Eco-Friendly Evolution: Natural Zeolite as Sustainable Substitute to Traditional Chelating Agents in Hair Bleaching

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Abstract

Bleaching melanin is crucial for achieving lighter shades in hair coloring, but complications arise from metal ions impurities. These can heavily interfere with the bleaching process, catalyzing undesirable reactions, leading to unwanted color shifts, hydrogen peroxide decomposition, and formation of reactive oxygen species that damage hair fibers. In order to reduce transition metal, chelating agents are often used. Unfortunately, most chelants lack biodegradability, facing ecological criticism. The current research aims to replace traditional chelating agents with natural zeolite, providing an eco-friendly and bio-compatible alternative for metal chelation. Additionally, this study explores the optimization of zeolite concentration and possible synergies among zeolite and other ingredients.

Keywords: natural zeolite, hair bleaching, metal chelation, sustainable agent, hair care.

Introduction

Bleach is a common cosmetic treatment utilized to lighten the color of the hair [1]. During the process of bleaching, the hair is lightened by destroying the melanin pigment's chromophore, which is the actual group responsible for the coloring of the hair. Bleach is typically at a pH of between 9 to 11, as the alkaline pH softens the cuticle, allowing for access into the cortex. This is typically accomplished by a powder mixture of ammonia or a type of metasilicate, as well as persulfate salts. Bleach powder is activated by mixing it with hydrogen peroxide, which is a powerful oxidising agent. Once activated, the mixture releases oxygen molecules that break down the melanin pigments in the hair [2]. Bleaching has also been found to alter the amino acid residues in hair, including decreases in levels of cysteine, tyrosine, histidine, threonine, methionine, and lysine [3]. Despite these consequences, the largest complication of this process is due to the presence of metal ions impurities, which leads to the decomposition of hydrogen peroxide during bleaching. This decomposition results in the production of reactive oxygen species (ROS), such as hydroxyl radicals ($HO\cdot$), perhydroxyl radicals ($HO_2\cdot$), and superoxide anions ($O_2^{-}\cdot$). Therefore, they can react with lipids and proteins in the hair. Hydroxyl radical ($\cdot OH$) is highly reactive towards the organic matrix such as hair proteins, leading to significant hair damage; however, hydroperoxyl/superoxide anions radicals ($\cdot OOH/O_2^{-}\cdot$) were reported to display lower reactivity towards hair proteins [4], [5], [6]. These can further cause protein degradation leading to loss of hair fiber strength and consequent hair breakage.

In order to reduce the formation of reactive oxygen species (ROS) during the bleaching process, chelating agents are often used with the aim to chelate metals to stabilize hydrogen peroxide and prevent damage to hair fibers. The main criteria for choosing a particular chelating agent in a hair bleaching system are based on regulatory concerns, commercial availability, biodegradability, binding strength with the metal, and stability of the metal-chelating agent complex under the reaction conditions. Nowadays, there are, however, many discussions concerning its poor biodegradability. Additionally, existing chelating agents are criticized from an ecological standpoint.

The current research aims to replace traditional chelating agents with natural zeolite, providing an eco-friendly and bio-compatible alternative for metal chelation. Furthermore, this study explores the optimization of zeolite concentration in the bleaching process and possible synergies among zeolite and experimenting with two new ingredients such as L-Lysine and Rice Starch for improving sensoriality.

Zeolites represent a family of hundreds of microporous mineral members known for their ion-exchange properties. The term “zeolite” coined, in 1756 by the Baron Axel Fredrik Cronstedt, a Swedish mineralogist and chemist, derives from the Greek language ζέω (“zeo”, to boil) and λίθος (“lithos”, stone) meaning “boiling stone”. This name means “the stone that boils” because when it is heated, it releases water without changing the structure, resembling a bubble forming [7], [8].

Zeolite is a mineral of volcanic origin, characterized by a regular and microporous crystal structure, which contains a significant amount of void volumes inside the crystals. Chemically, it is a hydrated aluminosilicate with a three-dimensional framework consisting of AlO_4 and SiO_4 tetrahedra. These tetrahedra are interconnected in various regular arrangements through shared oxygen atoms, forming an open crystal lattice consisting of defined channels and cages [8], [9].

Zeolites have pores of uniform size (ranging from 3Å to 10Å), which are uniquely determined by the unit structure of the crystal. They contain water molecules and cations (such as Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}) within the structure. Additionally, these pores selectively exclude molecules larger than their diameter. The aluminia atoms in zeolites impart ion exchange capacity towards cations, while the defined pores contribute to the zeolite's selectivity towards different ions during ion exchange processes [9], [10], [11].

There are several synthetic or naturally occurring species of zeolites; the most widespread and studied are naturally occurring zeolites [12]. They have excellent detoxifying, antioxidant and anti-inflammatory agents. As a result, they have been used in many industrial applications ranging from environmental remediation to oral applications/supplementation in vivo in humans as food supplements or medical devices [13].

Materials and Methods.

Here is a brief description of raw materials and bleaching powders used during our research.

