Oxygen release of tetra acetyl ethylene diamine (TAED) and sodium perborate combination

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Abstract

Çelik EU, Türkün M, Yapar AGD. Oxygen release of tetra acetyl ethylene diamine (TAED) and sodium perborate combination. *International Endodontic Journal*, **41**, 571–576, 2008.

Aim To evaluate the effect of tetra acetyl ethylene diamine (TAED) on oxygen release from sodium perborate and to compare it with sodium perborate–hydrogen peroxide (H_2O_2) and sodium perborate–distilled water mixtures.

Methodology Six groups were evaluated: control groups (groups I–III), sodium perborate was mixed with distilled water or 3% or 30% H_2O_2 ; experimental groups (groups IV–VI), sodium perborate was mixed with TAED in different ratios and then distilled water was added to these mixtures. The amount of oxygen released from the samples was measured with a digital oxygen meter at room temperature (25 °C) and body temperature (37 °C) after 1 min, 1–6 h and 12 h and

Introduction

In the walking bleach technique, oxidizing agents such as sodium perborate mixed with water or hydrogen peroxide are placed in the pulp chamber of root-filled teeth and sealed with a temporary restoration (MacIsaac & Hoen 1994). Hydrogen peroxide is a widely accepted bleaching agent because of its excellent aesthetic results in a short time (Baratieri *et al.* 1995). However, its use has been associated with complications such as cervical root resorption (Harrington & Natkin 1979), increased dentinal permeability (Rotstein *et al.* 1991), alteration in the chemical structure of dentine (Rotstein *et al.* 1996) and changes on each day up to 1 week. The results were statistically evaluated by one-way analysis of variance and *post hoc* Tukey's tests.

Results The TAED groups demonstrated significantly higher amounts of released oxygen after 1 min and 1 h at 25 °C and 1 min at 37 °C (P < 0.05). At all other measurement times, the amount of TAED in each mixture did not alter the amount or speed of oxygen release (P > 0.05).

Conclusions Tetra acetyl ethylene diamine groups achieved their maximum oxygen release 2 h earlier at 25 °C and 1 h earlier at 37 °C than the other groups. Thus, TAED accelerated oxygen release from sodium perborate–distilled water mixtures regardless of its proportions up to 24 h.

Keywords: hydrogen peroxide, oxygen release, sodium perborate, TAED.

Received 12 July 2007; accepted 4 January 2008

in the biomechanical properties of dentine (Lewinstein *et al.* 1994). Because of these complications, it has been recommended that use of hydrogen peroxide applications should cease (Cvek & Lindvall 1985, Rotstein *et al.* 1991) and alternative bleaching agents used (Spasser 1961, Weiger *et al.* 1994).

Spasser (1961) introduced sodium perborate-water mixtures into the pulp chamber as a bleaching agent. Some report later replaced the water with 30-35%hydrogen peroxide to improve the whitening (Nutting & Poe 1967). Although mixing sodium perborate with 3-30% H₂O₂ was found to be more effective than mixing it with water, postoperative external cervical root resorption has been reported to occur after intracoronal bleaching of discoloured root-filled teeth (Friedman *et al.* 1988). Therefore, sodium perborate application with water rather than hydrogen peroxide has been recommended to prevent or minimize the occurrence of bleaching-related external root

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resorption; however, the whitening effect of the sodium perborate–water mixtures can take longer to be effective (Weiger *et al.* 1994).

Tetra acetyl ethylene diamine (TAED) is an oxygen activator that has been used to activate bleaching reactions at low temperatures, and its use enables bleaching under such condition and milder pH. As a result of its unique characteristics, it is widely used in the bleaching system of detergent, textile and papermaking industries (Hera (Human & Environmental Risk Assessment on ingredients of European household cleaning products) 2002). In view of its widespread industrial uses, TAED may be an alternative means of increasing the effectiveness of bleaching and reducing the period of bleaching therapy of sodium perborate– water mixtures in dentistry.

The purpose of this study was to examine the effect of TAED on the oxygen release of sodium perborate by comparing it with sodium perborate– H_2O_2 and sodium perborate–distilled water mixtures.

