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Evaluation of pH and Optical Properties of Dual Rinse HEDP Irrigating Solution

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Abstract: This study investigates the pH values and optical characteristics of Dual Rinse HEDP, either independently or combined with sodium hypochlorite (NaOCl), and compares them to other irrigants used in endodontics. The solutions used in this study were commercially acquired and prepared, followed by pH measurements using a pH meter and spectral analysis using UV/Vis spectrophotometry in specified wavelengths of the ultraviolet (UV) C (190–280 nm), UVB (281–315 nm), UVA (316–400 nm), visible light (VL) (401–780 nm), and near-infrared (NIR) spectra (781–1100 nm). The pH analysis revealed alkaline values for NaOCl, EDTA, Dual Rinse HEDP, and the HEDP + NaOCl combination, an acidic value for citric acid, and nearly neutral values for chlorhexidine and distilled water. Spectral analysis revealed the notable absorption characteristics of endodontic irrigants. In the UV range, all solutions exhibited higher absorption values to water ($p < 0.05$), with Dual Rinse HEDP resembling EDTA and citric acid, and HEDP + NaOCl resembling NaOCl. The NIR region highlights absorption peaks around 975 nm for all solutions, including NaOCl and Dual Rinse HEDP + NaOCl, suggesting potential applications in laser-activated irrigation. This study provides comprehensive insights into the pH and optical features of endodontic irrigants, emphasizing their potential roles in enhancing disinfection strategies and optimizing laser-activated irrigation protocols.

Keywords: root canal irrigation; endodontics; pH value; UV/Vis spectrophotometry; absorption coefficient; laser activated irrigation; etidronic acid

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1. Introduction

Successful endodontic therapy is achieved through a combination of proper preparation, effective cleaning, and three-dimensional obturation of the root canal. However, it is widely accepted that the disinfection of the root canal, through procedures involving the removal of pulp tissue, microorganisms, and the smear layer, is crucial for successful outcomes with endodontic therapy [1,2]. Conventional endodontic treatment involves the chemo-mechanical preparation of the root canal, which includes mechanical cleaning and irrigation with various solutions. The irrigation process allows for the removal of dentin debris and dissolution of necrotic and residual pulp tissue, acts as a lubricant, and eliminates the smear layer [1,3]. It is particularly important that irrigation achieves a cleaning effect in the complexities and extremities of the canal system that are not accessible to mechanical preparation [4].

Based on the effects they produce, irrigating solutions are divided into groups of antiseptics and chelating agents [1]. Most notable antiseptics are chlorhexidine (CHX) and sodium hypochlorite (NaOCl). The latter is commonly used due to its antimicrobial activity, tissue-dissolving properties, detergent role, and ability to neutralize toxic by-products [5,6]. However, antiseptics lack the capability to remove the smear layer. Therefore, chelating agents such as 15–17% Ethylenediaminetetraacetic acid (EDTA) and 10–20% citric acid (CA) are additionally introduced to irrigation protocols [1,3]. It is recommended to alternate the use of both groups of irrigating solutions during mechanical preparation [3], which led to the development of various combined irrigants with debatable effectiveness [7–11]. HEDP (1-hydroxyethylidene 1,1-disphosphonate), also known as etidronic acid, belongs to the group of chelating agents that is commercially available as Dual Rinse HEDP [12,13]. It can be utilized independently, where it produces a mild chelating effect, or in combination with NaOCl for a procedure known as continuous chelation irrigation [13,14]. This combination retains the desired antimicrobial and proteolytic effects of NaOCl whilst adding an element of decalcification to the mixture and simplifying the procedure [12–15].

The chemical efficacy of irrigants is influenced by various factors, with pH value emerging as a significant one. The pH of the solution has been demonstrated to affect the tissue-dissolving, antimicrobial, and chelating properties of diverse irrigants [16,17]. Furthermore, combining NaOCl with other solutions is shown to alter its pH values, thereby modifying the effectiveness of this solution [18,19]. To date, studies on the combination of HEDP with NaOCl have mostly shown an alkaline pH for this mixture [20–22]. However, these results were often arising from the application of chemically synthesized etidronate rather than the commercial product, as well as different solutions or concentrations of NaOCl with distinct pH values.

The properties of the sole irrigating solutions are not sufficient to achieve the desired disinfection of the canal. Another pivotal factor in the efficiency of irrigants is their activation in the root canal. Active irrigation methods, such as ultrasonic activation and laser-activated irrigation (LAI), have been shown to achieve superior antimicrobial and chelating effects compared to passive irrigation [1,23]. LAI is effective in eliminating bacterial infection and significantly contributes to the enhanced long-term success of endodontic therapy when compared with conventional techniques [24]. The effectiveness of LAI is based on a phenomenon called cavitation. This phenomenon occurs when the static pressure in a liquid descends below its vapor pressure, leading to the formation of vapor-filled voids. The subsequent collapse of these bubbles generates shock waves, thereby augmenting the cleaning efficacy through the propulsion of the irrigants within the canal. This irrigation flow expels debris from the canal, eliminating the smear layer and biofilm from the dentin surface [25,26]. Currently, wavelengths ranging from 1000 to 3000 nm are predominantly employed in LAI [26–28]. However, other groups of lasers are gaining popularity in endodontics such as diode lasers which operate at wavelengths in the visible and near-infrared (NIR) region [29]. As of now, the literature on the application of diode lasers in LAI is limited [30]. Additionally, the literature on the application of diode lasers in combination with Dual Rinse HEDP in endodontics is notably scarce.

