



Short Communication

A bio-safety tooth-whitening composite gels with novel phthalimide peroxy caproic acid

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ABSTRACT

For hundred years, researchers have applied themselves to maintain people's white smiles. However, there are many reasons for teeth discoloration, such as personal habits, dental problems or exposure to other substances. Nowadays, bleaching process is the mainly used tooth whitening technology. Owing to the whitening effect of the used oxidant, this process can change the color formation of the teeth surface. Nevertheless, most of the bleaching products are peroxides, which will cause a variety of diseases and discomfort in the mouth. Herein, in this work, it was found that the phthalimide peroxy caproic acid (PAP) could be selected as the effective ingredient of polymer composite gel with non-hydrogenperoxide. Moreover, by comparing the new whitening gels with hydrogen peroxide (HP, the traditional whitening ingredients) gels, the whitening effect of polymer composite gels with 5% PAP were equivalent to the gels with 3% HP, and the composite gels with 12% PAP were also equally to the gels with 8% HP. More importantly, it was proved that PAP tooth whitening gel was much safer and more reliable than HP based products. It indicated that PAP based tooth whitening product can replace low-concentration peroxide teeth whitening gel in the home-market.

1. Introduction

With the improvement of people's material life, more and more attentions have been paid to teeth beauty. However, the beautiful, white teeth were often easily destroyed by extrinsic discoloration and/or intrinsic discoloration [1,2]. Extrinsic discoloration would be occurred among the people whose daily dietary sources contained tea, coffee, tobacco, or other foods that can support the chromogens [3]. Moreover, intrinsic discoloration may be caused by systemic factors, such as fluorosis or side effects of some drugs [4]. Therefore, teeth whitening has become an indispensable beauty project for many consumers.

Currently, the widely used tooth-whitening methods can be generally divided into two parts, the invasive whitening methods and the noninvasive whitening methods [5]. Due to the teeth would be irreversibly damaged by cutting and grinding the enamels when using the invasive whitening technology, noninvasive methods by using active ingredients gained more and more attentions [6–8]. Nowadays, the main active ingredients of teeth whitening products are peroxides, such as hydrogen peroxide [9–14], carbamide peroxide [15,16]. Although some peroxides, such as peroxy caproic acid (PAP) [17,18], were thought to be more safety, the hydrogen were still used as the most

efficient tooth-whitening products. However, whitening of teeth by hydrogen peroxide is very harmful to the health of oral mucosa and dentin. There are many phenomena such as oral mucosa burns [19] and dentin allergies [20]. As early as 2013 in Europe, in European markets, more than 0.1% of hydrogen peroxide (HP) was not allowed in law [21]. Moreover, sodium phytic acid [22,23], baking soda [24], and sodium hypochlorite [25] are allowed in law, but the main efficacy of the non-peroxide products are teeth cleaning rather than teeth whitening in short time.

Herein, by using polymer gel for the fine vehicle [26–30], an efficient, peroxy caproic acid (PAP) based composite gel was prepared for tooth-whitening process. According to the tooth bleaching results measured by Spectro shade computerized spectrophotometry, PAP showed equivalent tooth whitening effect to HP, and the composite gel with 12% PAP was found to have the best whitening effect. More importantly, learning from the scanning electron microscopy (SEM) measurement and the volunteers' report, PAP was proved nearly harmless to the teeth enamel, while HP would certainly cause the teeth hypersensitivity and burning sensation. Therefore, when comparing with PAP and HP, the composite tooth whitening gels with PAP had equivalent tooth whitening effect to HP gels, but much safer and more