Material and methods

Preparation of degassed water

Degassed water was used as a medium and the amount of released oxygen from the samples into the liquid was measured. Deionized water was degassed by a vacuum method: 8.5 L water was stirred under vacuum (160 mm Hg) for 20 min. Half of the degassed water medium was stored in an incubator at 25 °C for a day whilst the other half was stored at 37 °C.

Preparation of the experimental groups

Sodium perborate (tetra hydrate), distilled water, 3% and 30% hydrogen peroxide and TAED were utilized. Six groups were prepared (Table 1). In the control groups (groups I–III), sodium perborate was mixed with distilled water or 3% or 30% H_2O_2 in a ratio of 2 : 1 (g mL⁻¹) (Attin *et al.* 2003). In experimental groups (groups IV–VI), TAED was mixed with sodium perborate in different ratio (1 : 8 to 1 : 2) and then distilled water was immediately added to these mixtures in a ratio of 2 : 1.

Transfer of mixtures to degassed water

Samples were mixed according to the proportions mentioned above and then transferred to a glass container holding 15 mL degassed water. The orifices of the glass containers were sealed with a silicon mould that had a hole at the centre for entrance of the oxygen probe. The diameter of the hole was standardized according to the diameter of the oxygen probe to prevent possible O_2 release from the container during the measuring process. The half of the glass containers including the sample was kept in an incubator at 25 °C, whilst the other part was maintained at 37 °C.

Amount of released oxygen

The amount of released oxygen (in p.p.m.) from the samples was measured with a digital oxygen meter at 25 °C and 37 °C, after 1 min, after every hour from 1 to 6 h and at 12 h and then once a day for 7 days (Hargreaves & Tucker 2002, Global Learning and Observations to Benefit the Environment Program 2005). Fresh samples were used for each time period. Three measurements were performed for each of six groups at 15 different time periods at room temperature (25 °C) and body temperature (37 °C). The pH and temperature of the solutions were also measured with the digital oxygen meter. The probe was calibrated each day (Hargreaves & Tucker 2002, Global Learning and Observations to Benefit the Environment Program 2005).

The results were statistically evaluated by one-way analysis of variance and *post hoc* Tukey's tests (P = 0.05).

Results

The amount of oxygen released, pH and temperature of each group for each measurement time are shown in Tables 2–5.

The TAED groups revealed significantly greater amounts of oxygen than the other groups at 1 min and 1 h at 25 °C and 1 min at 37 °C (P < 0.05). At all other times, the amount of TAED did not influence the volume or speed of oxygen release (P > 0.05).

Whilst the TAED groups (groups IV–VI) achieved their maximum oxygen release range after 1 min, the H_2O_2 and distilled water groups (groups I–III) achieved their maximum range after 2 h at 25 °C (Fig. 1). Similarly, at 37 °C, the H_2O_2 and distilled water groups achieved their maximum range after 1 h, whilst the TAED groups achieved this stage after 1 min (Fig. 2).

Discussion

Sodium perborate in the form of mono-, tri- or tetra hydrate is used as a H_2O_2 -releasing agent and has been

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I able 1 Preparation of the experimental groups	Study groups	Mixtures	S.P./solution ratio	TAED/S.P. ratio	n			
<u>Frontha</u>	I	Sodium perborate (1 mg)	2:1	_	90			
		Distilled water (0.5 mL)						
	II	Sodium perborate (1 mg)	2:1	-	90			
		3% H ₂ O ₂ (0.5 mL)	0.5 mL)					
	III	Sodium perborate (1 mg)	2:1	-	90			
		30% H ₂ O ₂ (0.5 mL)						
	IV	Sodium perborate (1 mg)	2:1	1:8	90			
		TAED (0.125 mg)						
		Distilled water (0.5 mL)						
	V	Sodium perborate (1 mg)	2:1	1:4	90			
		TAED (0.250 mg)						
		Distilled water (0.5 mL)						
	VI	Sodium perborate (1 mg)	2:1	1:2	90			
		TAED (0.5 mg)						
		Distilled water (0.5 mL)						

Table 2 The amount of released oxygen (ppm) at time intervals up to 12 h at 25 °C and their min-max pH during this period

