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Penetration of some commercially available fluoride gels into occlusal fissures in vitro

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Summary

The rate and ability of fissure penetration of several commercially available fluoride gels were assessed in an in vitro fissure model. It was found that penetration depended on the fissure form, the surface tension and the viscosity of the gel. With the exception of one very viscous gel, all gels were able to penetrate into the shallow-wide fissure, but only two out of the five fluoride gels tested could penetrate into the deep-narrow fissure within 4 min. All gels with good penetration characteristics contained a surface-active agent and had a viscosity lower than 1200 mPa s. Not only the fluoride component but also the gel composition are important factors which influence the efficiency of dental fluoride gels.

Introduction

During the past few decades, the overall incidence of dental caries has been reduced due to the widespread use of fluorides and improved oral hygiene education. Despite these successes, fissure caries is still not under control and above all difficult to diagnose in an initial stage (Lewis and Hargreaves, 1975; Sawle and Andlaw, 1988). A possible explanation for this may be found in the intricate morphology of fissures (Juhl, 1983) which hinders appropriate hygiene and provides shelter for microorganisms. The enamel of the fissure wall is also reported to be more porous and vulnerable (Tagesen et al., 1988). Another possible factor

may be the incomplete penetration of products used for the prevention of dental caries, since fluoride-containing gels and toothpastes are known to be less effective at preventing caries on occlusal surfaces than on smooth surfaces (Fanning et al., 1971; Horowitz and Doyle, 1971). Experimental data on etching liquids or gels used for fissure sealing showed that penetration into a fissure did not occur in vitro (Garcia-Godoy and Gwinnett, 1987). This is also supported by theoretical considerations (Newman, 1968). Experimental data on laboratory-made fluoride-containing hydrogels showed that penetration of these gels is dependent on the viscosity of the gel, the presence of a surface-active agent and the fissure form (deep-narrow or shallow-wide). Penetration of a gel into deep-narrow fissures was possible only when the gel had a viscosity lower than or equal to 1200 mPa s and contained a surface-active agent (Bot-

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tenberg et al., 1989). To date, very few studies have been published regarding the properties of the excipients of fluoride gels (Braden et al., 1976) and, at least for commercially available products, no data are available on their ability to penetrate into fissures. The aim of the present study is the evaluation of the fissure penetration ability of some commercially available fluoride gels *in vitro*.

The penetration will be related to two physico-chemical variables of the gels: viscosity and surface tension. As in the previous study (Bottenberg et al., 1989), two different fissure forms are used: shallow-wide and deep-narrow fissures.

Materials and Methods

Five different gels available on the Belgian market were tested. Gel specifications, as obtained from the manufacturer, are listed in Table 1. The gels are designated A–E in order of increasing viscosity.

Determination of viscosity and surface tension

The viscosity was determined at room temperature ($21 \pm 1^\circ\text{C}$) using a rotating cylinder viscosimeter (Haake RV 3, Haake Messtechnik, Karlsruhe). In addition to the determination of viscos-

ity at 32 s^{-1} , as performed in the previous study, shear stresses were recorded at increasing and decreasing shear rates with the following intervals: 1, 2, 4, 8, 16, 32, 64, 128, 256 and 512 s^{-1} in order to evaluate thixotropy.

The surface tension of the gels was determined using an electrobalance (model RM 2, Cahn/Ventron, Paramount, CA) tensiometer at room temperature, calibrated with deionised water (73.0 dyn/cm at 20°C).

Penetration measurement

The set-up for *in vitro* determination of penetration has previously been detailed by Bottenberg et al. (1989). It consists of an injection appliance driven by a hydraulic plunger, a microscope equipped with a micrometer eyepiece and a fissure model cut from the crown of an extracted human third molar. Two different fissure forms were used: shallow-wide and deep-narrow.

