Atomic emission spectroscopy comparison of KNO₃ concentration in sensitive teeth toothpastes: Are labels accurate?

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ABSTRACT

Sensitive teeth are common, and a typical treatment people use for sensitive teeth is toothpaste. A common desensitizer in these specialized toothpastes is potassium nitrate. In this study I measured the amount of potassium in Sensodyne, Colgate, and Top Care using atomic emission spectroscopy. Potassium nitrate and toothpaste samples were made in a nitric acid and cesium chloride solvent. The average percent of potassium nitrate was calculated to be 5.08%, 8.14%, and 5.72% in Sensodyne, Colgate, and Top Care, respectively. One sample t-tests showed that Colgate and Top Care had a significant difference from the labeled 5% KNO₃, while Sensodyne was accurate to the label claims. All tubes of toothpaste analyzed in this study had at least 5% potassium nitrate. Sensodyne was the only brand that was most accurate. The limitations of this project would be only testing one tube of toothpaste from each brand. For future studies I would suggest using higher quality deionized water to minimize potassium contamination, confirm potassium nitrate by measuring nitrate with ion chromatography, and also test more tubes of toothpaste to be able to generalize results.

INTRODUCTION

Having sensitive teeth is very common and many consumers use specialized toothpastes to treat their sensitive teeth. This experiment aims to determine the concentration of potassium nitrate (KNO₃) in toothpaste, because this is the active ingredient in most sensitive teeth toothpastes. This current study will determine if the toothpaste we are testing has the amount of potassium nitrate reported on the product label and if buying the most expensive toothpaste is necessary if the generic contains the same for a cheaper price.

Dentinal hypersensitivity: prevalence and theory

Dentinal hypersensitivity, commonly known as sensitive teeth, "is defined as pain arising from exposed dentin typically in response to thermal, chemical, tactile or osmotic stimuli " (1). Thermal stimuli are the most common and usually occur when teeth are exposed to hot or cold temperatures like drinking hot coffee or ice water. Receding gums and plaque on teeth are confounding factors, as they both can cause tooth sensitivity. In a newsletter Everyday Health, it states that "having an excessive buildup of plaque can cause tooth enamel to wear away" (2). Teeth are sensitive when the enamel is worn because nerves are exposed. Although multiple studies have reported a prevalence of dentinal hypersensitivity in different populations, it is difficult to truly quantify the percent of people in the world suffering from this ailment. For instance, studies thirteen years apart by different researchers show a prevalence as low as 4% (3) or as high as 74% (4) in the United Kingdom. Murray and Roberts compared questionnaire results about sensitive teeth from 1,000 participants in each of 6 countries in 1994 (5). The countries that were in the study were Indonesia, USA, Japan, France, Germany, and Australia. Their results indicated that the prevalence ranged around 13% through 27% with 27% being the highest of those countries.

For at least four decades, the accepted explanation for dentinal hypersensitivity has been Brannstrom's hydrodynamic theory. This theory states that dentinal hypersensitivity stems from a change in the fluid pressure in the tubules, which results in a nerve response that sends a pain signal (6). Figure 1 shows the anatomy of a tooth on the right and an enlarged image on the left that illustrates how the tubules connect to the surface of the tooth and the nerves. Recall that extreme temperatures cause tooth pain for those suffering from dentinal hypersensitivity. Specifically, Brannstrom's theory would explain the pain of eating something cold, like ice cream, as leading to a contraction of the fluid in the tubules. This contraction results in a decrease in pressure experienced by the nerves. When the tubules are exposed to hot temperatures, their fluid expands, leading to an increased pressure detected by the nerves. Using Brannstrom's theory, scientists proposed several solutions to treat sensitive teeth; for instance blocking the surface opening to the tubules to prevent exposure of the fluid to stimuli and resulting in pain from the nerves.