Groups	1 min	1 h	2 h	3 h	4 h	5 h	6 h	12 h	pH min–max
I	5.2 ± 0.74	17.6 ± 0.63	27.9 ± 0.35	28.2 ± 0.82	27.9 ± 0.67	27.4 ± 1	28.1 ± 0.97	25.9 ± 0.55	9.5–10.1
II	7.5 ± 0.33	19.3 ± 0.82	28.6 ± 0.74	29.1 ± 1.2	29.4 ± 0.46	28.8 ± 0.73	28.3 ± 0.36	29.2 ± 1.06	9–9.3
III	12.7 ± 0.63	22.5 ± 0.57	34.1 ± 1.18	33.2 ± 0.71	33.7 ± 0.38	33.9 ± 1.08	32.5 ± 0.53	32.9 ± 0.74	8.2-8.8
IV	32.4 ± 1.06	32.8 ± 0.95	32.2 ± 0.47	31.6 ± 1.02	32.5 ± 0.41	33.1 ± 0.35	33.4 ± 1.11	31.1 ± 0.26	10.1-10.2
V	33.3 ± 0.77	32.7 ± 0.84	33.9 ± 0.27	33.4 ± 0.59	32.1 ± 1.14	34.9 ± 0.82	33.3 ± 0.44	33.4 ± 1.27	10.1–10.3
VI	35.2 ± 1.02	35.7 ± 0.52	34.6 ± 0.58	35.1 ± 1.29	35.7 ± 1.2	34.4 ± 1.2	35.5 ± 0.71	34.1 ± 0.68	10.2-10.2

Table 3 The amount of released oxygen (ppm) at time intervals up to 7 days at 25 °C and their min-max pH during this period

Groups	1 day	2 days	3 days	4 days	5 days	6 days	7 days	pH min–max
1	26.3 ± 0.27	22.7 ± 0.63	20.4 ± 1.3	17.8 ± 0.86	16.2 ± 1.15	15.7 ± 0.51	15.9 ± 0.49	10.1–10.1
II	28.7 ± 0.42	25.9 ± 0.14	22.7 ± 0.77	20.3 ± 1.21	18.2 ± 0.48	17.7 ± 0.38	15.5 ± 0.84	9.2–9.3
III	33.1 ± 0.93	32.8 ± 0.53	30.3 ± 0.85	27.7 ± 1.06	24.6 ± 0.43	23.5 ± 0.78	19.9 ± 0.88	8.7–8.8
IV	32.7 ± 0.66	31.6 ± 1.02	28.1 ± 0.71	26.5 ± 0.88	24.89 ± 0.14	23.6 ± 0.37	18.3 ± 0.57	10.1–10.3
V	33.2 ± 0.58	31.3 ± 1.2	30.2 ± 0.55	27.9 ± 0.64	25.7 ± 0.92	23.8 ± 0.46	19.4 ± 0.72	10.1–10.3
VI	34.5 ± 0.53	31.6 ± 0.44	30.9 ± 1.34	28.6 ± 0.78	24.6 ± 0.83	22.1 ± 0.52	20.3 ± 0.37	10.2–10.3

Table 4 The amount of released oxygen (ppm) at time intervals up to 12 h at 37 °C and their min-max pH during this period

Groups	1 min	1 h	2 h	3 h	4 h	5 h	6 h	12 h	pH min–max
I	2.7 ± 0.45	18.4 ± 0.83	18.1 ± 0.64	19.2 ± 0.38	18.6 ± 1.32	18.4 ± 0.18	19.3 ± 0.62	18.9 ± 0.27	9.4–10.1
II	5.4 ± 0.92	20.6 ± 0.48	21.3 ± 1.09	20.1 ± 1.15	20.2 ± 0.62	20.6 ± 0.81	21.4 ± 0.52	20.8 ± 0.34	9–9.3
III	10.3 ± 0.41	22.5 ± 0.57	22.6 ± 0.19	21.8 ± 0.35	21.9 ± 1.45	22.3 ± 0.13	21.7 ± 0.28	22.5 ± 1.05	8.2-8.7
IV	23.8 ± 1.02	24.6 ± 0.43	24.1 ± 0.14	24.4 ± 0.55	23.7 ± 0.28	24.1 ± 0.36	24.8 ± 0.73	23.3 ± 0.29	10.2-10.2
V	25.4 ± 0.44	26.6 ± 0.48	25.8 ± 0.17	25.6 ± 0.47	26.5 ± 0.23	26.7 ± 0.14	25.3 ± 0.36	26.8 ± 1.21	10.2–10.2
VI	27.3 ± 0.11	28.1 ± 0.63	28.7 ± 0.18	28.9 ± 0.76	27.6 ± 1.15	28.2 ± 0.32	27.9 ± 0.24	28.4 ± 0.42	10.2–10.3