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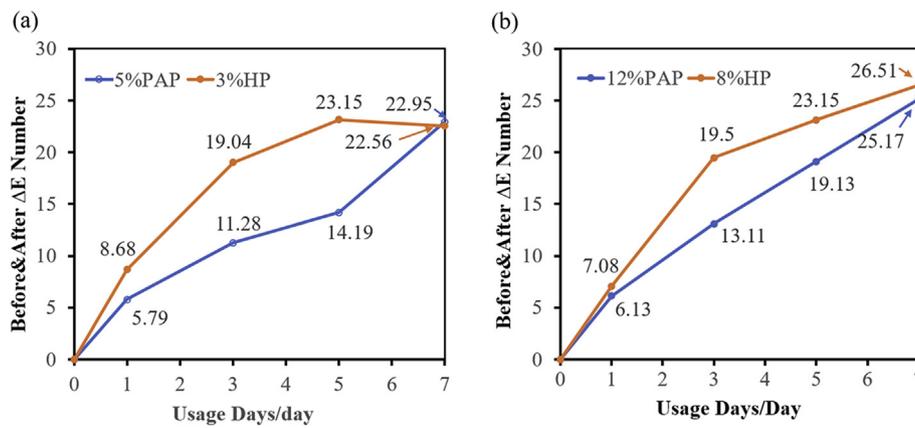


Fig. 1. The color change (ΔE) values of the bovine teeth whitened by a) 3% HP whitening gel against 5% PAP whitening gel, and b) 8% HP whitening gel against 12% PAP whitening gel. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

