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ORIGINAL ARTICLE

Impact of ultraviolet radiation-induced damage to hair fiber integrity: A multi-technique physicochemical characterization of surface and cortex properties

Nathana Cindy Barros Silva Ramos[1] | **Alzira Xavier Pinto Dini**[2] | **Viviane Cristina Albarici**[2] | **Mayara Miranda Ferreira**[2] | **Caroline Vieira Hercolino**[2] | **Adriano da Silva Pinheiro**[2] | **André Rolim Baby**[1] | **Maria Valéria Robles Velasco**[1]

[1] Faculdade de Ciências Farmacêuticas, University of São Paulo, São Paulo, Brazil

[2] Grupo Kosmoscience Ciência e Tecnologia Cosmética Ltda, Valinhos, Brazil

Correspondence
Nathana Cindy Barros Silva Ramos, Faculdade de Ciências Farmacêuticas, University of São Paulo, São Paulo, SP, Brazil.
Email: nathana-ramos@usp.br

Abstract

The combination of chemist techniques is a powerful tool on hair's structure observation and its behaviour when submitted to different process, like chemical damages as bleaching and ultraviolet (UV) radiation exposition, or physical damages, like successive brushing. This study mimicked solar radiation damage suffered by hair fibres during Brazilian summer (3 months of intense sun), and compared this kind of damage with the ones caused by bleaching process using different techniques. Natural hair after radiation exposition showed cuticular damages compared to the effects caused by bleaching. The increase of cystic components oxidation and hydrophobicity decrease were observed when natural hair was exposed to 96 and 192 h of UV radiation. Additionally, keratin's degradation enthalpy of damaged hair showed a decrease of mean values in comparison to natural healthy hair. These changes can be explained due to the protein and lipidic oxidative damages caused by UV radiation, causing alterations on cuticular level. Regarding cortical damage, natural hair tresses exposed to UV radiation for 192 h exhibited a significant decrease in mechanical parameters—specifically, force at 20% elongation and elongation at break—when compared with the initial condition of both bleached and double-bleached hair. This suggests that UV exposure exerts a more pronounced impact on these mechanical properties than chemical oxidation alone. In contrast, the enthalpy measurements show that the chemical degradation caused by bleaching is comparable to the structural alterations observed after 192 h of UV exposure. Moreover, double bleaching results in substantially more severe degradation of structural proteins than that induced by UV radiation.

KEYWORDS

bond oxidation, contact angle, differential scanning calorimetry, hair's mechanical behaviour, infrared spectroscopy, protein integrity, ultraviolet radiation

Résumé

La combinaison de techniques issues de la chimie constitue un puissant outil pour observer la structure du cheveu et son comportement lorsqu'il est. soumis à différents types de procédés, tels que des agressions chimiques (comme la décoloration et l'exposition aux rayonnements ultraviolets) ou des agressions physiques (comme des brossages répétés). Cette étude a reproduit les dommages liés au rayonnement solaire subis par les fibres capillaires pendant l'été brésilien (3 mois d'ensoleillement intense) et a comparé ce type de dommages à ceux induits par des procédés de décoloration utilisant différentes techniques. Les cheveux naturels exposés aux rayonnements ont présenté des altérations de la cuticule comparables à celles observées après une décoloration. Une augmentation de l'oxydation des composants cystéiniques et une diminution de l'hydrophobicité ont été observées après 96 et 192 heures d'exposition aux rayonnements ultraviolets. Par ailleurs, l'enthalpie de dégradation de la kératine des cheveux endommagés a montré une diminution des valeurs moyennes par rapport à des cheveux naturels sains. Ces modifications s'expliquent par des dommages oxydatifs affectant les protéines et les lipides sous l'effet des UV, entraînant des altérations au niveau de la cuticule. Concernant les dommages corticaux, les mèches de cheveux naturels exposées aux UV pendant 192 heures ont présenté une diminution significative des paramètres mécaniques (notamment la force à 20% d'allongement et l'allongement à la rupture) par rapport à l'état initial, y compris en comparaison avec des cheveux décolorés et doublement décolorés. Ce résultat suggère que l'exposition aux UV a un impact plus marqué sur ces propriétés mécaniques que l'oxydation chimique seule. En revanche, les mesures d'enthalpie montrent que la dégradation chimique induite par la décoloration est. comparable aux altérations structurelles observées après 192 heures d'exposition aux UV. De plus, une double décoloration entraîne une dégradation nettement plus importante des protéines structurales que celle induite par les rayonnements UV.

INTRODUCTION

Human hair fibres exhibit a hierarchical organization comprising the cuticle, cortex, and, when present, the medulla. Their biochemical structure is predominantly proteinaceous, with keratins forming the major component. High cysteine content enables extensive disulfide cross-linking, which underpins the fibre's tensile strength and structural resilience. Melanin pigments embedded within the cortex determine hair colour. Furthermore, a diverse lipid fraction—including 18-methyleicosanoic acid—contributes to cuticular cohesion, surface hydrophobicity and modulation of inter-fibre friction, parameters closely associated with the sensory perception of hair dryness [1–7].

Natural hair can undergo changes in fibre properties, such as strength, gloss, flexibility and protein content, when exposed to physical damage (e.g., brushing, blow drying and flat ironing) or chemical damage (e.g., dyeing, acid or alkaline straightening) and ultraviolet (UV) radiation.