Dentinal Hypersensitivity: Treatment Options and Effectiveness

There are a variety of available treatments for dentinal hypersensitivity that range in invasiveness and expense; the most common and cost-effective is sensitive-teeth toothpaste, which is a form of home care for patients (6). Sensitive-teeth toothpaste usually contains one or more common desensitizers, such as potassium nitrate, stannous fluoride, sodium monofluorophosphate, or sodium fluoride (1). Of these, potassium nitrate is more common. Orchardson and Gillam (7) reviewed sixteen clinical trials of potassium-containing toothpastes, which were all double-blind, randomized studies. There were sixteen studies and in each study everyone had hypersensitive teeth. Only three studies out of the sixteen did not include a placebo/control and in those three studies they used potassium chloride instead. For each study, percent reduction in sensitivity was calculated for experimental and control/placebo groups. In these studies there were three different methods used such as tactile stimuli, cold air stimulus, and patient subjective ratings on surveys. Every paper but one had at least two methods of scoring to calculate hypersensitivity. More than half of the studies used both tactile and cold air stimulation. Eight of eleven studies that assessed sensitivity using a cold air stimulus showed a statistically significant difference between experimental and control groups with respect to percentage reduction in sensitivity. All of the six studies using threshold force as the measure showed a significant difference between experimental and control (7). This evidence supports that potassium nitrate is effective because based off of the results only a select few showed a difference from the 0.05%.

Although potassium nitrate is the most common desensitizer for sensitive teeth, alternatives exist such as stannous fluoride and calcium sodium phosphosilicate. Stannous fluoride obstructs the dentinal tubules and prevents fluid movement in the tubules to decrease nerve pain (8). Calcium sodium phosphosilicate also blocks the tubules by forming apatite crystals and it shows longer term effects (9). Using the calcium sodium phosphosilicate helped maintain a high buffering resulting in a neutral pH (9). In a study using calcium sodium phosphosilicate, it was stated that this desensitizer was more effective in reducing dentin hypersensitivity than potassium nitrate(9). However commercial desensitizing toothpaste shows potassium nitrate as a common treatment and several toothpaste manufacturers have options of using stannous fluoride instead.

Analysis of Toothpastes

Toothpaste ingredients have been analyzed by a variety of methods, such as HPLC, ion chromatography, and gas chromatography; alkali metals like potassium are typically measured using emission spectroscopy (10). To prepare toothpaste samples for analysis of alkali metals, the toothpaste simply needs to be dissolved in nitric acid or hydrochloric acid (10). In the present study, flame atomic emission spectroscopy was used to measure the concentration of potassium. Using flame atomic emission spectroscopy, heat energy is absorbed by an atom, then is transformed to potential energy as the electron is excited, then it falls back down and a photon of light is released. Light that's emitted gets detected, that's the signal or peak that'll be shown. Each element has its own wavelength. These wavelengths are 766.5 nm, 769.9 nm and 404.4 nm for potassium (8). It is recommended that cesium chloride is used because the cesium suppresses the ionization of potassium so that the signal for potassium is greater (11).

Atomic absorption spectroscopy



Figure 2.

In this study, we aim to answer the following research questions: (1) Does the concentration of KNO₃ measured in the lab match the concentration listed on the ingredient label of each toothpaste? and (2) Which brand provides the most cost effective potassium nitrate treatment? To answer these questions, we will analyze three different potassium nitrate-containing toothpastes using atomic emission spectroscopy. Measurements will be made using the atomic absorption spectrometer at Robert Morris University. Results will be analyzed using a one-sample t-test to compare to the concentration reported on the product label. The results of this study will contribute to the scientific community that, in spite of the price of Sensodyne, generic toothpaste may work and provide just as much as Sensodyne.

EXPERIMENTAL

Data was collected at Robert Morris University with the help of Dr. Paul Badger. Chemicals and Equipment

The following toothpastes were purchased from a local retailer: Sensodyne (Fresh Mint), Colgate (Mint Clean), and Top Care (Fresh Mint). Distilled water was purchased bottled from Giant Eagle. Cesium Chloride (99.99%) was purchased from Alfa Aesar. Potassium standard solution 1 mg/ml in 2-5% HNO₃ was purchased from Acros Organics. Nitric Acid was purchased from Fisher Chemical.

Emission data was collected using a Perkin Elmer AAnalyst 800 in flame mode and outfitted with autosampler AS-90. The instrument was controlled by Perkin Elmer's WinLab32 for AA (version 7.3.0.0697).

Preparation of standards

Six standards of potassium nitrate were prepared in the range of 0-20 ppm by diluting 1000 ppm potassium standard in a solvent composed of 3-4% HNO₃ and containing 1,000 ppm of cesium chloride.

Toothpaste Selection

Eight toothpaste options for sensitive teeth were identified at a local grocery store. Two listed stannous fluoride, one listed amino acid arginine and calcium carbonate, and five listed potassium nitrate as the active desensitizer ingredient. Three different toothpaste brands were selected for comparison. The three toothpaste brands that will be tested are Colgate sensitive complete protection, Top Care sensitive fresh mint, and Sensodyne fresh mint. Then the price per ounce in each toothpaste was compared to decide if the top rated is worth buying if they're all effective, Colgate is \$1.00 per ounce, Top Care is \$1.00 per ounce, and Sensodyne is \$1.75 per ounce.