The primary objective of our study was to investigate the pH value of the independent solution of Dual Rinse HEDP or in combination with NaOCl, and compare it with other irrigants used in endodontics. Additionally, we aimed to examine the optical characteristics of these solutions within the ultraviolet, visible and NIR wavelength range up to 1100 nm. The null hypothesis that was tested assumes that there is no difference in pH values and optical characteristics between Dual Rinse HEDP solutions and other tested irrigants.

2. Materials and Methods

2.1. Preparation of Irrigating Solutions

The solutions used in this study were commercially acquired and prepared, if needed, according to the manufacturer's instructions. Dual Rinse HEDP was prepared following the user instructions by dissolving the content of one capsule in 10 mL of distilled water

or in 10 mL of 2% NaOCl. Citric acid at a concentration of 20% was prepared by diluting 5 mL of 40% citric acid in 5 mL of distilled water. A list of the used solutions and their concentrations, brand names, and manufacturers is shown in Table 1.

Table 1. Tested irrigating solutions with their corresponding concentrations and sources.

Product	Concentration % (*w*/*v*)	Brand Name	Manufacturer
Distilled water	100	N/A	N/A
Sodium hypochlorite	2	Chloraxid 2%	Cerkamed, Stalowa Wola, Poland
Ethylenediaminetetraacetic Acid	17	E.D.T.A.	Imicryl Dental, Konya, Turkey
1-hydroxyethylidene 1,1-disphosphonate	9	Dual Rinse HEDP	Medcem, Weinfelden, Switzerland
1-hydroxyethylidene 1,1-disphosphonate + sodium hypochlorite	9 HEDP 2 NaOCl	Dual Rinse HEDP Chloraxid 2%	Medcem, Weinfelden, Switzerland
Citric acid	20	Citric Acid 40%	Cerkamed, Stalowa Wola, Poland
Chlorhexidine digluconate	2	Gluco Chex 2%	Cerkamed, Stalowa Wola, Poland

2.2. pH Measurements

The pH measurements were conducted using the SevenDirect SD20 pH meter (Mettler Toledo, Greifensee, Switzerland). The methodology adhered to the manufacturer's guidelines and standard practices for reliable pH determinations in solutions. Prior to sample analysis, pH electrodes were calibrated with buffer solutions. Following calibration, the pH electrode was carefully immersed into 5 mL of each sample solution, and sufficient stabilization time was allowed. The pH readings were recorded, and the electrode was rinsed with distilled water between measurements to prevent cross-contamination. Calibration with buffer solutions and periodic checks ensured the accuracy of the pH readings. Measurements were performed five times for each solution.

2.3. Spectral Analysis

Irrigating solutions were subjected to UV/Vis spectrophotometry (Shimadzu UV-1800; Shimadzu, Kyoto, Japan) following the previously described method [31]. Spectral data were collected at 1 nm intervals covering the wavelength range of the 190–1100 nm region using a synthetic quartz cuvette free from OH absorption (Hellma Analytics, Müllheim, Germany). The obtained absorption values were used to calculate the transmission (T) and the absorption coefficient alpha (α) using UVProbe software (v2.42; Shimazu). These calculations resulted in transmission measurements between 0.01 and 99%, which were plotted relative to the wavelength, and alpha values between 0.04 and 40 cm^{-1} [31]. To properly discern statistically significant differences in the alpha values among the various test solutions, we specified the wavelength ranges among which the solutions were compared. The wavelength ranges were as follows: ultraviolet (UV) C range (190–280 nm), UVB range (281–315 nm), UVA range (316–400 nm), visible light (VL) range (401–780 nm), and near-infrared (NIR) range (781–1100 nm).

2.4. Statistical Analysis

After obtaining data from pH measurements and spectral analysis, statistical analyses were conducted using IBM SPSS Statistics software (v23.0; IBM Corp, Armonk, NY, USA). Descriptive statistics were employed to characterize the pH values, while alpha values, derived from spectral measurements, underwent a comparative analysis between different solutions using the Kruskal–Wallis test. This non-parametric test was chosen due to the nature of the absorbance coefficient data and their non-normal distribution as derived from the Kolmogorov–Smirnov test. All data are presented as means $\pm$ standard deviation (SD). A p value less than 0.05 was set to indicate statistical significance.