Robbins and Bahl [8] reported that UVA and UVB radiation can alter the mechanical properties of hair fibres by affecting the disulfide bonds in cystine. Tolgyesi [9] described, via photolysis mechanisms, that structural amino acids in hair—such as cystine, tyrosine, phenylalanine and parts of tryptophan—absorb UV radiation, leading to the formation of free radicals.

Joekes and Nogueira demonstrated that UVA and UVB radiation cause damage primarily to the disulfide bonds in the cuticle's amino acids, as this layer absorbs the greatest amount of radiation and is therefore most affected [9–11]. This exposure also leads to cortical damage, resulting in colour loss, protein degradation and altered mechanical behaviour of the fibres. Moreover, it was observed that darker pigmentation offers greater resistance to UV damage [11, 12].

Several studies [9, 12–18] have shown that UVB radiation (295–315 nm) is responsible for the degradation of proteins and amino acids vital to hair fibre structure, such as tryptophan (absorption at 280 nm), tyrosine (275 nm), cystine, methionine and histidine. This protein degradation disrupts disulfide bonds and leads to cysteic acid formation as a byproduct, alongside pigment degradation that causes hair colour loss, commonly referred to as photoaging. Protein degradation occurs within the UV range of 254–400 nm.

After 600 h of UV exposure, Richena and Rezende [2, 15] used Small Angle X-ray Scattering and Transmission Electron Microscopy to observe that the most affected hair structures were the cuticular components: the endocuticle and the cellular membrane complex (CMC).

Differential scanning calorimetry (DSC) is often employed to measure the energy required to denature alpha-keratin, a critical component of the hair protein matrix [19]. This technique tracks the endothermic denaturation of alpha helices, which form the cortical network of intermediate filaments embedded within an amorphous keratin-associated protein matrix [20]. From the DSC curve, it is possible to calculate the enthalpy related to water loss (up to 180°C), the thermal event enthalpy between 230°C and 250°C (denaturation) and the peak denaturation temperature [19–24].

Fourier transform infrared spectroscopy with attenuated total reflectance (FTIR-ATR) enables the analysis of the hair's external surface—specifically the cuticular layers—by detecting molecular vibrations of chemical compounds at specific infrared spectral regions [25–27]. The literature showed that the cuticle predominantly consists of proteins arranged in alpha conformation that can be temporarily converted to beta-sheet after thermal or chemical damages. This spectroscopic method, with its shallow penetration depth, allows for surface-level analysis of the hair material [26, 27].

According to Fedorkova et al. [28], UV radiation leads to the formation of thiols in hair proteins. Their study, which combined various spectroscopic techniques and chromatography, revealed a decrease in cystine-related cross-links and an increase in soluble proteins within the hair fibre.

In a 2014 study, Fedorkova et al. [29] further concluded that UV exposure accelerated the degradation of lipids and proteins, as demonstrated by soluble protein assays and chromatographic and spectroscopic analyses.

Hair hydrophobicity, essential for minimizing friction and maintaining fibre alignment [7, 30], serves as a crucial indicator of damage. Both chemical treatments and photodegradation remove 18-MEA and degrade the epicuticle [7, 15, 30]. This loss increases hair hydrophilicity, leading to higher friction, roughness and disorder, which negatively impact sensory perception [7, 30]. Hydrophobicity is commonly measured via contact angle to quantify surface damage and structural integrity [7, 30].

Therefore, this study uniquely aims to comprehensively and directly compare, through a multi-technique physicochemical and mechanical characterization, the distinct damage profiles sustained by hair fibres under simulated prolonged solar UV exposure (mimicking intense tropical conditions) versus chemical bleaching, thereby offering original insights into their relative impacts and underlying mechanisms crucial for advancing targeted hair care formulations.

MATERIALS AND METHODS

Material

Tresses of natural Caucasian hair weighing 5.0 g and measuring 25 cm in length were subjected to a standardized pre-cleaning process using a solution of 10% Sodium Lauryl Ether Sulfate (SLES) for 1 min, followed by rinsing in running water. The tresses were maintained in a standardized environment at 55% ± 5% of relative humidity and a temperature of 22.0°C ± 2.0°C during all experiments.

Methods

Bleaching and double bleaching of hair

Dark brown natural Caucasian tresses (in triplicate) underwent the bleaching process using a mixture with a ratio of 1:2 (Cless Lightner Ametista® bleaching powder—ammonium persulfate/potassium persulfate: 30 volume hydrogen peroxide Cless Lightner®) and 5 g of mixture per 1 g of hair. The tresses were kept at a temperature of 36.0°C in an oven (Quimis Q-316 M4®) for 35 min. After the chemical process, the tresses were rinsed for 2 min in running water (25°C ± 2°C, 4L/min) and subjected to a wash with a SLES 10% dispersion and dried in a standardized environment (22°C ± 2°C, 55 ± 5 R.H) for 24 h. Figure 1 shows the baseline condition of hair tresses (natural, bleached and double bleached).

Another three dark brown natural Caucasian tresses were subjected to double bleaching damage using the bleaching process described in the previous paragraph, twice consecutively, according to the experimental flowchart described in Figure 2.

Exposure to UV radiation

The exposure to violet radiation used to mimic the Brazilian summer (approximately 3 months of intense sunlight) was carried out considering 8 h of daily sunlight and therefore 768 h of UV exposure.