A gel volume of 0.05 ml was injected at $t = 0$ on the cuspal slope of the fissure model. Six replicates were performed for each gel and fissure combination. Penetration depth was recorded as fissure depth minus the distance from the reference point, the fissure bottom, to the meniscus of the gel and is expressed as a percentage of the total fissure depth in order to compare different fissure types. Penetration depth was recorded 10,

TABLE 1

Composition, viscosity and surface tension of the gels

Gel designation	Gel composition	Surface-active agent	Viscosity (mPa s)		Surface tension (dyn)	Fluoride composition
			1 s^{-1}	32 s^{-1}		
A	hydroxyethylcellulose, flavours (cinnamon), preservatives	aminhydro-fluoride (0.4%)	618	143	13	aminhydro-fluoride (0.4%)
B	sodium carboxymethylcellulose, glycerol, sodium carrageenan, flavours, preservatives	Brij 56	2432	634	14	NaF (0.55%), NH_4F (1.13%)
C	not specified, pH about 3 (phosphoric acid)	none	8368	2048	44	NaF and HF (1.23%)
D	hydroxyethylcellulose (Natrosol 250H/HHR, 2%), phosphoric acid (0.1 M)	Brij 58 (0.3%)	20733	2610	7.4	NaF (0.4%)
E	identical to gel A, but differing viscosity	aminhydro-fluoride (3.3%)	14797	3362	6.7	aminhydro-fluorides (3.3%), NaF (2.21%)

TABLE 2

Penetration (in %) for the different gels in the shallow-wide fissure

p.t., time (in s) needed to achieve complete penetration; p.r., calculated penetration rate (slope of the regression line). Values are means \pm S.D.

<i>t</i> (s)	Gel				
	A	B	C	D	E
10	31.5 \pm 23.3	77.0 \pm 4.5	72.3 \pm 6.8	62.8 \pm 4.6	50.0 \pm 6.3
20	100.0 \pm 0	91.0 \pm 3.2	85.0 \pm 6.3	71.2 \pm 4.2	63.7 \pm 11.2
30	100.0 \pm 0	100.0 \pm 0	95.0 \pm 2.4	78.0 \pm 6.2	76.2 \pm 8.2
60	100.0 \pm 0	100.0 \pm 0	100.0 \pm 0	100.0 \pm 0	76.2 \pm 8.2
Values remained unchanged for <i>t</i> = 120, 180 and 240 s					
p.t. (s)	18.7 \pm 1.96	25.5 \pm 2.1	36.17 \pm 6.3	39.5 \pm 3.1	–
p.r.	5.38 \pm 0.66	3.14 \pm 0.05	2.57 \pm 0.65	2.18 \pm 0.2	0.16 \pm 0.02

20, 30, 60, 120, 180 and 240 s after injection. In those cases where complete penetration occurred within the 4 min interval given, the penetration time (in s) was also recorded.

In order to facilitate statistical processing and interpretation of the data on penetration depth, the penetration depth vs. time graphs were linearized by a linear regression. The slopes of the graphs thus obtained provide a measure of the penetration rate. Values (means \pm S.D.) of the depth, time and rate of penetration were calculated and a non-parametric Mann-Whitney U-test

was performed on the variables, penetration rate and penetration time.

Results

The viscosity characteristics of the gels at various shear rates show that all gels have slightly pseudoplastic flow properties (Fig. 1). Gels C and D showed minor thixotropic characteristics. The surface tension of most gels (except gel C) is low, due to the addition of a surface-active agent. Gels

TABLE 3

Penetration (in %) for the different gels in the deep-narrow fissure

p.t., time (in s) needed to achieve complete penetration; p.r., calculated penetration rate (slope of the regression line). Values are means \pm S.D.