3. Results

3.1. pH Values of Endodontic Irrigants

The pH measurements of different irrigating solutions showcased significant variations in acidity and alkalinity (Figure 1, Table 2). Distilled water (DW) presented a nearly neutral pH, while sodium hypochlorite (NaOCl) demonstrated a markedly alkaline pH. Dual Rinse HEDP and the HEDP + NaOCl combination displayed substantial alkalinity, with HEDP presenting the highest pH value out of all tested irrigants. Similarly, EDTA exhibited an alkaline pH, while CA showed high acidity. Chlorhexidine (CHX) displayed a slightly acidic pH. Concurrently, the potential difference (mV) readings underscored the electrochemical variances between the solutions, with each irrigating solution demonstrating distinctive millivolt values as presented in Table 2.

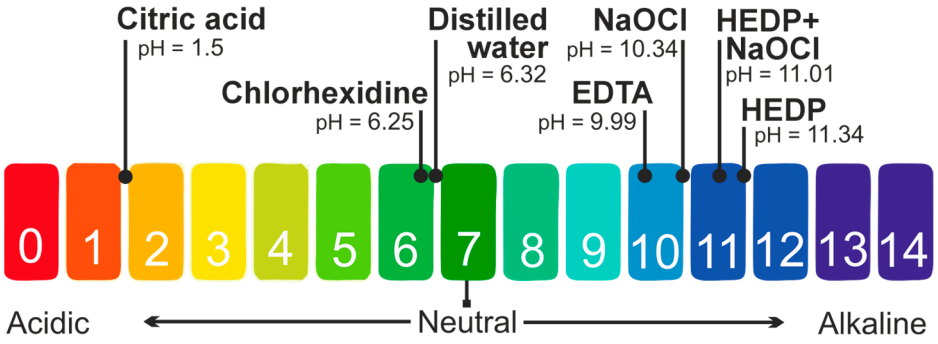


Figure 1. Illustration of pH values of each irrigating solution on a pH scale from the most acidic to the most alkaline. Abbreviations: NaOCl—sodium hypochlorite; EDTA—ethylenediaminetetraacetic acid; HEDP—1-hydroxyethylidene 1,1-disphosphonate; HEDP + NaOCl—1-hydroxyethylidene 1,1-disphosphonate + sodium hypochlorite.

Table 2. Chemical properties of distilled water and endodontic irrigants presented as pH value, difference in potential, and temperature of the solutions. Results are presented as mean ± standard deviation (SD) of five measurements.

Irrigating Solution	pH	Potential Difference (mV)	Temperature (°C)
DW	6.32 ± 0.01	54.5 ± 0.02	24.5 ± 0.4
NaOCl	10.34 ± 0.01	−184.5 ± 0.01	24.7 ± 0.3
HEDP	11.29 ± 0.01	−239.0 ± 0.01	23.6 ± 0.6
HEDP + NaOCl	11.01 ± 0.02	−223.8 ± 0.03	24.4 ± 0.1
EDTA	9.99 ± 0.01	−164.4 ± 0.02	24.6 ± 0.2
CA	1.5 ± 0.01	328.5 ± 0.01	24.4 ± 0.3
CHX	6.25 ± 0.02	53.5 ± 0.05	24.9 ± 0.2

Abbreviations: DW—distilled water; NaOCl—sodium hypochlorite; EDTA—ethylenediaminetetraacetic acid; HEDP—1-hydroxyethylidene 1,1-disphosphonate; HEDP + NaOCl—1-hydroxyethylidene 1,1-disphosphonate + Sodium hypochlorite; CA—citric acid; CHX—chlorhexidine digluconate.

3.2. Spectral Analysis

The transmission values of the different irrigating solutions in wavelengths from 190 to 1100 nm are presented on Figure 2. Differences in the absorption coefficient alpha (α) values between all irrigating solutions in different wavelength ranges are presented in Table 3.