The equipment was set to deliver a constant irradiance of 0.35 W·m^{-2} at 340 nm, a parameter widely employed in accelerated photoaging protocols to simulate the UV fraction that reaches the Earth's surface. The equivalence between artificial exposure time and natural sunlight exposure time was established based on the concept of radiant dose, defined as the product of spectral irradiance and exposure duration.

FIGURE 1 Baseline condition of hair tresses (natural, bleached and double bleached).

The dose received under each condition was calculated multiplying the irradiance (W·m^{-2}) and the exposure time (h). Equivalence is achieved when the radiant doses of equipment and natural sunlight are equal. Considering the information content on Table 1, and assuming an average solar irradiance of 0.0875 W·m^{-2} at 340 nm—compatible with moderate natural sunlight conditions, it is possible to conclude that 1 h of exposure in the Q-Sun Xe-1-BC chamber at 0.35 W·m^{-2} (340 nm) corresponds to approximately 4 h of natural sunlight exposure, considering average solar irradiance at this wavelength.

This approach enables a direct comparison between photo-induced degradation processes generated under controlled artificial conditions and those occurring outdoors, ensuring both experimental reproducibility and environmental relevance.

Considering that 1 h in the simulator corresponds to 4 h of exposure to sunlight, 192 h of exposure was carried out.

Dark brown natural Caucasian and bleached Caucasian hair tresses (in triplicate) were exposed to the UV solar radiation simulator Q-Sun Xenon Test Chambers®, model Xe-1-BC equipped with xenon lamps. On the chamber, light exposure is considered homogeneous (according to equipment specifications). The equipment settings are shown in the Table 1.

After exposure to UV radiation, the tresses were washed with a SLES 10% p/p dispersion and dried in a

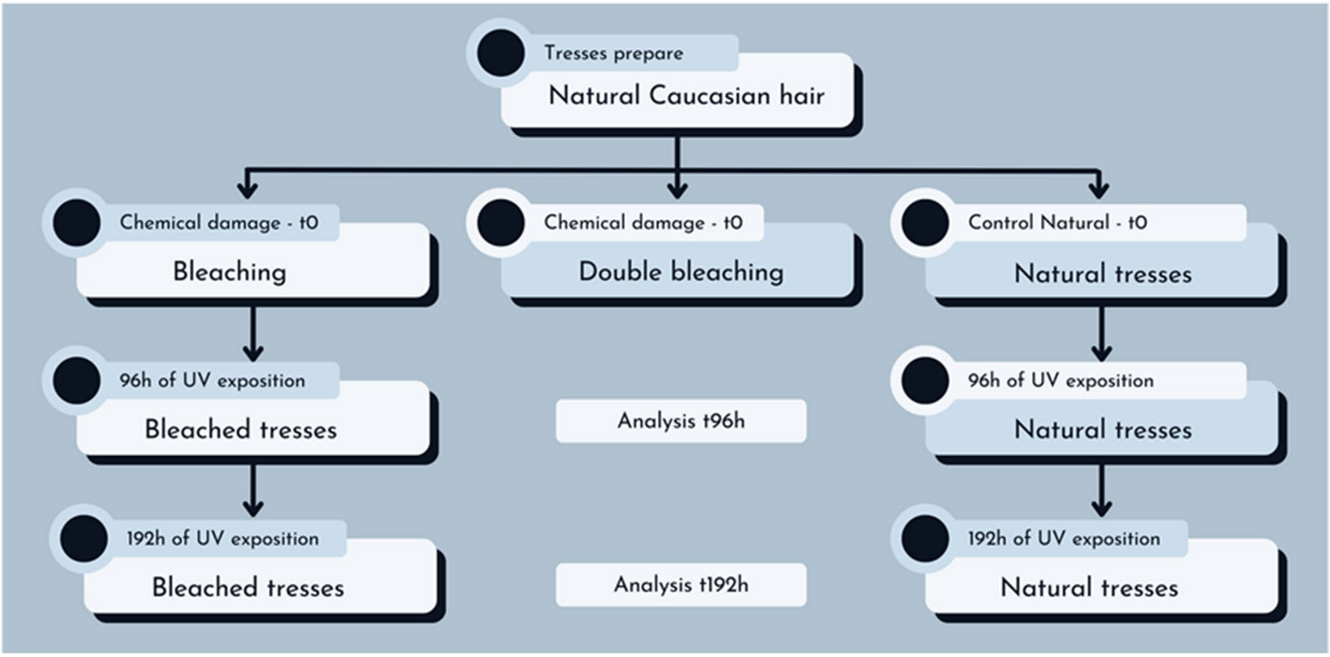

FIGURE 2 Experimental flowchart for tresses preparation and ultraviolet exposure.

TABLE 1 Q-Sun Xenon Test Chambers® equipment settings.

Instrument	Q-Sun Xenon Test Chambers®, model Xe −1-BC
Radiation source	1800 W air-cooled xenon lamp, with daylight filter.
Ageing cycle	Sunstroke only, no exposure to rain.
Control	0.35 W/m^2 irradiance on 340 nm[a].
Humidity	55% ± 5% U.R.

[a]Equivalence: 1 h in the simulator = 4 h of exposure to sunlight.

standardized environment for 24 h. The hair was evaluated in the initial condition (before exposure to radiation), after 96 and 192 h of exposure to UV radiation (Figure 2).