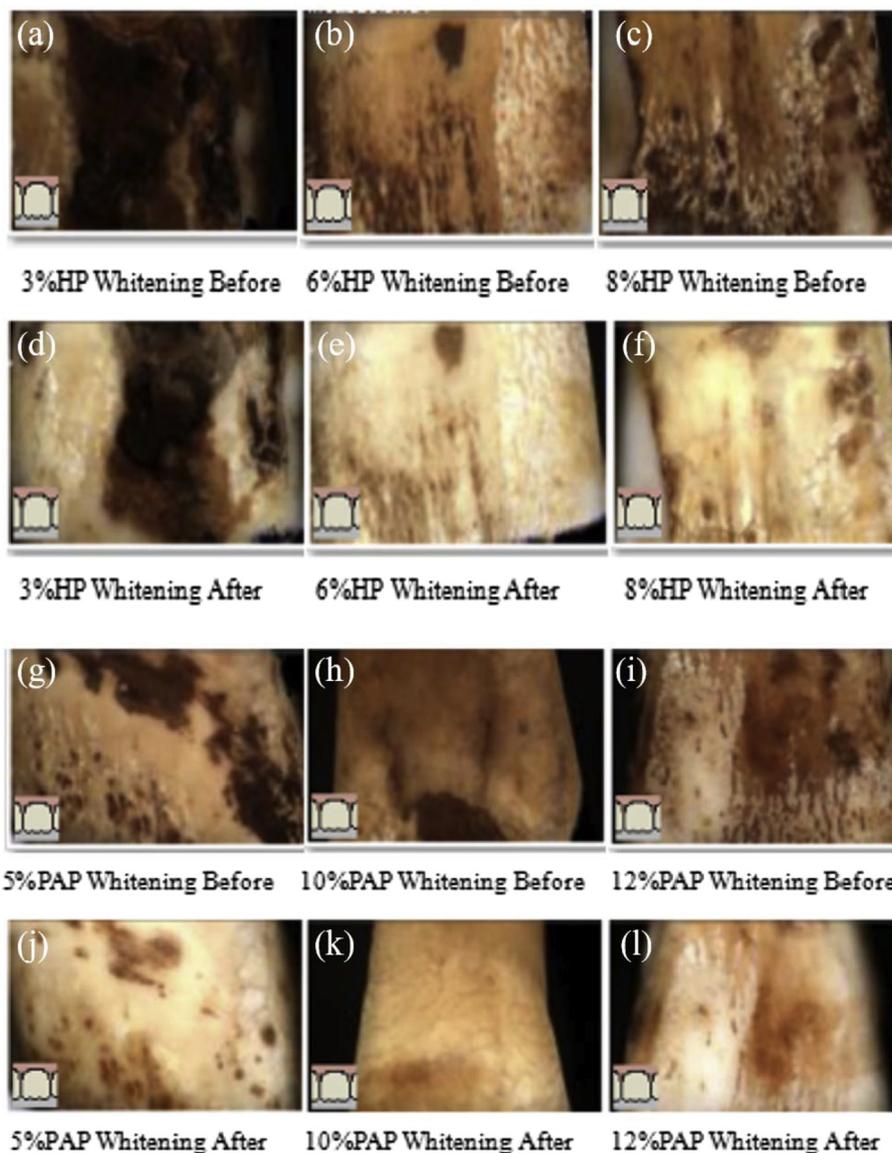


Fig. 2. The optical images of bovine teeth before whitening with a) 3%, b) 5% and c) 8% HP gels; and the corresponding teeth after whitening with d) 3%, e) 5% and f) 8% HP gels for 7 days. The bovine teeth before whitening with g) 5%, h) 10% and i) 12% PAP tooth whitening gels; and the corresponding teeth after whitening with j) 5%, k) 10% and l) 12% HP gels for 7 days.

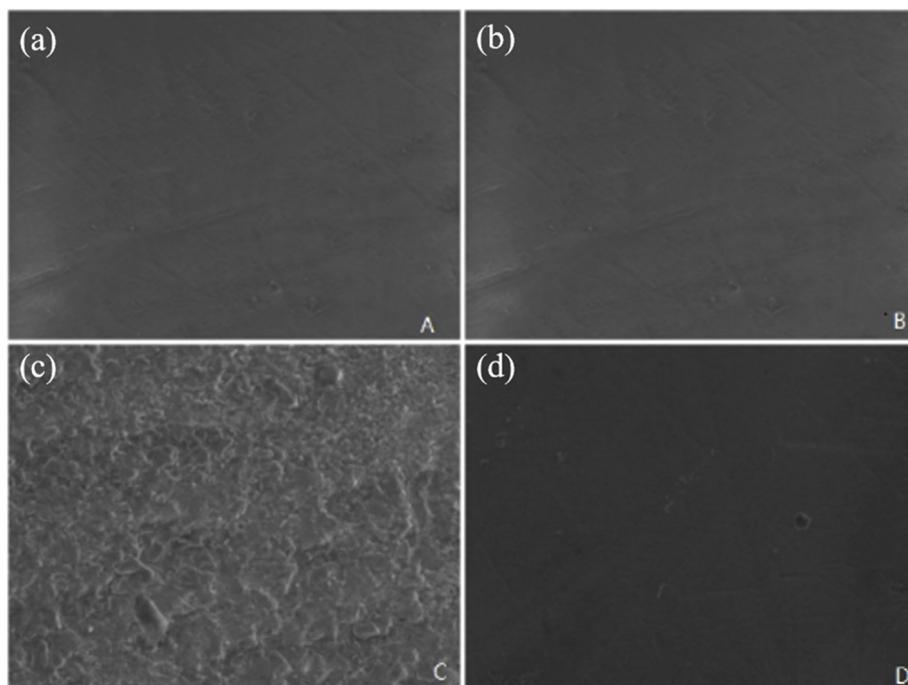


Fig. 3. Scanning electron microscopy images of a) the enamel of dyed bovine teeth, b) the bovine tooth enamel using 5% PAP gel, c) the bovine tooth enamel using 6% hydrogen peroxide gel, and d) the bovine tooth enamel using 12% PAP gel.

Table 1
Microhardness test results of each group of the bovine teeth.

Samples	Whitening gels	Vickers-hardness (kg/mm ²) ^a
Group A	–	292.91 ± 16.17 ^b
Group B	5% PAP	301.33 ± 31.72
Group C	6% HP	204.51 ± 15.73
Group D	12% PAP	345.19 ± 25.86

^a All average values with standard deviations were calculated from 20 samples.

^b The value was calculated from the dye bovine teeth before bleaching.

reliable. Moreover, the measurements and reports also indicated that composite tooth whitening gels with PAP can perfectly alternative the tooth whitening products with HP.