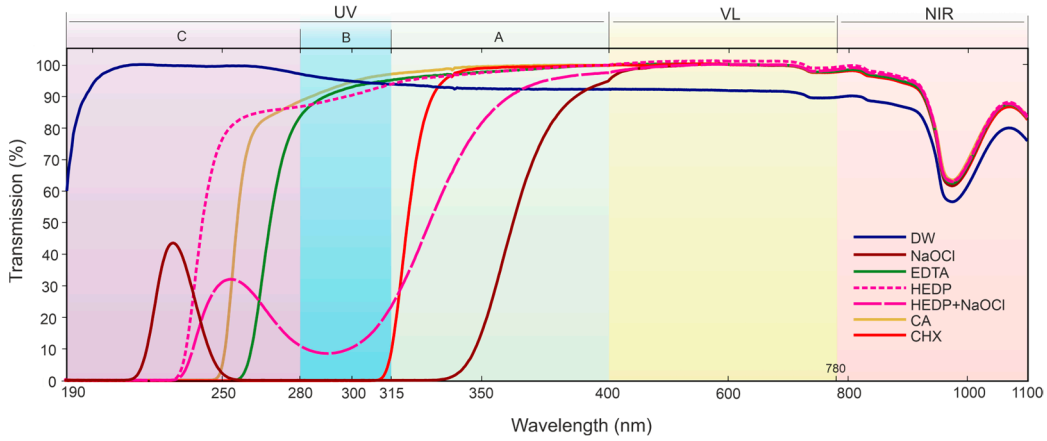


Figure 2. Transmission values of distilled water, NaOCl, EDTA, Dual Rinse HEDP, Dual Rinse HEDP + NaOCl, citric acid, and chlorhexidine at different wavelengths of ultraviolet, visible light, and near-infrared regions. Abbreviations: UV—ultraviolet; VL—visible light; NIR—near infrared; C—ultraviolet C; B—ultraviolet B; A—ultraviolet A DW—distilled water; NaOCl—sodium hypochlorite; EDTA—ethylenediaminetetraacetic acid; HEDP—1-hydroxyethylidene 1,1-disphosphonate; HEDP + NaOCl—1-hydroxyethylidene 1,1-disphosphonate + sodium hypochlorite; CA—citric acid; CHX—chlorhexidine digluconate.

Table 3. Mean values of absorption coefficients (α) of distilled water and endodontic irrigants at different wavelengths of ultraviolet, visible light, and near-infrared regions. Results are presented as mean ± standard deviation (SD).

	Wavelength Region of the Spectrum				
Irrigating Solution	**UVC**	**UVB**	**UVA**	**VL**	**NIR**
DW	0.14 ± 0.33 †,‡	0.22 ± 0.04 †,‡	0.34 ± 0.02 †,‡	0.39 ± 0.04 †,‡	1.11 ± 0.65 †,‡
NaOCl	24.66 ± 13.85 *	38.67 ± 0.84 *,‡	12.51 ± 14.56 *,‡	0.04 ± 0.05 *,‡	0.73 ± 0.65 *
HEDP	27.52 ± 12.81 *,†	0.47 ± 0.10 *,†	0.11 ± 0.08 *,†	0.02 ± 0.03 *,†	0.66 ± 0.63 *
HEDP + NaOCl	20.98 ± 14.18 *,‡	9.49 ± 1.27 *,†,‡	1.43 ± 1.70 †,‡	0.04 ± 0.04 *,‡	0.68 ± 0.63 *
EDTA	28.85 ± 14.13 *	0.40 ± 0.15 *,†	0.10 ± 0.06 *,†	0.02 ± 0.04 *,†	0.71 ± 0.63 *
CA	24.59 ± 16.20 *	0.31 ± 0.12 †	0.05 ± 0.04 *,†	0.02 ± 0.04 *,†	0.68 ± 0.59 *
CHX	39.40 ± 1.20 *,‡	35.55 ± 7.31 *,‡	0.70 ± 1.84 *,†,‡	0.03 ± 0.04 *,†,‡	0.75 ± 0.65 *

* indicates statistically significant difference ($p < 0.05$) compared to distilled water; † indicates statistically significant difference ($p < 0.05$) compared to sodium hypochlorite; ‡ indicates statistically significant difference ($p < 0.05$) compared to ethylenediaminetetraacetic acid. Abbreviations: UV—ultraviolet; VL—visible light; NIR—near infrared; DW—distilled water; NaOCl—sodium hypochlorite; EDTA—ethylenediaminetetraacetic acid; HEDP—1-hydroxyethylidene 1,1-disphosphonate; HEDP + NaOCl—1-hydroxyethylidene 1,1-disphosphonate + sodium hypochlorite; CA—citric acid; CHX—chlorhexidine digluconate.

3.2.1. Spectral Properties of Water

The absorption coefficient of DW in the UVC range remained consistently low (mean $\alpha = 0.14$ cm^{-1}) with notable absorption peak observed at the beginning of the spectrum, around 190 nm ($\alpha = 2.18$ cm^{-1}). In the UVB region, the mean α values were slightly higher but still considerably low, as well as in the UVA and VL ranges. Significant fluctuations in transmission and absorption values were observed in the NIR range. The absorption in DW began to increase around 920 nm, reaching a peak value at 975 nm ($\alpha = 2.48$ cm^{-1}), then decreased towards 1070 nm, with another increase observed towards 1100 nm ($\alpha = 1.21$ cm^{-1}).

3.2.2. Spectral Properties of Endodontic Irrigants

As shown in Figure 2, the transmission values of the different irrigating solutions in the VL and NIR ranges mostly followed that of DW, with similar decrease and increase curves in the NIR range from 915 nm to 1100 nm. The most notable differences were

observed in UV regions, with all solutions showing significantly lower transmission values at different ranges of the UV spectrum compared to DW.