The double bleached tresses were not exposed to UV, due to the study objective being the comparison between oxidation damage extension (of double bleaching) in relation to the UV radiation damage caused on natural and single bleached tresses.

DSC

The TG/DSC measurements were made using the equipment Hitachi High-Tech Sciences TA7000 Series Simultaneous Thermogravimetric Analyser, STA7200®.

The analyses were performed in triplicate with approximately 10 mg of chopped hair. The flow rate used was 100 mL/min of nitrogen gas with a heating rate of 10°C/min (from 30°C to 300°C).

FTIR-ATR

The FTIR-ATR measurements were performed in triplicate using an infrared spectrophotometer (Frontier Model – PerkinElmer®), with an ATR cell (PIKE Technologies) and ZnSe crystal.

The spectrum region from 1800 to 950 cm^{-1} was used, as this is where the main bands of hair fibres are found, such as the characteristic band at 1040 cm^{-1}, referring to the asymmetric stretching vibration of the S═O bond, of the groups $—SO_3^-$ and $—S—O_2$, which are components of cystine oxidation [7]. The spectra obtained were normalized using the absorbance at 1650 cm^{-1}, referring to the primary amide C═O bond.

Hydrophobicity assessment

Hydrophobicity was assessed by pipetting a 20 μL drop of water onto each strand of hair aligned and fixed on a support. The drop was filmed by a digital camera, using a 60 mm macro lens, from its positioning until its absorption.

Two parameters were assessed (triplicate): the contact angle of the water drop in relation to the hair, and the time until it was completely absorbed. The greater the contact angle and the longer the absorption time, the greater the hydrophobicity of the hair.

Mechanical properties

Fifty strands that were randomly collected from the tresses were evaluated to each study group (diameter range 55–65 μm). The EMIC® model DL500 equipped with a dynamometer with a 20 N load cell was used for this test.

The load cell was preloaded, and the increase in load was measured using traction speed rates of 100 mm/min. Each tress was held by a lower gripper and an upper gripper connected to the load cell of a dynamometer at the top. The following parameters were evaluated: deformation at break and force obtained in the region of specific deformation of 20%.

All the experiments were performed in a standardized environment at 22°C ± 2°C and 55% ± 5% of relative humidity.

Statistical analysis

The data obtained was statistically compared using GraphPad Prism® software version 8.4.3. The analysis methods used for statistical comparisons were: Student's *t*-test, one-way analysis of variance with Dunnett's post-test, considering a 95% confidence interval, to check whether there was a significant difference between the samples analysed ($p \leq 0.05$).

RESULTS AND DISCUSSION

Assessment of cuticular damage via DSC, FTIR-ATR and hydrophobicity

From the DSC curve obtained, it is possible to calculate the enthalpy of water vaporization ($\Delta H_{\text{vaporization}}$), which corresponds to the initial thermal event related to water loss up to 180°C. This energy reflects the release of water molecules bound by hydrogen bonds primarily in the cuticle, and to some extent within the cortical core of the hair fibre [19, 22–24].

Lima et al. [19] described the combined use of several analytical techniques, including DSC and FTIR-ATR, to observe cuticular and cortical changes in hair fibres caused

