

A comparison of commercial tissue conditioners using International Standard (ISO) 10139-1

TETSUYA TAKAMATA^{1,2}, SANDRA PARKER³, SHIHO KATASE⁴
HISAYA NUMAO^{1,2}, YUMIKO KATO^{1,2}, KOZABURO HASHII^{1,2} and MASAOKI ANZAI⁵

¹*Department of Dental Diagnostic Sciences, School of Dentistry, Matsumoto Dental University*

²*Department of Oral and Maxillofacial Biology, Graduate School of Medicine,
Matsumoto Dental University*

³*Department of Biomaterials in Relation to Dentistry, St. Bartholomew's and Royal London,
School of Medicine and Dentistry, Queen Mary and Westfield College*

⁴*Department of Crown and Bridge Prosthodontics, Matsumoto Dental University, School of Dentistry*

⁵*Department of Conservative Dentistry, School of Dentistry, Matsumoto Dental University*

Summary

The aim of this study was to analyze the composition of eight commercial tissue conditioners, assess each material using the International Standard (ISO) 10139-1 and relate differences in properties to composition. A visible light spectrophotometer (IR-460 : Shimadzu, Kyoto, Japan) was used to analyze the principal components of both liquids and polymer powders. A High Pressure Liquid Chromatograph (HPLC : Shimadzu, Kyoto, Japan) was used to measure the principal ingredients. A gas chromatograph (GC-15A : Shimadzu, Kyoto, Japan) was used to measure the alcohol concentration. Consistency and penetration tests were carried out as detailed in the ISO 10139-1. The penetration test measurements were also made at 2, 3 and 4 weeks. All materials satisfied the standard in the consistency test and in the penetration test after one week. However, three materials failed to satisfy the standard in the penetration test after two hours. In all materials the powder was found to be poly (ethyl methacrylate), while plasticizers and ethanol content varied. Two materials contained other components thought to be antibiotic or antibacterial agents. There appeared to be a correlation between the ethanol content and the consistency results.

Introduction

Tissue conditioners are temporary denture lining materials used to improve the health of the soft tissues of the denture bearing areas. They allow bruised or swollen tissues to return their more normal shape and size¹⁻⁴. Their viscoelastic nature allows good adaptation to be maintained while the contours of the mucosa change⁵. This property also makes tissue conditioners useful as functional

impression materials ; other uses include cleft palate speech aids and immediate surgical splints^{1,6-8)}. There have been many articles and reports published concerning the physical and mechanical properties of temporary soft lining materials. Most papers have described the viscoelastic characteristics under static and dynamic conditions by measurement of creep and dynamic modulus⁹⁻¹³⁾. However, there has never been a direct comparison, in spite of the availability of many commercial tissue conditioners.

In 1991, ISO 10139-1 was established by technical committee ISO/TC 106, Dentistry, Sub-Committee SC2, Prosthodontic Materials. The purpose of this study was to compare eight commercial tissue conditioners using this standard test. Their composition was also analyzed to determine any correlation with the results from the standard tests.

Materials and Methods

Details of the materials tested are listed in Table 1, which also includes powder/liquid ratio used and mixing time. All samples were prepared according to the manufacturer's instructions, where a range of mixing times was given the mean was used.

Table 1 : Tissue conditioner evaluated

Brand (Cord)	Manufacturer	Batch No. Powder/Liquid	Powder/Liquid (g/ml)	Mixing time (sec)
Tissue Conditioner (TC)	Shofu Inc.	029955/029972	6.0/5.0	90
Fit Softer (FS)	Sankin Ind.Co.	324-160/324-053	3.1/2.5	45
Soft-Liner (SL)	GC Dent.Ind.Co.	060592/230491	2.2/1.8	45
Denture-Soft II (DS)	Chem.Ins.Co.Ltd	2011/2011	3.0/2.0	30
Visco-gel (VG)	Detrey Division	10/4, 21	3.0/2.2	30
Caulk-Lynal (CL)	L.D.Caulk Co.	981003/981026	3.0/2.0	30
Coe-Comfort (CC)	Coe Labo. Inc.	052798A/082098A	6.0/5.0	30
Hydro-Cast (HC)	K.C.Dental Co.	30398/04199	5.0/6.2	60