Each endodontic irrigating solution presented a high absorption coefficient in the UVC range of the spectrum, with the mean α values showing a statistically significant difference to DW (Table 3; $p < 0.05$). NaOCl presented peak absorption at the start of the spectrum around 205 nm ($\alpha = 39.21$ cm^{-1}), followed by a strong decrease at 230 nm ($\alpha = 3.62$ cm^{-1}) and an increase to a second peak at 270 nm ($\alpha = 39.59$ cm^{-1}). Similar absorption values were observed for Dual Rinse HEDP at the beginning of the UVC range with peak absorption at 210 nm ($\alpha = 34.32$ cm^{-1}); however, going to the UVB range of the spectrum, a strong decrease was observed. HEDP showed no statistical difference to EDTA and CA ($p > 0.05$) but presented statistically significant higher mean α values compared to DW and NaOCl ($p < 0.05$). HEDP + NaOCl presented high absorption at the start of the spectrum, reaching a peak around 210 nm ($\alpha = 39.21$ cm^{-1}), followed by strong decrease reaching the lowest absorption at 255 nm ($\alpha = 4.46$ cm^{-1}), after which the absorption coefficient rose to the end of the UVC region. The mean α values for HEDP + NaOCl showed a statistically significant difference to DW and EDTA ($p < 0.05$) but no difference to NaOCl ($p > 0.05$). EDTA and CA showed similar α values to each other and statistically significant higher absorption values compared to DW ($p < 0.05$), with major peaks at around 240 nm for EDTA ($\alpha = 39.59$ cm^{-1}) and around 230 nm for CA ($\alpha = 39.21$ cm^{-1}). After initial high absorption, both solutions presented decreased absorption values going to the UVB range of spectrum. Unlike all previous solutions, CHX presented constant high absorption values throughout the UVC region (mean $\alpha = 39.40$ cm^{-1}) with only minor oscillations.

The UVB region of the spectrum was characterized by decreasing absorption values for all endodontic solutions, except NaOCl and CHX, which remained constantly high throughout (mean $\alpha = 38.67$ cm^{-1} and $\alpha = 35.56$ cm^{-1}, respectively). All endodontic irrigants, except CA, presented statistically significant higher α values compared to DW ($p < 0.05$). However, both EDTA and HEDP, while not showing a statistical difference between them, showed statistically significant lower absorption values in the UVB region compared to NaOCl ($p < 0.05$). On the contrary, HEDP + NaOCl showed significantly higher absorption values compared to EDTA ($p < 0.05$), with a peak value of $\alpha = 10.72$ cm^{-1} at around 290 nm. Still, its absorption coefficient was significantly lower to that of NaOCl ($p < 0.05$). It should be noted that CHX presented a significant decrease in absorption at the end of the UVB region of the spectrum.

In the UVA region, the absorption coefficients were low in almost all tested solutions, with the mean α values being lower than in DW. Only considerably higher absorption values were observed at the beginning of the UVA spectrum for NaOCl and HEDP + NaOCl, showing values of $\alpha = 39.21$ cm^{-1} and $\alpha = 6.32$ cm^{-1} at 316 nm, respectively, and dropping to $\alpha = 0.24$ cm^{-1} and $\alpha = 0.12$ cm^{-1} at 400 nm.

The trend of low-to-no absorption of the irrigant solutions was continued through the VL range of the spectrum. All tested solutions presented significantly lower absorption values (mean values of $\alpha \leq 0.04$ cm^{-1}) compared to DW ($p < 0.05$). Only minor peaks were observed at 757 nm for NaOCl ($\alpha = 0.11$ cm^{-1}), at 738 nm for HEDP ($\alpha = 0.06$ cm^{-1}), at 737 nm for HEDP + NaOCl ($\alpha = 0.10$ cm^{-1}), at 738 nm for EDTA ($\alpha = 0.11$ cm^{-1}), at 739 nm for CA ($\alpha = 0.06$ cm^{-1}), and at 741 nm for CHX ($\alpha = 0.12$ cm^{-1}).

All tested solutions showed significantly lower mean absorption values compared to DW ($p < 0.05$) and no statistically significant difference between them ($p > 0.05$) in the NIR region of the spectrum. Interestingly, all tested solutions presented a strong increase in absorption from around 940 nm, all reaching a similar peak absorption at 975 nm followed by a decrease in higher wavelengths. The peak values at 975 nm were $\alpha = 2.11$ cm^{-1}, $\alpha = 2.00$ cm^{-1}, $\alpha = 2.02$ cm^{-1}, $\alpha = 2.05$ cm^{-1}, $\alpha = 1.98$ cm^{-1}, and $\alpha = 2.11$ cm^{-1} for NaOCl, HEDP, HEDP + NaOCl, EDTA, CA, and CHX, respectively. Notably, none of the tested solutions showed a statistically significant difference to NaOCl and EDTA ($p > 0.05$). At the end of the observed spectrum (1100 nm), all tested solutions presented a tendency towards

increased absorption coefficients, which corresponded to decreased transmission values, as presented in Figure 2.