2. Materials and methods

2.1. Materials and the apparatus

HP tooth whitening gels with 3%, 6% and 8% were all provided by mixing water, glycerin, carbomer, peppermint oil and different concentrations of hydrogen peroxide into the composite gels. Meanwhile, all of the tooth whitening gels with 3%, 5%, 10% and 12% PAP were also prepared by mixing water, glycerin, carbomer, peppermint oil and different concentrations of PAP into the composite gels. The color changes of the bleached teeth were performed on a Spectro shade computer colorimeter (model: HXS-1000A, China). The SEM images were carried out on a scanning electron microscope (model: JEOL JSM-6380LV, Japan). The microhardness results were measured on a Micro Vickers under 300 g load for 15 s (model: HXS-1000A, China) [31].

2.2. Experiment section

2.2.1. Preparation of tea solution

4 g red tea was boiled in 80 mL pure water at 100 °C for 5 min. Then, the mixture was cooled to room temperature and then was filtered through the filter paper. After filtering, a solution of 100 mL was

prepared by fixing the solid with pure water [32].

2.2.2. Pre-treatment of bovine teeth

The commercial bovine teeth were disinfected with 3% hydrogen peroxide solution and soaked for 24 h to obtain the standard samples. The teeth were cut apart with a linear cutter, and the enamel part was taken as the observation object. The samples obtained from the cutting were polished to ensure that the samples had 1 mm thick enamel tissue [33] at least.

2.2.3. Post-treatment of bovine teeth

The pretreated bovine teeth samples were soaked in the prepared red tea solution. The red tea solution was changed every 24 h, and the dyed bovine teeth samples were taken out after 48 h and then soaked in artificial saliva for use.

2.2.4. Tooth bleaching effect measurements

Different concentrations of tooth whitening gels were smeared on the surface of stained bovine teeth samples. Then the samples were rinsed with clean water for 15 min and then wiped up. All the operation above should be performed once a day, and the whole whitening process was completed for 7 days. After bleaching, the Spectro shade computer colorimeter [34] was used to test the color of samples before and after whitening and record.

2.2.5. Surface morphology tests

The bovine teeth samples before and after whitening were respectively dried, fixed on the specimen carrier and treated with gold spraying on the surface. The surface morphology was then analyzed by SEM measurements [35].

2.2.6. Per irritation tests

Dental whitening composite gels with 12% PAP were applied to the teeth of 24 volunteers. After rinsing and cleaning for 15 min, the experiences of the volunteers were recorded. Similarly, composite gels with 8% HP were tested by the same 24 volunteers.

Table 2
Empirical trial test results of HP and PAP tooth whitening gels.^a

Evaluation Product Name	Taste (%)			Soreness (%)				Discerption of the Soreness
	Cool	Mid	Bitter	None	Mild	Moderate	Severe	
8% HP Gel	50.0	41.7	8.3	12.5	41.7	33.3	12.5	Burning sensation; Mildly allergic; Moderate hypersensitivity
12% PAP Gel	91.6	8.4	0	83.3	12.5	4.2	0	Mildly allergic

^a The results were reported by 24 volunteers.

3. Results and discussion

In order to evaluation the tooth bleaching effect of PAP. The whitening effect of both HP and PAP were also analyzed by Spectro shade computerized spectrophotometry, and the color change (ΔE) values were shown in Fig. 1. According to Fig. 1a, after using for 7 days, the whitening effect of dental whitening gels with 5% PAP were equivalent to the dental gels with 3% HP. Furthermore, learning from Fig. 1b, when 12% PAP was used, the higher ΔE values were appeared, indicating that higher concentration of PAP led to better whitening effect. Moreover, it also shown that the whitening effect of 12% PAP is equivalent to 8% HP. In addition, the linear ship between the ΔE values and the usage days also indicated that the whitening effect of the tooth whitening composite gels with 12% PAP were more controllable.

To intuitively learn the whitening effects of HP and PAP tooth whitening gels. The Spectro shade measurements were also used for intuitively learning the whitening effects of the tooth whitening gels with HP or PAP, and the results were given in Fig. 2. Comparing Fig. 2a–c with d–f. The HP gels shown well whitening effect. Moreover, the PAP gels also shown significant whitening effect on the stained bovine teeth (Fig. 2j–l vs. Fig. 2g–i). Furthermore, when comparing Fig. 2d–f with j–l, PAP gels exhibited the similar whitening effect to the commercialized HP gels, which was also agreed with the ΔE values in Fig. 1a and b.