Analysis of Composition

A visible light spectrophotometer (IR-460 : Shimadzu, Kyoto, Japan) was used to analyze the principal components of both liquids and polymer powders. The infra-red spectra of both the liquid and polymer were within the 400 to 4,000⁻¹ region. A High Pressure Liquid Chromatograph (HPLC : Shimadzu, Kyoto, Japan) was used to measure the principal ingredients using pure substances such as butyl phthalyl butyl glycollate (BPBG), butyl benzyl phthalate (BBP), benzyl benzoate (BB) and ester stearic acid (ES) as standards. A gas chromatograph (GC-15A : Shimadzu, Kyoto, Japan) was used to measure the alcohol concentration, with a $\pm 2\%$ margin of error.

Consistency Test

The apparatus and method used were as specified by ISO 10139-1. A delivery tube or syringe of a suitable material (e.g. glass or polytetrafluoroethylene), with an internal diameter between 10mm and 15mm, was used to dispense 2 ± 0.1 ml of material onto a glass plate. 30 ± 1 sec after mixing, the sample was compressed by vertically applying another glass plate termed the minor load, having a mass of 100 ± 1 g and a thickness not less than 2mm. The plates and the sample were then main-

tained at temperature at $37 \pm 1^\circ\text{C}$. 120 ± 1 sec after mixing the major load (1000 ± 5 g) was applied vertically, so that the total load (major and minor) was 1100 ± 6 g. This load was maintained for 60 ± 1 sec after which it was removed, leaving the minor load in place. 8 ± 0.5 min after mixing the maximum and minimum diameters of the resulting disc of material were measured to an accuracy of ± 0.5 mm. The average of the two measurements was calculated. To satisfy the standard specification the value should be between 25mm and 75mm.

Penetration Test (behavior in water)

The penetrometer apparatus and method used were as specified by ISO 10139-1. The apparatus comprises cylindrical penetrator (1mm in diameter) which is fixed on a vertical rod. The vertical rod plus the penetrator has a mass of 50 ± 5 g. There is a locking device which fixes the penetrator at any vertical position and is used to measure the depth of penetration. A sample of approximately 20ml was placed in a metal ring (30 ± 1 mm internal diameter and 3 ± 0.1 mm high) on the flat plate ($50 \times 50 \times 4$ mm³) made of unplasticized poly (methyl methacrylate) (PMMA). There was a slight overflow. An unplasticized polyester film of 50 ± 30 μm thickness was used to cover the material. A glass plate ($50 \times 50 \times 60$ mm³) was then placed on top and a weight of 2 ± 0.1 kg was applied. 10minutes after mixing, the load, the glass plate and polyester film were removed and the sample was stored in a water bath at $37 \pm 1^\circ\text{C}$. 115 ± 0.5 min after mixing the specimen was removed from the water bath and placed in the penetrometer. The penetrator was brought just into contact with surface of the specimen and locked in position. The rod from the dial gauge was brought into contact with the vertical rod and adjusted to zero. 120 ± 0.5 min after mixing the vertical rod was released for 1.5 ± 0.5 sec, allowing the penetrator to penetrate the specimen and it was then locked in position. The dial gauge was used to measure the depth of penetration. The test was carried out five times on each specimen using a different area each time. The mean of the five measurements was recorded as value A. To satisfy the standard specification A had to be 1.8mm or less. The specimen was stored again in water at $37 \pm 1^\circ\text{C}$ and the test was repeated after seven days and the mean value was recorded as value B. To satisfy the standard specification B had to be 0.18mm or more. The penetration ratio was calculated as A/B and the result was expressed to one decimal place. To satisfy standard specification A/B had to be 5 or less. The ISO standard test recommended measurements only at two hours and one week, but measurements were also made at two, three and four weeks as reference data.

Statistical Treatment of Results

Bartlett's test for homogeneity of variance was applied to the data. If the resultant test value was less than the critical χ^2 value at the 0.10 level of significance, and analysis of variance (ANOVA) was greater than the critical value (sample variances not homogenous), the Welch test was substituted for the ANOVA. When there was a significance among the means, multiple comparisons using Tukey's test were performed.