Preparation of toothpaste samples

A 1 mL aliquot of each toothpaste was dissolved in 100 mL of 3-4% HNO₃ containing 1,000 ppm of cesium chloride. This solution was subsequently diluted by a factor of 10 using the same solvent. Hence the 1 mL of toothpaste was ultimately diluted by a factor of 1,000. *Atomic emission spectroscopy measurements*

Emission intensities were measured at a wavelength of 404.4 nm. The instrument was calibrated with the nitric acid cesium chloride solvent; a blank was run after every 5 samples. Each data point is an average of four replicates.

RESULTS

The emission intensity of each standard was plotted as a function of the concentration of potassium to yield the calibration curve in Figure 3.



Figure 3. Standard curve for potassium.

The concentration of potassium nitrate (KNO₃) in each of the toothpaste samples tested is reported in Table 1. These concentrations were calculated using the equation of the line from the calibration curve by substituting the emission intensity at 404.4 nm for y and solving for x..The intensity value needed to be diluted by a factor of ten for the experiment, the concentration determined from the standard curve was multiplied by 10 to calculate the concentration. The mass of 1mL of toothpaste was calculated using g KNO₃/ 100 mL of toothpaste, the experimentally determined concentration, and the molar mass of KNO₃ 39.10 g/K divided by ~101.1 g/KNO₃, to get from potassium to potassium nitrate..

Toothpaste Brand	Trial	Intensity	% KNO3 (w/v)	Average % KNO ₃	p Value
Colgate	1	25,468	8.08	8.14	0.000170*
	2	25,484	8.13		
	3	25,513	8.22		
TopCare	1	24,658	5.61	5.72	0.01*
	2	24,684	5.68		
	3	24,748	5.88		
Sensodyne	1	24,502	5.13	5.08	0.18
	2	24,497	5.11		
	3	24,461	5.00		

Table 1. Intensity and concentration data for toothpaste samples.

Statistical Analysis

A one-sample t-test was performed for each toothpaste brand. In all cases, the hypothesis was that the percent of potassium nitrate in that brand's sensitive teeth toothpaste would not have a significant difference from the 5%. For Colgate the one sample t-test found a significant difference of 5% potassium nitrate (t=76.7, (\bar{x})=8.14, s=0.0709, p=1.69, n=3). The Colgate sample had a much higher level of potassium than the label claims. For Top Care the one sample t-test found a significant difference of 5% potassium

nitrate (t=8.94, (\bar{x})=5.72, s=0.14, p= 0.01,n=3). The TopCare sample had a higher level of potassium than the label claims and this was a statistically significant result. Lastly, a one sample t-test found that Sensodyne seems to claim roughly 5% potassium nitrate (t=1.97, (\bar{x})=5.08, s=0.07, p= 0.18, n=3). The Synsodyne sample had roughly the same level of potassium that the label claims.

DISCUSSION

My results section shocked me knowing how common treating sensitive teeth with toothpaste is. The average concentration of potassium nitrate in Colgate was 8.14% and it did not match the label because the label said 5%. The average concentration of potassium nitrate in TopCare was 5.72% and it did not match the label because the label said 5%. The average concentration of potassium nitrate in Sensodyne was 5.08% and the label said 5% so it was relatively close. The brands of toothpaste are comparable by the percentage of potassium. It is worthwhile to pay more for Synsodyne because it has the amount of potassium that the label claims to have.

Although my results were relatively close there were some limitations in the study such as only testing one tube of toothpaste from each brand which only gave us 3 trials from those 3 tubes. Another limitation would be possible errors in the results because distilled water contains potassium and it's nearly impossible to get distilled water without potassium. We've attempted to correct the possible errors from the distilled water but each blank (the distilled water) varied in the results. Another limitation was not being able to use the ICP machine at Robert Morris because it was broken.

If this was to be retested in the future I would suggest using an ICP machine to test for potassium and the ion chromatography for nitrate. I would also suggest testing more tubes of toothpaste to make claims about the company as a whole such as if each tube contains a different amount of potassium nitrate. Also using more tubes of toothpaste would give more trials so there's a greater statistical power for the analysis.

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