Development of techniques for the analysis of total fluoride in varnishes

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Background

Although fluoride varnishes are commonly used for caries prevention, fundamental research on how different formulations affect adherence to teeth, fluoride release into saliva and uptake by teeth is scarce. Laboratory methods to accurately and repeatable determine fluoride content in varnish would be useful for these types of studies; however, there is no universally-accepted method to determine fluoride in varnishes.

Objective

This study aimed at developing a method to test total fluoride in 5% NaF varnishes using hexane either with direct measurement or a modified hexamethyldisiloxane diffusion method. The toxicity of chloroform, the ISO-proposed solvent for varnishes prior to water extraction, prompted an investigation into the use of hexane.

Materials and Methods

Twenty seven fluoride varnishes were tested for content. Different combinations of reagents and procedures were tested. Once a method was defined, 0.025 g of each varnish sample were weighed onto paper, placed into polypropylene tubes and 2.25 ml hexane were added. Tubes were capped, vortexed for 1 minute and periodically swirled for 20-25 minutes to dissolve the varnish. Subsequently, 42.25 ml deionized water were added, solutions were vigorously mixed for 15 seconds, and allowed to separate. Then, the aqueous layer was sampled.

Results

Fluoride concentrations ranged from 25,478±137 µg F/g (direct) and 22,532±408 µg F/g (diffusion) to 4,506±39 µg F/g (direct) and 4,257±333 µg F/g (diffusion). 21 products showed >90% recovery of labeled fluoride while six showed <90%. No significant differences were found among the methods (20,525±4,997 µg F/g for direct vs. 20,173±4,698 µg F/g for diffusion) with an ICC=0.97 for repeated measures. Results previously obtained using chloroform were not statistically significantly different when compared to those obtained using hexane (p=0.53). The six products exhibiting <90% recovery of labeled fluoride were more viscous and resistant to dissolution. Hexane fluoride extraction for varnish analysis achieved repeatable values falling within 90% of labeled values when the samples could be completely dissolved.

Discussion and Conclusion

No single method seems to be suitable for every varnish type. Clear formulations, those with calcium, and viscous varnishes that cannot be dissolved consistently yielded lower results. It is therefore concluded that the direct measurement of fluoride with a choice of hexane or chloroform as solvent can be used to analyze varnishes for total fluoride content when dissolution can be completely achieved.

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