4. Discussion

As previously noted, irrigation presents a crucial step in endodontic procedures that ensures the successful outcome of the treatment [1,2]. It usually involves the alternating use of antiseptic and chelating solutions to ensure antimicrobial and tissue-dissolving effects together with the removal of the smear layer. Considering potential chemical interactions, some of which are deemed potentially dangerous, care should be taken to prevent the mixing of different solutions by intercepting the procedure with saline or distilled water [32]. In 2005, the concept of continuous chelation emerged, introducing the possibility of combining NaOCl with a weak chelating agent, such as HEDP [12]. Dual Rinse HEDP presents a versatile endodontic irrigation product of etidronic acid. It can be dissolved in distilled water to create a 9% HEDP solution, acting as a mild chelator. Alternatively, when dissolved in NaOCl, it yields a solution with both antiseptic and chelating properties, suitable for continuous chelation and simplifying the irrigation procedure [13]. While the combination of NaOCl and HEDP has shown promising results in terms of antimicrobial and chelating effects, it is noteworthy that certain aspects of this innovative irrigating solution, such as its pH values, remain relatively unexplored. Understanding the pH characteristics is crucial as it directly influences the chemical environment within the root canal. Additionally, the optical properties of Dual Rinse HEDP in combination with NaOCl are of significant interest, particularly in the context of laser irrigation. However, limited information is currently available on these aspects, warranting further investigation to comprehensively assess the irrigants' properties and potential implications for clinical use. The null hypothesis of the study is partially rejected. Although there were differences in pH values between all irrigating solutions, optical characteristics were significantly different only between Dual Rinse HEDP solutions and some of the other tested irrigants at specific wavelengths.

The pH of endodontic irrigants is a critical parameter that directly impacts their efficacy in dissolving tissue, removing the smear layer, providing antimicrobial action, ensuring biocompatibility, preserving dentin, and contributing to overall treatment success [16]. Clinicians should carefully consider the pH of irrigants as part of the decision-making process when selecting and using these solutions during endodontic procedures. The mechanism of action of NaOCl involves its hydrolysis to hypochlorous acid and its further dissociation into mainly hypochlorite and hydrogen ions. This reaction is reversible and is highly dependent on the pH of the solution [33]. The antimicrobial effectiveness of NaOCl is primarily attributed to its elevated pH. At high pH, it disrupts the integrity of the cytoplasmic membrane, resulting in irreversible enzymatic inhibition, disturbances in cellular metabolism, and the degradation of phospholipids through lipid peroxidation processes [34]. Our findings align with previous research regarding the highly alkaline nature of NaOCl, supporting its well-established role as an effective antimicrobial agent in endodontics [34]. Another antimicrobial agent in the focus of our study was CHX. CHX is a wide-spectrum antiseptic that, due to its cationic nature, is capable of binding to the negatively charged surfaces. It can bind to bacteria, damaging the outer layers of the cell wall, or to surfaces of the oral mucosa or hydroxyapatite of dentine, leading to substantive antimicrobial activity [35]. In our study, CHX presented a pH of 6.25, which is in range for its optimal antimicrobial activity (the range of 5.5 to 7.0) reported previously [36].

While both EDTA and CA achieve their mechanisms of action through the chelation of calcium ions from dentine, they differ in pH levels. Specifically, the CA in our study possessed a highly acidic nature with a pH of 1.5. Previously, the low pH of citric acid was shown to yield more active chelating properties in this solution [37]. On the contrary, most EDTA solutions used in endodontics have pH values from neutral (pH = 7.5) to more alkaline (pH = 9.0) [38]. The 17% EDTA solution tested in our study showed alkalinity with

a pH of 9.99, which is known to have a milder chelating effect compared to more neutral pH levels [38].

In our investigation, Dual Rinse HEDP revealed an alkaline pH, consistent with findings by other authors [20]. This alignment with the observed alkalinity of EDTA suggests a shared capacity for effective chelation [39]. However, the most important factor to note is the effect of combining HEDP and NaOCl on the pH of this mixture. In the continuous chelation concept, NaOCl should maintain its pH and available hypochlorite with an additional decalcifying effect during the approximate course of an endodontic treatment. When EDTA is mixed with NaOCl, it usually results in a rapid and dramatic decrease in free available chlorine, significantly reducing the ability of NaOCl to dissolve organic tissue [40]. Recently, more alkaline solutions of EDTA (tetrasodium salt EDTA) were introduced for continuous chelation with NaOCl. This combination resulted in the longer maintenance of both free available chlorine and desirable pH conditions [41]. It has been previously shown that HEDP in aqueous solution, when freshly mixed with NaOCl, impedes neither the antimicrobial properties of the NaOCl [15,42] nor its ability to dissolve necrotic tissue [43]. This is reflected in our study, where mixing Dual Rinse HEDP with NaOCl did not produce a significant change in the pH value of this mixture. However, we did not explore the effects of mixing these solutions on chemical aspects such as available chlorine and stability. This limitation underscores the need for future research in these areas.

Spectral analysis offers insights into how these solutions interact with light across different wavelengths, providing valuable information for applications such as laser-activated irrigation. LAI in endodontics involves the use of lasers with specific wavelengths that are well suited for interactions with water, a key component of irrigating solutions. The effect of LAI is based on cavitation, a physical phenomenon that induces the formation and implosion of vapor bubbles at the fiber tip, causing very rapid fluid movement in the canal [25,26]. LAI has been shown to have a greater efficacy in the removal of canal debris and greater antibacterial effect than other activation techniques [44–46]. For LAI to be effective, the high absorption of laser energy into the irrigating liquid is necessary. In a root canal setting, it has to be taken into account that the radiation that is not absorbed by irrigants can potentially cause unwanted side effects such as damage to the canal wall or to the periradicular tissues. As Meire et al. [31] proposed, LAI is safe to use if the absorption coefficient of the irrigants is not below 10 cm^{-1}. In the present study, we employed UV–Visible–NIR spectrophotometry to explore the transmission values of different irrigating solutions across a broad wavelength range, from ultraviolet (UV) to visible light (VL) and near-infrared (NIR), and to compare them to the well-established spectral properties of water.