Results

Composition

Table 2 gives the weight to volume ratio (%) of the esters, the % ethanol concentration and the sum of these two components in the liquid. From the analysis of the polymer powder, the infra-red

Table 2 : Liquid composition

Brand Cord	Ester	(W/V %)	Ethanol (W/V %)	Ester + ethanol
VG	BPBG	94.6	7.1	101.7
FS	BPBG	92.9	8.6	101.5
DS	BPBG	88.7	12.5	101.2
SL	BPBG	86.7	15.7	102.4
CL	BPBG	67.9	0.4	68.3
HC	BBP	86.1	13.8	99.9
TC	BBP	85.6	13.9	99.5
CC	BB+ES	71.9	6.2	78.1

Table 3 : Penetration test (mm) and A/B ratio

Code	A	B				A/B
	2 hours	1 week	2 weeks	3 weeks	4 weeks	
	X (S.D.)	X (S.D.)	X (S.D.)	X (S.D.)	X (S.D.)	
TC	1.8 (0.14) a*	1.3 (0.27) a	1.4 (0.25) a	1.8 (0.14) a	1.6 (0.10) a	1.4
CC	2.5 (0.10) b	2.3 (0.13) b	2.5 (0.20) b	2.5 (0.10) b	2.5 (0.12) b	1.1
DS	1.4 (0.12) c	1.4 (1.18) a	1.2 (0.16) a	1.4 (0.12) a,c	1.3 (0.19) c	1.0
SL	1.7 (0.17) a	1.6 (0.23) c	1.3 (0.33) a	1.7 (0.17) a	1.7 (0.29) a	1.1
FS	1.8 (0.12) a	1.9 (0.22) d	1.7 (0.36) c	1.7 (0.12) a	1.7 (0.25) a	1.0
CL	1.5 (0.14) c	1.4 (0.24) a	1.4 (0.20) a	1.5 (0.14) a,d	1.3 (0.33) a,c	1.1
VG	2.2 (0.14) d	2.1 (0.21) c	2.1 (0.18) d	2.3 (0.24) b	2.3 (0.32) b	1.0
HC	3.1 (0.35) e	2.7 (0.25) e	2.4 (0.33) b,d	3.1 (0.35) b	2.4 (0.30) b	1.5

*Multiple comparisons using Tukey's procedure. At $p < 0.05$, groups means designated by the same letter are not statistically different.

spectra of all the powders showed the general characteristics of poly (ethyl methacrylate) (PEMA).

International Standard ISO 10139-1 test

Table 3 showed the results of the penetration tests. Figures 1 and 2 present the diagram of the consistency and penetration tests. In Figure 1, those means that are not statistically different from one another (Tukey's procedure for multiple comparisons, $p < 0.05$) are joined by a line. In the consistency test, all materials in this study were up to ISO standards. In the penetration test, on the other hand, some materials were below these requirements. For the ratio of penetration (A/B) all

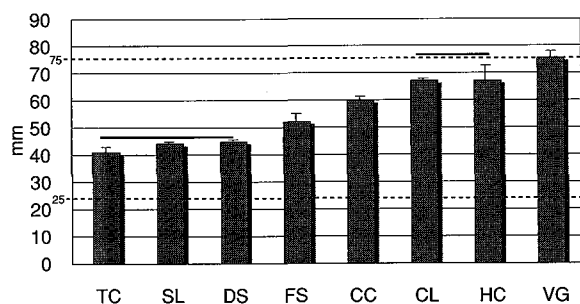


Figure 1 : Histogram of the consistency test results. Means not statistically different ($p < 0.05$) are joined by a line. Broken lines represent upper and lower limits of ISO 10139-1.