employed as an oxidizer and bleaching agent especially in washing powder and other detergents. The whitening efficacy of sodium perborate mono-, tri- or tetra hydrate mixtures with either water or hydrogen peroxide is similar. Hydrogen peroxide is released during the decomposition of perborate. The released H₂O₂ can generate different radicals or ions depending on the pH value, the influence of light, temperature, existence of cocatalysts and metallic reaction partners (Feinman et al. 1991). Perhydroxy radicals are created

Groups	1 day	2 days	3 days	4 days	5 days	6 days	7 days	pH min-max
I	18.3 ± 0.52	15.6 ± 0.14	12.9 ± 0.63	10.4 ± 0.12	9.2 ± 0.37	8.3 ± 0.62	7.6 ± 0.74	10.1–10.1
II	21.8 ± 0.44	18.2 ± 0.26	16.3 ± 0.48	14.4 ± 0.73	12.6 ± 0.17	10.7 ± 0.19	9.9 ± 0.58	9.3–9.3
III	22.7 ± 0.35	19.9 ± 0.54	17.8 ± 0.83	16.1 ± 0.68	15.3 ± 0.54	12.6 ± 0.23	10.4 ± 0.43	8.7-8.7
IV	23.6 ± 0.15	21.4 ± 0.19	19.5 ± 0.34	17.8 ± 0.44	15.7 ± 0.76	14.8 ± 0.71	12.6 ± 0.64	10.2–10.3
V	27.1 ± 1.03	25.3 ± 0.28	23.2 ± 0.92	21.3 ± 0.82	19.2 ± 0.46	17.4 ± 0.65	15.7 ± 0.51	10.2-10.2
VI	29.4 ± 0.27	27.6 ± 0.39	25.6 ± 0.38	23.5 ± 0.71	22.8 ± 0.33	20.7 ± 0.14	19.4 ± 0.75	10.2-10.2

Table 5 The amount of released oxygen (ppm) at time intervals up to 7 days at 37 °C and their min-max pH during this period



Figure 1 The amount of released oxygen (ppm) from the mixtures tested at 25 °C.



Figure 2 The amount of released oxygen (ppm) from the mixtures tested at 37 °C.

preferably in an alkaline environment resulting in effective bleaching agents (Feinman *et al.* 1991). These products are formed after the cleavage of H_2O_2 and are responsible for the oxidative and reductive and, therefore, the bleaching properties of H_2O_2 . The radicals can crack unsaturated double bonds of long, coloured molecules or reduce the coloured metallic oxides such as Fe₂O₃ (Fe⁺³) to colourless FeO (Fe⁺²).

Several studies have reported variations in bleaching effectiveness by comparing mixtures of sodium perborate with distilled water or H_2O_2 in different concentrations. Rotstein *et al.* (1993) and Weiger *et al.* (1994) report no significant differences in the effectiveness of

sodium perborate mixed with 3% or 30% H_2O_2 or distilled water. The shade stability of teeth treated by a mixture of perborate and water is as high as the shade stability of teeth in which a mixture of sodium perborate with 3% or 30% H_2O_2 was used. Other surveys found that mixing sodium perborate with 30% H_2O_2 was more effective than mixing it with water (Ho & Goerig 1989, Warren *et al.* 1990). Although, Freccia *et al.* (1982) reported that the walking bleach technique with a mixture of 30% H_2O_2 and sodium perborate was as effective as the thermocatalytic technique. Studies of the efficacy of intracoronal bleaching agents in artificially discoloured teeth indi-

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cate that further bleaching sessions are required to achieve the desired aesthetic result when a sodium perborate–distilled water mixture is used (Ho & Goerig 1989, Frysh *et al.* 1995). The increased number of treatment sessions will increase the cost of the treatment. From both economic and safety perspectives, it will be desirable to achieve the aesthetic change in the minimum number of treatment sessions, thus the exposure of hydrogen peroxide to the periradicular tissue will be minimized.

