2.78

Novus than for Molloplast. The behaviour of the materials differed greatly in their response to wetting and drying. Novus and Molloplast showed little difference between conditions while the other silicones all showed reduced strength in the wet condition. The greatest effect was seen in the case of the acrylic Super Soft which showed a doubling of its strength in the wet condition. The plasticizing effect of water on this product seems to relieve stress concentrations that otherwise lead to premature failure.

3.4. Peel strength

The adhesion of the soft lining material to the denture base is normally measured as the peel strength. This characteristic differs from the others usually measured in that it is not exclusively a characteristic of the soft lining material alone. The values obtained are dependent upon the material used as the denture base and particularly upon the technique used to fabricate the specimens. Care must be used in evaluating the results not to attribute the results exclusively to the material. The fabrication method used here followed that of Wright [18]. It corresponds to a standard relining technique and was selected in part to permit comparison with the results of other studies. Some of the observed bonding failures could result from differences between the standard and manufacturers recommended techniques and may not be representative of the materials' potential. Table V summarizes the peel test results. Three distinct patterns of failure were observed. Molloplast and Super Soft failed in cohesion, the other silicones had little or no bond strength and failed in adhesion. Novus and Kurapeet showed mixed adhesive/cohesive failure

TABLE V Peel strength of soft liners

Product	Peel rate ($m/s \times 10^{-4}$)	Peel energy (kJ/m^2)	Failure mode
Molloplast B (dry)	1.0	368	Cohesion
Molloplast B (dry)	0.1	350	Cohesion
Molloplast B (wet)	1.0	372	Cohesion
Molloplast B (wet)	0.1	407	Cohesion
Mollosil (dry)	0.1	17	Adhesion
Lite Line (dry)	NA	NA	No bond
Evatouch (dry)	NA	NA	No bond
Super Soft (dry)	0.1	295	Cohesion
Novus (dry)	10	1355	Mixed C/A[a]
Novus (dry)	1.0	1697	Mixed C/A
Novus (dry)	0.1	901	Mixed C/A
Novus (wet)	0.1	893	Mixed C/A
Kurapeet (dry)	0.1	120	Mixed C/A

[a] C/A = cohesion/adhesion.

implying either closely similar bond and tear strengths or else a patchy pattern of adhesion. No consistent differences were observed between wet and dry conditions or as a result of differences in the stress rate except a possible increase of peel strength with increases in peel rate for Novus. The increase is not monotonic and, considering the mixed mode of failure, may be coincidental. The peel strength for Novus was clearly greater than that for all other materials. The values for Super Soft acrylic were significantly lower than those for either Molloplast or Novus, but were determined only in the dry condition. Considering the large change in tear strength seen for this material as a result of hydration it may be premature to judge its relative ranking in the absence of wet values.

4. Discussion

The primary objective of this study was to evaluate the characteristics of a wide variety of commercially acceptable denture soft lining materials as a guide to establishing criteria for the selection and evaluation of proposed experimental materials. It was based upon the assumption that continued commercial success must reflect the attainment of at least minimally acceptable values for each property. It was further felt that the range of properties offered by a group of products, and the clinically perceived deficiencies of particular types of materials could be combined to determine desirable as well as minimal levels for each property.

Any study of this sort automatically generates a number of secondary effects even though they were not originally identified as objectives of the study. It is desirable to identify the materials tested by name to increase the utility of the results to the individual clinician who might have a valid interest in product comparison but who might not be able to identify the products from chemical composition and structure alone.

The products tested in this investigation appear to be a reasonably representative sample of the various types of product in widespread current use. In a few instances, as seen from the measurements of peel strength, materials primarily included for the sake of compositional completeness served to highlight deficiencies in properties and technique. Similarly, with a few exceptions, the test methods used were appropriate and effective for the characterization of these products. The main deficiencies revolve about the procedures used for specimen preparation and conditioning.

There have been two main approaches to the matter of specimen preparation. One has been that the only fair comparison of products occurs when each material is prepared completely in accord with the manufacturers directions. Under these circumstances any deficiencies are clearly attributable to the product itself. However, a material which required unusual or extreme fabrication procedures might suffer when processed in the normal commercial dental laboratory. This situation is further aggravated when the use of specialized auxiliary materials are required. The alternative is to use a single standard procedure for the preparation of all materials as was done here. This has the advantage of increasing the intercomparability of results with those of other studies, but damages the standing of the affected products.

In this study the chemically and light cured silicone materials Mollosil, Evatouch and Liteline showed little or no adhesion to the denture base materials when processed by the standard technique. Since such results are not compatible with clinical success, they are judged to be the result of a technique deficiency and have been excluded from consideration in setting minimal and desirable characteristics.

Water sorption and desorption and the associated property changes are important characteristics of soft lining materials since they are normally processed in a "dry" condition and used in a water-saturated "wet" condition. These changes as they related to material composition and structure are of particular importance in the development and selection of new compositions. However, for estimation of clinical effectiveness only the properties in the wet condition are appropriate guides for material selection. In the absence of prior information about the relationship between the values for particular properties and the water content, the only safe estimate of properties in the clinical situation are values obtained in the fully saturated condition. In this study the unexpectedly long time required for equilibration of some of the materials in comparison to the planned schedule precluded the acquisition of all such information.

This problem is not severe for the silicone-based materials where the total water sorption and solubility are limited and the associated property changes are small. Of the silicones, only the chemically initiated products showed sizable changes between the wet and dry conditions (see Tables II and IV). The two products with the highest water sorption are Super Soft and Novus, although the effects in these two materials are quite different.

TABLE VI Property standards for soft lining materials

Property	Limiting value	Desired value
Water sorption	5.00% max.	2.00% max.
Solubility	2.00% max.	1.00% max.
Tensile strength	3.00 MPa min.	3.75 MPa min.
Elongation	100% min.	300% min.
Hardness (Shore A)	50 max.	30 max.
Tear strength @ 5 mm/min	1.4 kJ/m^2 min.	12.0 kJ/m^2
Peel strength @ 0.1 mm/s	300 kg/m min.	500 kg/m min.
Modulus E (DMA)	8.0 MPa max.	5.5 MPa max.
Damping tan δ (DMA)	0.05 min.	1.0 min.

All values for water equilibrated specimens.

In the case of Super Soft, and presumably other similar acrylics, the effect of water sorption on the properties are generally favourable. Absorbed water acts as a plasticizer and the perceived relatively high hardness of the dry condition is reduced. At the same time the toughness of the material is significantly increased (see Table IV).

The water sorption of Novus is slow but eventually reaches levels almost an order of magnitude greater than the other materials. The modulus as measured by DMA shows a marked decrease in the wet condition [9], but the other properties measured here showed little or no change. It might be expected that such large water sorptions would be accompanied by sizable volume changes which might cause adverse effects on clinical fit. However, in this case the material is widely used without reported problems with fit. Perhaps the molecular structure of this product will accommodate these large volumes of water without significant dimensional change, if so, sizable increases in density should be seen in the wet condition. Additional future research is needed to clarify the apparent conflict in findings. However, it is well known [19, 20] that water uptake of elastomers is much less in aqueous solutions, because uptake is osmotically driven. Hence uptake in saliva will be much less.

5. Conclusions

In spite of these noted deficiencies in methods and results, the information gained in this investigation does appear adequate to establish standards for both minimal and desirable properties of soft lining materials. These standards can be used in selecting among proposed new materials for further development and testing. The suggested values are summarized in Table VI.

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