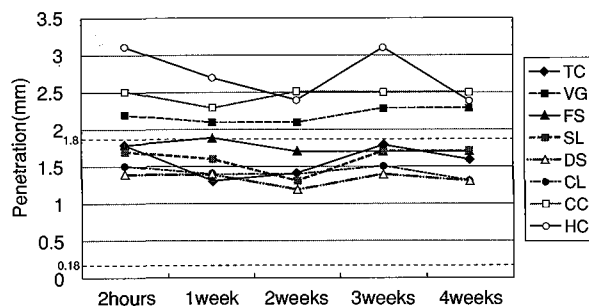


Figure 2 : Penetration test results as a function of time. Broken lines represent maximum A (0.18mm) and minimum B (1.8mm) values of ISO 10139-1.

eight materials satisfied the standard.

Discussion

From the analysis of the liquids (Table 2), VG, FS, DS, SL and CL appear to contain butyl phthalyl butyl glycolate (BPBG). The formation of butyl alcohol, and phthalic acid on hydrolysis could indicate *n*-butyl phthalate, however the viscosity and density of the latter preclude this and the infrared spectrum is consistent with the spectrum of BPBG. HC and TC appear to contain butyl benzyl phthalate (BBP) and CC benzyl benzoate (BB), as previously reported¹⁴, and another plasticizer identified as an ester of stearic acid (ES). These primary constituents are aromatic esters and they act as plasticizers to the system and as such control the compliance and elastic properties. It has been suggested that dibutyl phthalate, a commonly used phthalate plasticizer, should not be used in tissue conditioners because of possible carcinogenic effect⁵.

The ethanol contents (see Table 2) are consistent with those found by others^{1,14-17}. Ethanol is a necessary additive as it enables the large aromatic ester molecules to penetrate the polymer beads. When the polymer powder and ethanol/ester mix are combined, the ethanol swells the polymer enabling dissolution by the ester and a gel is formed by polymer chain entanglement. The amount of ethanol required will depend on the molar volume of plasticizer used and the molecular weight and particle size of the polymer.

For materials, CC and CL the combined ester/ethanol percentage did not account for 100% of the liquid. It is thought that other components such as antibiotic or antibacterial agents may also be present¹⁸. It is also apparent from this study that the dental profession needs to properly define requirements of this type of material both for the material itself in function and as a treatment through delivery of various medicinal agents. Further work should be carried out to evaluate the effects of these agents on patient comfort and health.

All the materials studied satisfied the standard in the consistency test, although they covered a wide range of values as shown in Figure 1. The general trend appears to be that the higher the ethanol content, the stiffer the gel. HC is the one major exception. This is probably linked to the very low powder/liquid ratio (p/l) recommended by the manufacturers (see Table 1), at 0.81g/ml it is far lower than the other materials. There is no significant difference in the results for TC, SL and DS ($p < 0.05$) despite having different levels of ethanol, although they are all relatively high. This can be explained by other differences in their formulation. SL has the highest ethanol content, TC contains a lower molecular weight plasticizer (BBP) and DS has a higher powder/liquid ratio. However, the results still show a linear relationship between ethanol content and consistency as determined by the standard test (Fig. 3). This is in agreement with results found by Jones et al¹⁷ who showed that gelation time decreased with increase in ethanol content while gel strength increased. A decrease in gelation time is a result of a faster viscosity build up, so at a given time a material with a higher ethanol content would have a higher viscosity which

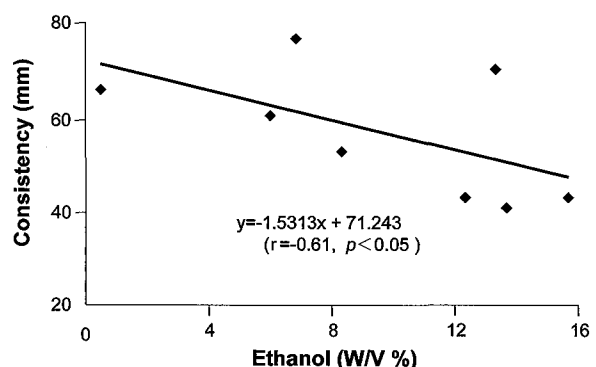


Figure 3 : Consistency test results as a function of ethanol concentration

would result in a lower measurement in the consistency test.

VG, CC and HC failed the penetration test after two hours (A). As for the consistency test, the result for HC is linked to the very low powder/liquid ratio. CC also has a low powder/liquid ratio (1.11 g/ml) and it contains something other than plasticizer and ethanol, as these accounts for only part of the liquid. For VG it is probably due to the low ethanol content. CL has the lowest ethanol content and a low powder/liquid ratio but still satisfies the standard, however 30% of its liquid component was not identified.