It is known that sodium perborate has a great bleaching effect at higher temperatures. Thus, the application of heat during bleaching increases its effectiveness; however, the heat can promote cervical root resorption (Harrington & Natkin 1979, Attin *et al.* 2003).

Tetra acetyl ethylene diamine is an excellent lowtemperature bleaching activator in household detergents (Hera (Human & Environmental Risk Assessment on ingredients of European household cleaning products) 2002). In aqueous liquor, anions of hydrogen peroxide react with the ester or amide of the bleaching activators to generate a corresponding peroxyacid, which oxidizes the stained substrate. At alkaline pH and in the presence of a source of hydrogen peroxide such as perborate or percarbonate, TAED undergoes rapid perhydrolysis yielding peracetic acid (PAA) and diacetyl ethylene diamine (DAED). The reaction is a stepwise process via the intermediate triacetyl ethylene diamine (TriAED). PAA and its salt, peracetate, are highly efficient oxidizing agents that bleach a number of stubborn stains (tea, red wine, food stains, etc.). Peracetate oxidizes stains and is itself converted to acetate. Perhydrolysis is completed within 15 min at 30-60 °C. Thus, this activator accelerates the oxygen release of the bleaching material (Clariant AG -Surfactants Division 1999).

Tetra acetyl ethylene diamine/sodium perborate ratio differs from 1/8 to 1/2 in detergent formulations. Quantities of more than this ratio in the formulation only provide insignificant improvement in the bleaching performance of the entire formulation (Hera (Human & Environmental Risk Assessment on ingredients of European household cleaning products) 2002). Thus, TAED was used in our study according to this ratio.

In the present study, TAED accelerated the oxygen release of the sodium perborate–water mixture both at 25 °C and 37 °C. Furthermore, it increased the amount of released oxygen because of the oxygen molecules in its structure. These effects of TAED are important in

terms of decreasing the duration of treatment with sodium perborate–water mixtures and increasing the effectiveness of the bleaching process.

Application of TAED to the mixture of sodium perborate-water did not decrease the pH of the mixture. During bleaching, an alkaline pH is desirable because the whitening effectiveness of buffered alkaline H_2O_2 is significantly higher than that of unbuffered H₂O₂ (Frysh et al. 1995). In addition, an acidic pH of the bleaching agent can contribute to the complications associated with the walking bleach technique. When 30% H₂O₂ is mixed with sodium perborate in a ratio of 2:1 (g mL⁻¹), the pH is alkaline. If further 30% H₂O₂ is added, it becomes acidic (Kehoe 1987, Rotstein et al. 1991); whereas the pH increases significantly as the concentration of H₂O₂ decreases. As with previous studies, sodium perborate-water mixtures demonstrated higher pH values than sodium perborate-H₂O₂ mixtures; however, all of the solutions were alkaline.

Conclusions

Tetra acetyl ethylene diamine accelerated oxygen release from sodium perborate–distilled water mixtures and increased the amount of released oxygen in the first 1 or 2 h depending on the temperature. Thus, this oxygen activator has the potential to reduce the duration of bleaching therapy and may increase its effectiveness. However, further laboratory and clinical studies are needed to examine the effects of TAED on discoloured root-filled teeth and to evaluate whether the acceleration in the release of oxygen from bleaching agents has any significant effect on the duration and effectiveness of the bleaching process.

Acknowledgements

We would like to thank Dr Remzi Seyfioğlu (Department of Environmental Science, Dokuz Eylül University, Turkey) for his support in preparing the degassed water and the use of the oxygen probe.