<i>t</i> (s)	Gel				
	A	B	C	D	E
10	24.3 \pm 20.6	18.8 \pm 2.2	8.7 \pm 1.6	8.7 \pm 4.3	
20	85.3 \pm 10.9	21.7 \pm 4.2	17.3 \pm 2.7	10.7 \pm 4.2	18.0 \pm 8.3
30	100.0 \pm 0	23.5 \pm 4.5	23.3 \pm 2.2	12.5 \pm 2.9	24.0 \pm 6.7
60	100.0 \pm 0	31.5 \pm 9.6	23.3 \pm 2.2	13.2 \pm 2.9	24.7 \pm 7.3
120	100.0 \pm 0	84.0 \pm 21.3	23.3 \pm 2.2	13.8 \pm 3.8	24.7 \pm 7.3
180	100.0 \pm 0	100.0 \pm 0	23.3 \pm 2.2	14.5 \pm 3.6	24.7 \pm 7.3
240	100.0 \pm 0	100.0 \pm 0	23.3 \pm 2.2	16.2 \pm 3.8	24.7 \pm 7.3
p.t. (s)	23.8 \pm 2.7	139.2 \pm 21.5	–	–	–
p.r.	4.45 \pm 0.47	0.64 \pm 0.11	0.07 \pm 0.02	0.04 \pm 0.01	0.07 \pm 0.02

TABLE 4

Levels of significance according to the Mann-Whitney U-test between the different gels per fissure form

The test was performed for two variables: time of complete penetration (p.t.), and penetration rate, p.r. Data above the diagonal refer to deep-narrow fissure, those below, to shallow-wide fissure.

	A		B		C		D		E	
A	*		p.r.	0.002	p.r.	0.002	p.r.	0.002	p.r.	0.002
		*	p.t.	0.002	p.t.	—	p.t.	—	p.t.	—
B	p.r.	0.002	*		p.r.	0.002	p.r.	0.002	p.r.	0.002
	p.t.	0.002		*	p.t.	—	p.t.	—	p.t.	—
C	p.r.	0.002	p.r.	0.065	*		p.r.	0.041	p.r.	0.94
	p.t.	0.002	p.t.	0.06		*	p.t.	—	p.t.	—
D	p.r.	0.002	p.r.	0.002	p.r.	0.18	*		p.r.	0.065
	p.t.	0.002	p.t.	0.002	p.t.	0.39		*	p.t.	—
E	p.r.	0.002	p.r.	0.002	p.r.	0.002	p.r.	0.002	*	
	p.t.	—	p.t.	—	p.t.	—	p.t.	—		*

A and E contain aminhydrofluoride which has surface-active properties (Busscher et al., 1988), while gels B and D contain either Brij 56 or 58 as surfactant agent. The results of the penetration experiments are listed in Tables 2 and 3 for the shallow-wide and deep-narrow fissures, respectively. All gels, with the exception of gel E, were able to penetrate into the shallow-wide fissure. Penetration is achieved between 18.7 and 39.5 s with the low-viscosity gels being the fastest. In the deep-narrow fissure only two gels (A and B) were able to penetrate within the time limit of 240 s.

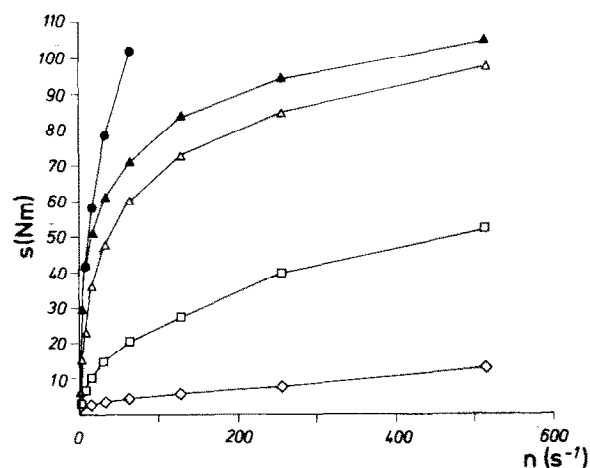


Fig. 1. Shear stress (s , in Nm) vs. shear rates (n , in s^{-1}) for the different fluoride gels. For shear rates below $100 s^{-1}$ not all values are given. (\diamond) Gel A, (\square) gel B, (\triangle) gel C, (\blacktriangle) gel D, (\bullet) gel E.

Their viscosity (at $32 s^{-1}$) is within the range of 1200 mPa s shown to be the limiting value for penetration (Bottenberg et al., 1989). Both gels contain molecules that possess surface-active properties. During penetration, the results obtained on the depth and time of penetration show considerable S.D. values. This can be explained as being caused by the inclusion of air bubbles on the fissure bottom in these cases. A statistically significant difference could be demonstrated for the variable penetration rate between gels A and B and the remaining gels. In the deep-narrow fissure gels C–E showed no significant differences. In the shallow-wide fissure, no significant difference could be established between gels B and C as well as between gels C and D. Significance levels are displayed in Table 4.