All materials gave values higher than the standard specification in the penetration test at seven days (B). However VG, CC and HC still have values higher than the maximum for A. Obviously, the standard values are such that the material is expected to harden i.e. the penetration value would be lower, on storage in water. Of the materials tested only TC and HC had B values significantly lower than A. As a result of all materials had for lower A/B ratios than specified standard maximum at five. A material would have to undergo significant hardening to have an A/B value that was above the standard. The longer term penetration results also showed little change with the exception of HC, again linked to its low p/l.

To summarize, all materials satisfied the consistency test standards. All except three materials satisfied the specified standards for two the hour penetration test (A). The three materials that failed would probably have passed if tested at the consistency they would be used at in practice. Although some materials were below standard after two hours in the penetration test, they fell within the standard after one week, and could be used in practice without problems or difficulties. At chair-side, tissue conditioners are more likely to be mixed to consistency rather than measured p/l. In particular, the data after one week would not be very important since the materials should be exchanged after one week by practitioners. If the three materials that failed the penetration test are used clinically, practitioners need to modify the mixing ratio (p/l). For instance, a somewhat larger quantity of powder or a lesser amount of liquid should produce good results. By modifying the powder to liquid ratio, we can use the materials with patient. On the other hand, if the materials were mixed according to the instructions, we should dip into the warm water (approximately 40°C) in 30 to 40 seconds before insert them in patient mouth. Thus, the standard value set for A appears to be set at a reasonable level. However, the values set for the seven day penetration (B) and A/B would appear to be too low or high, respectively. To approach these figures, a material would have to harden considerably when stored in water at 37°C, while in the mouth the hardening effect might not be as great as indicated by the higher solubility in artificial saliva reported by Kazanji and Watkinson¹⁹⁾.

Conclusions

The mechanical properties of eight commercial tissue-conditioning materials were evaluated using International Standard ISO 10149-1 test and the composition of these materials was also analyzed. The following conclusions were made :

1. All materials in this study were up to ISO standards in consistency test.
2. Some materials, after two hours, were below these requirements in penetration test. After one week, all tested materials fell within this standard.
3. There was evidence of a correlation between the consistency of the material and the concentra-

tion of ethanol.

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bility in, artificial saliva. Br Dent J 165 : 91-4.

抄録：国際規格 ISO 10139-1による市販ティッシュコンディショナーの比較検討

鷹股哲也（松本歯大・口腔診断・顎口腔機能制御学講座）

サンドラ・パーカー（ロンドン大学，クイーン・メアリー・アンド・ウエストフィールドカレッジ，生体材料）

片瀬志穂（松本歯大・歯科補綴Ⅱ）

沼尾尚也（松本歯大・口腔診断・顎口腔機能制御学講座）

加藤優美子（松本歯大・口腔診断）

橋井公三郎（松本歯大・口腔診断・顎口腔機能制御学講座）

安西正明（松本歯大・歯科保存）

日常臨床でティッシュコンディショナーは暫間的軟質裏装材あるいは機能印象材として広く用いられている。しかしこれら市販のティッシュコンディショナーの材料学的な統一規格による比較はまだなされていない。近年，ISO（国際標準化機構）から「10139-1 歯科-可撤式義歯床用暫間弾性裏装材 第1部 暫間用材料の規格」が作成され，これに基づき市販ティッシュコンディショナー8種類について粘調度試験と針入度試験ならびに成分組成分析を行った。その結果，粘調度試験では調査した8種類のティッシュコンディショナー全てが規格の範囲にあり，針入度試験では2時間後の値に規格から外れた材料が見られたが，これはエタノール濃度に影響されていることがうかがわれた。1週間後の測定値は全ての材料において規格の範囲内にあった。また調査した全ての材料にエタノールが含まれ，エタノールの含有濃度が高いと粘調度が低くなる関係が見られた。これらの結果は，臨床でティッシュコンディショナーを使用する際の基本的な知識として有用である。