References

- Attin T, Paque F, Ajam F, Lennon AM (2003) Review of the current status of tooth whitening with the walking bleach technique. *International Endodontic Journal* 36, 313–29.
- Baratieri LN, Ritter AV, Monterio S, de Andrada MAC, Vieira LCC (1995) Nonvital tooth bleaching: guidelines for the clinician. *Quintessence International* 26, 597–608.

- Clariant AG Surfactants Division (1999) Clean and clever way of bleaching, Clariant Marketing brochure. Muttenz, Switzerland.
- Cvek M, Lindvall AM (1985) External root resorption following bleaching of pulpless teeth with oxygen peroxide. *Endodontics & Dental Traumatology* **1**, 56–60.
- Feinman RA, Madray G, Yarborough D (1991) Chemical, optical, and physiologic mechanisms of bleaching products: a review. Practical Periodontics and Aesthetic Dentistry 3, 32–6.
- Freccia WF, Peters DD, Lorton L, Bernier WE (1982) An *in vitro* comparison of nonvital bleaching techniques in the discoloured tooth. *Journal of Endodontics* 8, 70–7.
- Friedman S, Rotstein I, Libfeld H, Stabholz A, Heling I (1988) Incidence of external root resorption and esthetic results in 58 bleached pulpless teeth. *Endodontics & Dental Traumatology* 4, 23–6.
- Frysh H, Bowles WH, Baker F, Rivera-Hidalgo F, Guillen G (1995) Effect of pH on hydrogen peroxide bleaching agents. *Journal of Esthetic Dentistry* **7**, 130–3.
- Globe (Global Learning and Observations to Benefit the Environment) Program (2005) Dissolved oxygen protocol.
- Hargreaves JA, Tucker CS (2002) Measuring Dissolved Oxygen Concentration in Aquaculture. Southern Regional Aquaculture Center, Stoneville, MS: SRAC publication No. 4601.
- Harrington GW, Natkin E (1979) External resorption associated with bleaching of pulpless teeth. *Journal of Endodontics* 5, 344–8.
- Hera (Human & Environmental Risk Assessment on ingredients of European household cleaning products) (2002) *Tetra acetyl ethylene diamine (TAED)*. Hera publication No: CAS 10543-57-4 Brussels, Belgium.
- Ho S, Goerig AC (1989) An *in vitro* comparison of different bleaching agents in the discoloured tooth. *Journal of Endodontics* 15, 106–11.

- Kehoe JC (1987) pH reversal following in vitro bleaching of pulpless teeth. Journal of Endodontics 13, 6–9.
- Lewinstein I, Hirschfeld Z, Stabholz A, Rotstein I (1994) Effect of hydrogen peroxide and sodium perborate on the microhardness of human enamel and dentin. *Journal of Endodontics* 20, 61–3.
- MacIsaac AM, Hoen CM (1994) Intracoronal bleaching: concerns and considerations. *Journal of Canadian Dental* Association 60, 57–64.
- Nutting EB, Poe GS (1967) Chemical bleaching of discoloured endodontically treated teeth. *Dental Clinics of North America* 31, 655–62.
- Rotstein I, Torek Y, Lewinstein I (1991) Effect of bleaching time and temperature on the radicular penetration of hydrogen peroxide. *Endodontics & Dental Traumatology* 7, 196–8.
- Rotstein I, Mor C, Friedman S (1993) Prognosis of intracoronal bleaching with sodium perborate preparations *in vitro*: 1-year study. *Journal of Endodontics* **19**, 10–2.
- Rotstein I, Dankner E, Goldman A, Heling I, Stabholz A, Zalkind M (1996) Histochemical analysis of dental hard tissues following bleaching. *Journal of Endodontics* 22, 23–5.
- Spasser HF (1961) A simple bleaching technique using sodium perborate. *New York State Dental Journal* **27**, 332–4.
- Warren MA, Wong M, Ingram TA 3rd (1990) In vitro comparison of bleaching agents on the crowns and roots of discoloured teeth. Journal of Endodontics 16, 463–7.
- Weiger R, Kuhn A, Lost C (1994) In vitro comparison of various types of sodium perborate used for intracoronal bleaching of discoloured teeth. Journal of Endodontics 20, 338–41.

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