The transmission and absorption data for water align with previously published optical absorption data on DW available in the literature [47]. In our research, DW exhibited consistently low absorption coefficients in the UV range of the spectrum, which is consistent with previous reports [31]. On the contrary, endodontic irrigants presented high variations in absorption values. In the UVC region, all irrigating solutions presented high absorption values over 30 cm^{-1}. This can be attributed to electronic transitions occurring within the molecular structures of the irrigants. Electronic transitions involve the movement of electrons between different energy levels within atoms or molecules, typically induced by the absorption of photons. These transitions may result from the excitation of electrons from the non-bonding n and bonding π orbitals to the π^* antibonding orbitals, leading to the absorption of photons with wavelengths corresponding to the UV range [48]. Interestingly, HEDP demonstrated comparable absorption to EDTA and CA, while the combination of HEDP + NaOCl presented a complex absorption profile, similar to that of NaOCl. This complexity in the absorption profile may arise from the interaction between the molecules of HEDP and NaOCl, leading to changes in the electronic structure and resulting in a unique absorption spectrum. NaOCl was observed to cause molecular electronic transitions when mixed with other substances, as previously shown in combination with EDTA [49].

In the UVB region, most solutions showed decreasing absorption values, except NaOCl and CHX, which maintained high absorption coefficients. Previous research reported that CHX absorbs light with the wavelength of 280 nm (UVB range), while it does not absorb light in the UVA range of the spectrum [50]. In fact, most solutions, including CHX, generally exhibited low absorption in the UVA region in our study. The only exceptions were NaOCl and HEDP + NaOCl, which displayed higher absorption values around 316 nm. High absorption values for NaOCl have been shown previously in wavelengths between 250 nm and 350 nm, with peak values at around 300 nm [31,51]. These absorption peaks can be attributed to specific electronic transitions occurring within the hypochlorite ion, leading to the absorption of photons in the UV region. Specifically, transitions involving the oxygen–chlorine bond within the hypochlorite ion and transitions from n orbitals to π^* orbitals may contribute to the observed absorption peaks [48]. However, absorption is inversely related to the decomposition of NaOCl, as products of its reduction, such as hydroxyl ions, do not absorb energy in this spectral region [51]. In clinical settings, NaOCl decomposes in the presence of organic matter, inflammatory exudate, tissue remnants, and microbial biomass in the root canal [35]. This implies that the absorption profiles of solutions can be different in clinical settings when both irrigants and lasers interact with other constituents of the root canal, necessitating further research. Excimer lasers such as argon fluoride, krypton fluoride, and xenon chloride operate in the UV spectrum at wavelengths of 193, 248, and 308 nm [52–54]. They have been studied for various applications, including refractive eye surgery [55], dermatological procedures [56], and dentistry [54], because of their ability to ablate tissues with minimal heat. The effects of excimer lasers are achieved when high-energy UV photons are absorbed by the target material, which leads to the formation of excited states and the generation of reactive chemical species through a photochemical process. Reactive chemical species contribute to the breaking of molecular bonds and the removal of material, resulting in ablation. Excimer lasers are also known for low absorption in water [57]. Conversely, our findings suggest high absorption values for NaOCl, HEDP + NaOCl, and CHX in all wavelengths utilized by excimer lasers, making them potential candidates for use in LAI. However, one limitation could be the reported degradation of chemical compounds by UV light [58], thus leaving the use of excimer lasers in LAI, and their effects on irrigating solutions, to be studied in the future.

Our study revealed that all tested irrigating solutions, as well as DW, showed little to no absorption in the VL range of spectrum. These results are in line with the previous findings of Meire et al. [31] and Pegau et al. [59]. However, unlike in our study, Meire et al. [31] reported a slightly higher absorption coefficient for CHX at 513 nm, with a value of α = 0.42 cm^{-1}. In our study, the only observable peak absorption of α = 0.12 cm^{-1} was registered at 714 nm for CHX. This discrepancy in results could be attributed to differences in composition as well as the concentration of irrigants used in both studies. In fact, their study reported the variability in the absorption profiles of CHX at different concentration [31]. Nevertheless, the low absorption values of irrigants in the VL range deems these wavelengths unsuitable for LAI. However, lasers operating in the VL range have been used in endodontics, such as KTP lasers emitting at 532 nm that have been studied for sterilizing root canals [60,61] and He-Ne lasers at 633 nm that were used for Laser Doppler Flowmetry [62]. Another promising aspect of laser-aided endodontics is photodynamic therapy that involves the use of a photosensitive dye (photosensitizer) and a low-intensity laser light to achieve antibacterial and tissue disinfection effects within the root canal system [63,64]. Photosensitizers aid the rise in the absorption coefficient of water in root canals, making lasers effective in the VL region [63]. Diode lasers operating at 635 nm have shown promising efficiency in photodynamic endodontic therapy, where the photosensitizer has been combined with conventional chemo-mechanical debridement [64].