Discussion

The rheological characteristics of the gels showed typical non-Newtonian behaviour, commonly observed for the cellulose derivatives as used in these gels. A shear thinning could be observed at higher shear rates. This is believed to be advantageous for clinical efficiency (Braden et al., 1976). Whilst the magnitudes of the shear rates exerted on the gels in the course of application to the tooth and during penetration are unknown, they are probably rather low. In this study it could

be observed that even after application with a syringe of diameter 1.2 mm, high-viscosity gels did not penetrate into the deep-narrow fissure. Thus, the amount of shear thinning of cellulose-based hydrogels is insufficient to ensure penetration. This *in vitro* study confirms the results reported in a previous investigation (Bottenberg et al., 1989). In the aforementioned work and in the present study, the variables exerting the strongest influence on penetration were found to be the fissure form, gel viscosity and the presence of a surface-active agent. The depth of penetration of all gels into the shallow-wide fissure was greater and required less time than in the case of the deep-narrow fissure. In the deep-narrow fissure, only those gels containing a surface-active agent and having a low viscosity were able to penetrate. This is consistent with the theoretical considerations stated by Newman (1968), who showed that the penetration of a liquid into a capillary is affected by the viscosity and surface tension of the liquid. The correlation between viscosity and penetration behaviour is dependent on the shear rate: penetration into the deep-narrow fissure cannot be related to the viscosity for all of the gels when the apparent viscosity at 32 s^{-1} is taken into consideration. The deeper penetration of gel E despite its higher viscosity must therefore be accounted for on the basis of its surfactant content (aminhydrofluorides) which compensates partially for its high viscosity. At a lower shear rate (1 s^{-1}), the penetration behaviour can be shown to be correlated to the viscosity. On the other hand, the influence of viscosity on the penetration behaviour in the case of the shallow-wide fissure can be explained provided that the apparent viscosity measured at 32 s^{-1} is taken into account. As in our previous study, interactive effects between viscosity, surface tension and fissure form can provide an explanation for this observation.

Since the gels, with the exception of gel E, displayed comparable penetration behaviour in the shallow-wide fissure, an assessment of the penetration characteristics of fluoride gels should be made with respect to the deep-narrow fissure. For this purpose, only gels A and B appear to be satisfactory.

Although some fluoride-containing hydrogels

are able to reach the enamel in the fissure *in vitro*, they show a low efficiency in preventing fissure caries *in vivo*. Two explanations for these findings are then possible: (i) the presence of microbial plaques in fissures, resulting in penetration of the gels being hindered; or (ii) in clinical studies, the failure to take the viscosity and surface tension of the gels into account. The role of plaque is dual: it may act as a physical barrier against penetration; secondly, diffusion of ionic fluoride through plaque is known to occur (Melsen et al., 1983). Thus, in the latter case, plaque may even act as a fluoride reserve beyond the time of application itself.

The results of clinical studies are difficult to interpret. Fissure caries itself is difficult to diagnose if one uses only a mouth mirror and probe, so a comparison between studies is problematic. Secondly, some studies do not differentiate between caries on smooth surfaces and fissures (e.g. Janczuk et al., 1981; Kerebel et al., 1985). Furthermore, there is a scarcity of data, if any, on the composition of the gels used. To date, the present authors are aware of the existence of two studies in which reference is made to occlusal fissure caries and data have been provided on gel composition. Horowitz and Doyle (1971) reported a low efficiency of fluoride-containing gels in fissures. Their gel contained 1.4% hydroxyethylcellulose and might have been found to be insufficient as regards the penetration characteristics. On the other hand, Hagan et al. (1985) used a 'thixotropic' gel and were able to demonstrate a reduction in fissure caries. However, to the best of our knowledge, studies relating clinical data to the characteristics of the excipients have thus far been neglected and remain a matter for future investigations.

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