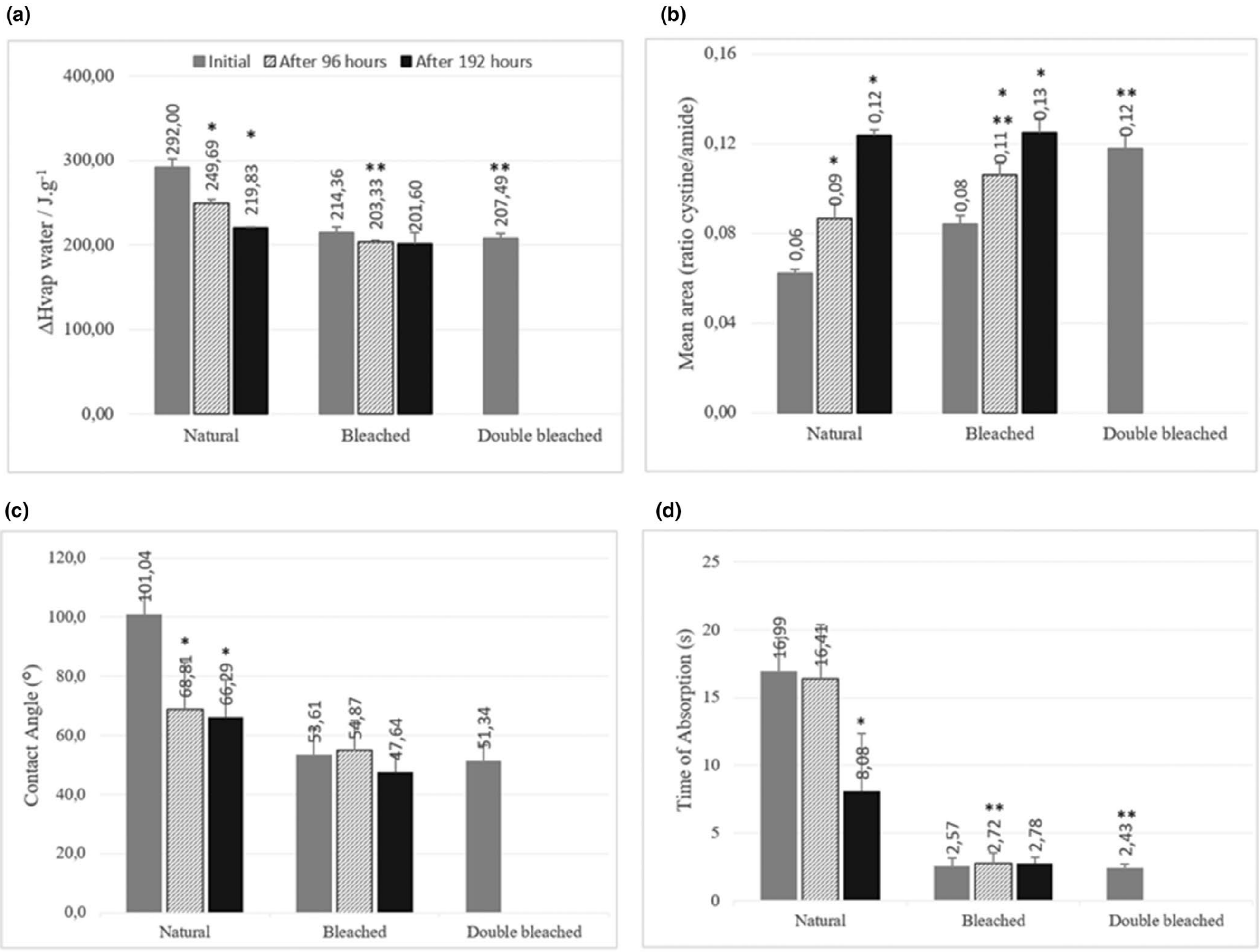

FIGURE 3 Data on (a) enthalpy of vaporization of water (J.g^{-1}), (b) ratio between the average area of the band related to cystine oxidation and the average area of the band related to primary amide; (c) contact angle (°), and (d) surface absorption time (s) of the Natural tresses and the ones subjected to bleaching and double bleaching damage at the Initial evaluation times (tresses without UV exposure), after 96 and 192 h of ultraviolet exposure. *$p < 0.05$ in comparison to respective Initial condition; **$p < 0.05$ in comparison to Natural tresses, mean + standard deviation.

by thermal treatments such as blow-drying and straightening. In their study, the integration of DSC (measuring water vaporization enthalpy), FTIR-ATR (detecting oxidation of cysteine residues) and hydrophobicity assessments provided insight into the cuticular damage observed in natural, bleached and double-bleached hair, as well as damage induced by UV radiation (96 and 192 h exposure).

Data presented in Figures 3 and 4, along with statistical analysis in Table 2, show that the water vaporization enthalpy in natural hair tresses is significantly higher than in bleached and double-bleached samples. This difference is attributed to the oxidation of melanin and cystine during bleaching, which produces cysteic acid and free radicals. Such oxidative damage breaks cross-links within the keratin crystal network, facilitating significant loss of internal water molecules—especially those weakly bound near the hair fibre's surface [4, 20].

No significant difference was found between the water vaporization enthalpy values for hair damaged by a single bleaching procedure versus double bleaching. Despite the expectation of greater structural degradation with repeated bleaching, this parameter remained unaffected, suggesting that the additional chemical damage did not measurably alter the energy required for water evaporation from the fibre.

Infrared spectroscopy results were consistent with the thermal analysis findings. Natural hair displayed a markedly lower ratio of cysteic acid-related bands to amide I–III signals compared with both bleached and double-bleached samples. This shift reflects the progressive oxidation of cystine residues induced by bleaching, leading to the conversion of disulfide bonds into cysteic acid. The increased intensity of the characteristic cysteic acid band at approximately 1040 cm^{-1} therefore serves as a direct