Diode lasers with wavelengths ranging from 810 to 980 nm and neodymium-doped yttrium aluminum garnet (Nd:YAG) lasers operating at approximately 1064 nm have been extensively studied in endodontics within the NIR spectrum [65]. However, most research has centered around their application in laser irradiation rather than LAI. Notably, previous

studies have suggested that water and endodontic irrigants exhibit low absorption and high transmission values in the NIR range, seemingly making these wavelengths suboptimal for LAI [31]. Despite this, both diode lasers and Nd:YAG lasers have demonstrated effectiveness in LAI. For instance, Lauterborn and Ohl [66] successfully generated cavitation bubbles in water using Nd:YAG lasers with very short pulses (8 ns) and lenses to tightly focus the laser beam, while Kushwah et al. [67] employed a diode laser at 980 nm in combination with intermittent irrigation using distilled water, achieving a superior bactericidal effect compared to the conventional use of NaOCl. In another study, de Macedo et al. [68] tested the effect of Nd:YAG (1064 nm) and diode (980 nm) lasers on EDTA agitation, showing the more positive effects of LAI than the conventional use of EDTA. Additionally, Neelakantan et al. [69] tested the efficacy of LAI with a diode laser (940 nm) in combination with 18% HEDP + NaOCl and found a highly bactericidal effect. However, their study used chemically synthesized 18% HEDP, unlike the commercially available 9% HEDP in our study. Nevertheless, the effectiveness of these wavelengths could be traced to the results of our study, where both water and endodontic irrigants presented a significant absorption peak at around 975 nm, with transmission levels of around 60%. Interestingly, the optical properties of NaOCl and HEDP + NaOCl in the NIR spectrum were found to be similar, as were those of HEDP and EDTA, in our investigation. The intriguing optical characteristics observed in our study underscore the need for dedicated research into the potential applications of these combinations in LAI within the NIR spectrum.

In discussing the observed absorption peaks, it is worth considering the possibility of overtones, where higher-energy transitions involving vibrational or rotational excitations within the molecules contribute to the observed absorption features. Previous studies have reported overtones in the absorption spectra of certain molecules in the NIR range, such as water and organic compounds, typically observed as weaker absorption peaks at wavelengths approximately twice the energy of the fundamental transitions [70,71]. While specific overtones were not explicitly reported in our study, the presence of absorption peaks beyond those expected solely from electronic transitions suggests the possibility of overtones contributing to the observed absorption spectra. Further investigations focusing on vibrational and rotational modes within the molecules could help elucidate the presence and significance of overtones in the absorption spectra of endodontic irrigants, providing additional insights into their optical behavior in the NIR spectrum.

Despite the comprehensive exploration of the optical properties of endodontic irrigants across different wavelengths in our study, it is important to acknowledge a significant limitation. Our investigation primarily focused on the UV–Visible–NIR wavelength range, due to the limitations of our spectrophotometer, which encompasses wavelengths up to approximately 1100 nm. However, the infrared spectrum, particularly the wavelengths commonly employed in erbium lasers (around 2940 nm), was not within the scope of our study. Erbium lasers have gained attention in LAI for their ability to interact with water and hydroxyapatite effectively, due to high absorption levels at that wavelength. Meire et al. [31] reported high absorption coefficients in the infrared spectrum, especially at 2940 nm, for DW, NaOCl, EDTA, CA, and CHX. Numerous studies have reported the superior antimicrobial and cleaning effects of erbium lasers in LAI with NaOCl and EDTA [46,72,73]. However, there are a limited number of studies on HEDP + NaOCl in LAI with erbium lasers showing bactericidal [69] and sealer-removal effects [15] with promising results. Given the similar optical properties of HEDP + NaOCl to those of NaOCl in our study, it is reasonable to speculate that they would demonstrate similar optical properties in the infrared spectrum. Considering its significance in endodontic applications, future research should focus on exploring the optical properties of Dual Rinse HEDP in the infrared spectrum, particularly in the context of erbium laser activation.

5. Conclusions

Within its limitations, our study provides valuable insights into the pH values and optical properties of endodontic irrigants, with a specific emphasis on Dual Rinse HEDP.

Notably, HEDP exhibited substantial alkalinity, aligning with its potential as an effective chelating agent that can be safely combined with NaOCl without changing its pH. Dual Rinse HEDP solutions demonstrated intriguing absorption profiles across various wavelength ranges, with notable peak absorptions at 210 nm and 975 nm and at 210 nm, 290 nm, 316 nm, and 975 nm for HEDP and HEDP + NaOCl, respectively, presenting potential applications in laser-assisted endodontics. While our study contributes to the understanding of HEDP's properties, future research should extend into the infrared spectrum to comprehensively evaluate its suitability for endodontic applications, including laser-activated irrigation.

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