Scanning electron microscopy (SEM) measurement of 4 groups of the teeth before/after bleaching with HP and PAP gels were then carried out to get insight into the demineralization phenomenon of the teeth using HP or PAP gels. As shown in Fig. 3, comparing with Fig. 3a and b, the enamels of the teeth were nearly unchanged. Moreover, even when the concentration of PAP increased to 12%, the enamel was also relatively unbroken, the demineralization phenomenon was not obvious, and the dissolution degree of enamel was still low. This results also indicated that PAP tooth whitening gels were nearly uninjurious for the enamels. However, when 6% HP gel was used, the enamel was obviously damaged and partly dissolved, and the degree of demineralization was also relatively serious. Therefore, in the process of whitening teeth, the whitening gel with PAP barely damaged the enamel on the tooth surface, and was proved to be safer than the hydrogen peroxide gel. On the other hand, according to the SEM measurement, the hydrogen peroxide gel would cause the damage of teeth enamel, then infiltrated into the dentin, and hence led to the teeth allergy.

The microhardness results of teeth in each sample groups before and after bleaching were shown in Table 1. When comparing with group A and group B, there was no significant difference between group A (292.91 ± 16.17) and group B ($301.33 + 31.72$) ($P > 0.05$). However, the differences among group A, B and D were statistically significant ($P < 0.05$). Moreover, as can be seen in Table 1, the microhardness of group B and group D was significantly higher than that of group C, indicating that PAP gel had almost no corrosion damage to teeth enamel. Nevertheless, the results of group B showed that hydrogen peroxide gel was damaging to the hardness of teeth. Therefore, it was clear that the PAP whitening gels were more safely and reliably to the teeth.

Empirical trial test results reported by 24 volunteers were also listed in Table 2. Learning from the reports, for the taste, 91.6% of the volunteers using tooth whitening products with 12% PAP gave their

experiences that the products tasted cool, while the other volunteers insisted that the products had general taste without any special experience. Nevertheless, when using 8% HP gels, 50% people think the taste is cool with no unpleasant smell, 41.7% people indicated that the taste was general and there was no special experience, and the other volunteers said the product tasted bitter. Moreover, in terms of causing soreness in the mouth, as shown in Table 2, the soreness columns, the major volunteers think the PAP gels had no soreness (83.3%), 12.5% volunteers had mild tooth pain, and only one reported that there were mild allergic symptoms in the dentin. Furthermore, the volunteers also reported that the pain and allergy symptoms completely were disappeared after 3 days since they discontinued the product. However, when the hydrogen peroxide tooth whitening products were used, only 12.5% volunteers reported no soreness, and 41.7% volunteers experienced mild toothache. More seriously, 33.3% volunteers reported to have moderately sore (one reported mild dentin hypersensitivity), and 12.5% people even bore severely sore (two reported that the lips were burning and the teeth were severely sensitivity). What's more, the teeth pain and allergy symptoms basically disappeared after 1–2 weeks when the product was discontinued. In summerly, it is obviously that PAP tooth whitening products are safer and more reliable in the sense of experience.

4. Conclusions

In this work, the PAP was firstly used in composite tooth-whitening gels instead of hydrogen peroxide. Comparing to HP tooth whitening gels, the PAP gels showed equally whitening effect to HP gels. Moreover, the surface morphology, and the hardness measurements of dyed bovine teeth indicated that the PAP based products were harmless to the teeth enamels, while HP gels would cause the damage of teeth enamel, and finally led to the teeth allergy. Furthermore, the taste irritation of teeth before/after whitening with PAP and HP gels were also carried out among 24 volunteers. These experiment results, as well as subjective feelings, also exhibited that the gels containing PAP had obvious whitening effect and no irritation to oral mucosa and dentin. Totally, PAP was proved to be an efficient material for composite tooth-whitening gels with peroxides and were much safer and more reliable than the commercialized HP based products.

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