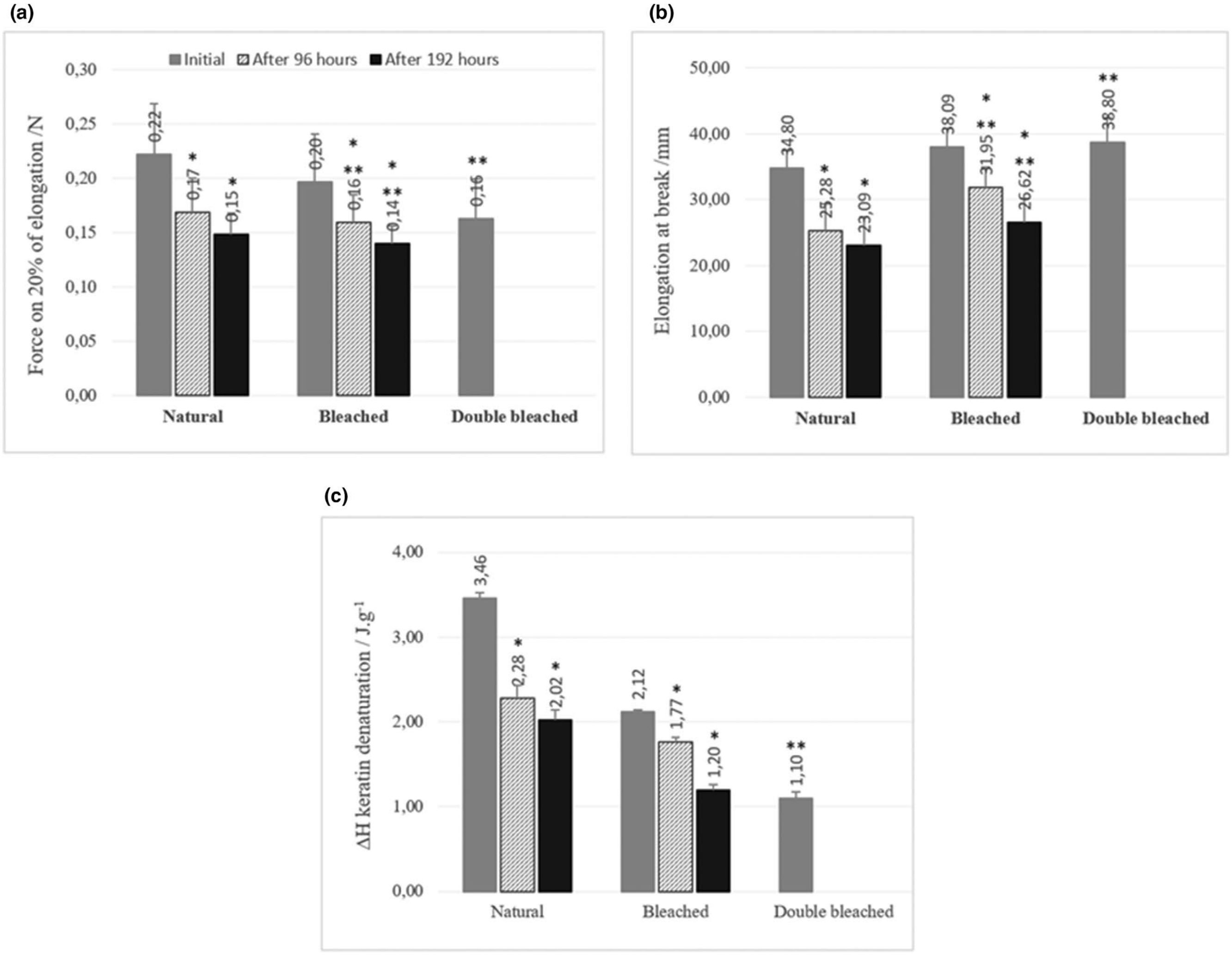

FIGURE 4 Data on (a) Force at 20% of elongation (N); (b) elongation at break (mm); (c) Enthalpy of keratin denaturation ($J.g^{-1}$), initial, after 96 h and 192 h of ultraviolet exposure. *$p < 0.05$ in comparison to respective Initial condition; **$p < 0.05$ in comparison to Natural tresses, mean + standard deviation.

TABLE 2 Comparison between initial condition (t0) of groups.

Comparison	Showed significant differences?			
	$\Delta H_{vaporization}$	FTIR-ATR	Contact angle	Time to absorb
Natural vs. Bleached	Yes N > B	Yes N < B	Yes N > B	Yes N > B
Natural vs. Double bleached	Yes N > DB	Yes N < DB	Yes N > DB	Yes N > DB
Bleached vs. Double bleached	No	Yes B < DB	No	No

Note: Statistical analysis for 95% confidence interval.

Abbreviations: B, bleached; DB, double bleached; N, natural.

spectral marker of oxidative degradation, confirming that chemical bleaching promotes substantial disruption of the hair's disulfide crosslinks [19, 30].

Measurements of contact angle and water absorption time for natural hair tresses were significantly higher than for bleached and double-bleached hair, indicating a

TABLE 3 Comparison between exposure conditions (Vaporization enthalpy, FTIR-ATR, Contact angle and Time to absorb), by hair type.

		Showed significant differences?			
Hair type	**Comparison**	**$\Delta H_{vaporization}$**	**FTIR-ATR**	**Contact angle**	**Time to absorb**
Natural	Initial vs. 96 h	Yes I > 96 h	Yes I < 96 h	Yes I > 96 h	No
	Initial vs. 192 h	Yes I > 192 h	Yes I < 192 h	Yes I > 192 h	Yes I > 192 h
	96 h vs. 192 h	Yes 96 h > 192 h	Yes 96 h < 192 h	No	No
Bleached	Initial vs. 96 h	No	Yes I < 96 h	No	No
	Initial vs. 192 h	No	Yes I < 192 h	No	No
	96 h vs. 192 h	No	Yes 96 h < 192 h	No	No

Note: Statistical analysis for 95% confidence interval.

Abbreviations: 192 h, 192 h of UV exposition; 96 h, 96 h of UV exposition; I, initial.

reduction in hydrophobicity following bleaching. This decrease is likely due to the removal of lipids from the hair cuticle and surface abrasion caused by oxidative processes [2, 31–33]. Similarly, no significant difference was observed between the contact angle and absorption time values of hair subjected to one versus double bleaching treatment.

After 96 and 192 h of UV exposure (Table 3), the natural hair tresses showed a significant reduction in the enthalpy of water vaporization compared to their initial condition. Additionally, after 192 h of UV exposure, there was a further significant decrease in vaporization enthalpy relative to the 96 h exposure.

Infrared Spectroscopy revealed oxidative damage caused by UV radiation: natural hair tresses exposed to 96 and 192 h of UV showed a significant increase in cysteic acid signal, indicating cysteine oxidation within the hair fibre. Notably, the 192 h exposure caused a significant increase in this oxidation ratio compared to 96 h.

Regarding hydrophobicity, natural tresses exhibited a significant decrease in contact angle after both 96 and 192 h of UV exposure. The water absorption time also significantly decreased after 192 h. These reductions in contact angle and absorption time reflect a loss of hydrophobicity in the hair fibres, which is associated with UV-induced degradation of lipids in the hair cuticle [2, 6, 15].

In contrast, statistical comparisons of bleached hair tresses under initial conditions and after 96 and 192 h of UV exposure showed no significant differences in vaporization enthalpy, contact angle or absorption time. This indicates that the cuticular changes affecting these parameters were not significant in already bleached hair. However, bleached hair tresses exhibited a significant increase in the cysteine oxidation ratio after both 96 and 192 h of UV exposure, with a further increase observed at 192 h compared to 96 h (Table 3).

The pattern observed in the data, supported by statistical analysis, aligns with findings reported in the literature, which describe that exposure of hair to low doses of UV radiation leads to degradation of the lipid layer, cuticular amino acids and fatty acids, as well as the formation of free radicals. These changes negatively affect the fibre's surface properties, including hydrophobicity, shine and roughness [15, 16, 34–37].

Based on this analysis of the damage caused by UV exposure at different durations (96 h and 192 h) in both natural and bleached hair tresses, a sequential statistical analysis was performed to assess the extent to which the chemical damage from bleaching compares to the damage induced by UV radiation.

The enthalpy of water vaporization in natural hair tresses exposed to 96 h of UV radiation is significantly higher than that of bleached and double-bleached tresses. However, after 192 h of exposure, natural hair shows no statistically significant difference compared to bleached hair in its initial condition (Table 4).

After 192 h of UV exposure, natural hair exhibits a significantly higher water vaporization enthalpy than double-bleached hair.

The ratio measuring oxidative damage to cysteine bonds did not differ significantly between natural tresses exposed to 96 h of UV and bleached tresses. However, after 192 h, natural hair displayed a greater degree of oxidative degradation compared to bleached hair. This suggests that extended UV exposure causes more extensive oxidative

TABLE 4 Comparison of bleached and double bleached (t0) tresses to natural hair, by time of exposition.

		Showed significant differences?			
Hair type	**Comparison**	**$\Delta H_{vaporization}$**	**FTIR-ATR**	**Contact angle**	**Time to absorb**
Bleached vs. Natural	96 h	Yes N > B	No	No	Yes N > B
	192 h	No	Yes N < B	No	No
Double bleached (t0) vs. Natural	96 h	Yes N > DB	Yes N < B	No	Yes N > B
	192 h	Yes N > DB	No	No	No

Note: Statistical analysis for 95% confidence interval.

Abbreviations: B, bleached; DB, double bleached; N, natural.

TABLE 5 Comparison between initial condition.

	Showed significant differences?		
Comparison	**Elongation**	**Force**	**$\Delta H_{degradation}$**
Natural vs. Bleached	Yes N < B	Yes N > B	Yes N > B
Natural vs. Double bleached	Yes N < DB	Yes N > DB	Yes N > DB
Bleached vs. Double bleached	No	Yes B > DB	Yes B > DB

Note: Statistical analysis for 95% confidence interval.

Abbreviations: B, bleached; DB, double bleached; N, natural.

damage to the structural bonds in the peripheral cuticular region than chemical bleaching [16].

Regarding hydrophobicity, contact angle measurements of natural hair after 96 and 192 h of UV exposure showed no significant differences compared to bleached and double-bleached hair. After 96 h, natural tresses had significantly longer water absorption times than bleached and double-bleached hair. Yet, after 192 h, absorption times for natural hair were comparable to those of bleached and double-bleached hair.

Therefore, these results statistically confirm that prolonged UV exposure causes lipid damage, leading to a decrease in natural hair's hydrophobicity to an extent comparable to that inflicted by chemical bleaching [15, 16, 34–37], thus validating our hypothesis that UV damage replicates chemical damage in terms of hydrophobicity.

Cortical damage via mechanical properties and DSC

At higher doses of UV exposure, disulfide bridges break down, lipids continue to decompose and melanin lightens. This damage extends to the cortical levels of the hair strand, affecting key properties such as mechanical strength, increasing hair breakage during combing, causing colour loss and converting the crystalline keratin structure into a more amorphous phase [4, 38].

Mechanical resistance measurements help us understand the extent of UV-induced damage to hair. Higher strength values at a given deformation indicate better preservation of the internal fibre arrangement (molecular bonds and interactions) [35, 38]. Meanwhile, an increase in deformation at break reflects greater hair plasticity, a hydration-dependent characteristic that allows the fibre to deform more [38].

Using the DSC curve, cortical damage can be assessed by calculating the enthalpy related to the thermal event between 230°C and 250°C—an endothermic process corresponding to the denaturation of the interconnected keratin network that forms the fibre's core structure [19–24].

Comparing initial conditions (Table 5) across hair types, natural hair shows significantly higher strength at 20% deformation and greater alpha-keratin degradation enthalpy than hair damaged by one or two bleaching treatments (Figures 3 and 4).

TABLE 6 Comparison between exposure conditions (Elongation, Force and Enthalpy degradation), by hair type.

		Showed significant differences?		
Hair type	**Comparison**	**Elongation**	**Force**	**$\Delta H_{\text{degradation}}$**
Natural	Initial vs. 96 h	Yes I > 96 h	Yes I > 96 h	Yes I > 96 h
	Initial vs. 192 h	Yes I > 192 h	Yes I > 192 h	Yes I > 192 h
	96 h vs. 192 h	Yes 96 h > 192 h	Yes 96 h > 192 h	No
Bleached	Initial vs. 96 h	Yes I > 96 h	Yes I > 96 h	Yes I > 96 h
	Initial vs. 192 h	Yes I > 192 h	Yes I > 192 h	Yes I > 192 h
	96 h vs. 192 h	Yes 96 h > 192 h	Yes 96 h > 192 h	Yes 96 h > 192 h

Note: Statistical analysis for 95% confidence interval.

Abbreviations: 192 h, 192 h of UV exposition; 96 h, 96 h of UV exposition; I, initial.

TABLE 7 Comparison to natural condition, by time of exposure.

		Showed significant differences?		
Hair type	**Comparison**	**Elongation**	**Force**	**$\Delta H_{\text{degradation}}$**
Bleached vs. Natural	96 h	Yes N < B	Yes N > B	No
	192 h	Yes N < B	Yes N > B	No
Double bleached (t0) vs. Natural	96 h	Yes N < DB	No	Yes N > DB
	192 h	Yes N < DB	Yes N < DB	Yes N > DB

Note: Statistical analysis for 95% confidence interval.

Abbreviations: B, bleached; DB, double bleached; N, natural.

Bleached and double-bleached hair do not differ statistically from each other overall. However, in the initial condition, bleached hair exhibits higher strength at 20% deformation and alpha-keratin degradation enthalpy than double-bleached hair. The reduced performance of double-bleached hair arises from the intensified oxidative cleavage of disulfide crosslinks and other cortical support bonds during the second bleaching cycle, leading to a more pronounced breakdown of the keratin macro-fibrillar network and, consequently, diminished mechanical resilience [1].

After 96 and 192 h of UV exposure, the natural tresses showed a significant decrease in strength at 20% deformation, deformation at break and alpha-keratin degradation enthalpy, indicating that the fibre's cortical core is substantially affected by UV radiation [19, 35, 38]. A similar reduction in these parameters was also observed in bleached tresses after 96 and 192 h of UV exposure (Table 6).

After 96 and 192 h of UV exposure, natural tresses showed a significant decrease in strength at 20% deformation and at break compared to the initial conditions of bleached and double-bleached hair. However, natural hair exhibited significantly higher alpha-keratin degradation enthalpy than double-bleached hair. This suggests that the chemical damage from bleaching is comparable to the damage caused by up to 192 h of UV radiation exposure. Meanwhile, double bleaching causes significantly greater degradation of structural proteins than UV exposure alone [17, 35, 39] (Table 7 and Figure 4).

CONCLUSION

This multi-technique investigation provides novel insights into distinct physicochemical and mechanical damage profiles from prolonged simulated tropical UV exposure versus chemical bleaching. Our findings demonstrate UV radiation causes cuticular damage (cysteine oxidation, significant hydrophobicity reduction) comparable to bleaching. This validates our hypothesis: long-term UV exposure replicates chemical aggression's effect on hair hydrophobicity.

Importantly, this study reveals a nuanced interplay of damage mechanisms. While enthalpy data showed comparable overall protein degradation between 192 h UV and single bleaching, UV radiation demonstrated a significantly stronger impact on cortical integrity. After 192 h UV, natural tresses experienced a more pronounced decline in mechanical properties (e.g., strength at 20% deformation, deformation at break) than bleached or double-bleached hair. Conversely, double bleaching led to more severe structural protein degradation (enthalpy data) than UV alone.

These comparative distinctions are important for understanding hair's vulnerabilities to environmental versus chemical stressors. Our work quantifies these distinct damage pathways, laying essential groundwork for highly targeted, effective hair care formulations, mitigating specific impacts from UV (especially in high-exposure climates) and chemical treatments.

AUTHOR CONTRIBUTIONS

Nathana Cindy Barros Silva Ramos: conceptualization, methodology, investigation, data acquisition and evaluation (statistical analysis, graphs plot), writing of original draft. **Alzira Xavier Pinto Dini**: methodology, data acquisition and evaluation (statistical analysis), data curation, formal analysis, writing of original draft. **Viviane Cristina Albarici**: data curation and evaluation (statistical analysis), formal analysis, writing of original draft. **Mayara Miranda Ferreira**: data acquisition and evaluation (statistical analysis), formal analysis, writing of original draft. **Caroline Vieira Hercolino**: data acquisition and evaluation (statistical analysis), formal analysis. **Adriano da Silva Pinheiro**: supervision, project administration, funding acquisition. **André Rolim Baby**: writing – review and editing. **Maria Valéria Robles Velasco**: writing – review and editing. All authors have read and agreed to the final version of the manuscript.

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CONFLICT OF INTEREST STATEMENT

The authors declare that there is no conflict of interest regarding the publication of this paper.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

ETHICS STATEMENT

This study did not involve human participants, human data or human tissue. Therefore, ethics approval and informed consent were not required.

INFORMED CONSENT STATEMENT

Informed consent was not required as the study did not involve human participants, human data or human tissue.

ORCID

Nathana Cindy Barros Silva Ramos https://orcid.org/0009-0000-4459-9922
André Rolim Baby https://orcid.org/0000-0001-9197-3024

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