- *Bleaching Powders:* During this study, four different hair bleaching powders were used, each containing a high concentration of peroxide and silicate, responsible for the bleaching process. These bleaching powders have several characteristics: they can achieve a bleaching level of up to 8 tones, come in compact powder form, and are formulated with either no ammonia or low ammonia concentration. Additionally, they are available in different colors and fragrances. Furthermore, these bleaching powders offer numerous benefits: they allow for easy and fast application and deliver excellent bleaching outcomes.
- *Hydrogen peroxide:* Commercial oxidizing emulsions containing 6%, 9%, and 12% of hydrogen peroxide (20, 30, 40 volumes) were used during the tests. The volumes of the oxidant were chosen based on the level of tones to lighten.
- *Chelating agent:* Natural zeolite, utilized in extra fine powder form without additives, preservatives, or other substances, undergoes extraction, crushing, and multiple sieving processes to achieve a dense and homogeneous powder. Renowned for its multifaceted

properties, it acts as a potent chelating agent. Its molecular sieve characteristics, absorbent nature, and high cation exchange capacity enable it to effectively reduce free radicals and draw out impurities, contributing to beneficial properties for the hair. Additionally, it aids in cell regeneration, counters inflammation, boosts the immune system, and provides antioxidant and immunostimulant effects.

- *Active ingredients: Rice Starch* is a starch derived from rice (Oryza sativa), a plant belonging to the Gramineae family. This starch is extracted from rice grains. Rice starch is primarily composed of carbohydrates, with amylose and amylopectin being the main polysaccharides present. Alongside carbohydrates, it may contain small amounts of proteins, lipids, minerals such as potassium and magnesium, and traces of other minerals found in rice. The composition can vary based on factors like rice variety and processing methods. Additionally, rice starch may contain some moisture and ash residue [13]. Rice Starch is used in a variety of industries, including food, pharmaceuticals, cosmetics, and textiles, for its properties of gelling, thickening, and stabilizing. In cosmetics, for example, the use of rice starch powder, which is a simple and inexpensive compound, can improve the barrier function of damaged skin [14].

- *L-Lysine* is an essential amino acid crucial for protein synthesis and collagen formation. It plays a vital role in maintaining healthy hair growth and strength. L-Lysine contains various functional groups including α-amino, ω-amino, and α-carboxyl groups, exhibiting high reactivity with the protein structure of hair fibers, showcasing its beneficial properties [15]. Widely utilized in cosmetics, food, and pharmaceutical industries, L-Lysine serves as a versatile ingredient with numerous applications [16].

- *Hair tresses:* Hair tressers without cosmetic hair treatment were used in this study. Dark brown hair tresses with a total length 6 cm and 0,3 g weight were utilized.

Bleaching process: In this study, four hair bleaching powders, high in peroxides, were diluted with 6%, 9%, or 12% hydrogen peroxide at different ratios (1:1 or 1:2) until obtaining creamy consistency. Dark brown hair tresses were treated for 45’ and 90’ at room temperature and 30°C. Tresses were then rinsed under warm water and dried with a hair dryer. All blends were compared with powders containing traditional chelating agents.

This study included three phases: i) applying bleaching powders with zeolite (1%, 2%), ii) experimenting new actives (1% L-lysine, 3% Rice Starch) for improving sensoriality, and iii) testing several application parameters like temperature, dilution, times and hydrogen peroxide volume.

i) Applying bleaching powders with zeolite (1%, 2%)

Two types of bleaching powder, each containing the traditional chelating agent, were used in the bleaching tests on the hair tresses. Each bleaching powder was diluted with 12% (40 volumes) of Hydrogen Peroxide (developer) according to the following ratio and parameters:

- *Mixing*: One part of powder was mixed with two parts of the developer until a creamy consistency was obtained, and then applied to the hair for several minutes.
- *Application*: The tests were conducted on dark brown hair.
- *Time*: The application on the hair was maintained for 45 minutes.
- *Temperature*: The tests were performed at 30°C.
- *Rinse and drying*: The samples were rinsed with warm water, and drying was accomplished using a hairdryer.

Following this method, additional tests were conducted to evaluate the efficacy of zeolite in the hair bleaching process. The hair tresses were treated with bleaching powders containing

1% zeolite in bleaching formulations in the first round of tests, and then the tests were repeated with 2% zeolite. Each formulation was compared with the standard formulations containing traditional chelating agents.

ii) Experimenting new actives (1% L-lysine, 3% Rice Starch) for improving sensoriality

During the hair bleaching process, bleaching powders formulated with ammonium persulphate and potassium persulphate produced satisfactory results but resulted in increased hair porosity. Considering these factors, research was conducted to identify new actives aimed at improving the sensorial properties of the hair. The identified actives include L-Lysine and Rice Starch.

Subsequently, tests were conducted using each active ingredient at various percentages (1% L-Lysine, 3% Rice Starch) in bleaching powders to evaluate if these actives modified the bleaching hair level. Initially, the actives were evaluated in bleaching powders without zeolite and then compared with those containing 1% zeolite.

<u>Method for tests without Zeolite:</u> *Mixing:* One part of bleaching formulation (bleaching powder + 1% L-Lysine or 3% Rice Starch) was mixed with two parts of the developer, hydrogen peroxide 40 volumes. *Application:* The tests were conducted on dark brown hair. *Time*: The mixture was applied to the hair and left on for 45 minutes. *Temperature*: The tests were performed at 30°C. *Rinse and drying*: The samples were rinsed with warm water, and dried using a hairdryer.

<u>Method for tests with 1% Zeolite:</u> *Mixing:* One part of bleaching formulation (bleaching powder + 1% L-Lysine or 3% Rice Starch + 1% Zeolite) was mixed with two parts of the developer, hydrogen peroxide 40 volumes. *Application:* The tests were conducted on dark

brown hair. *Time*: The mixture was applied to the hair and left on for 45 minutes. *Temperature*: The tests were performed at 30°C. *Rinse and drying*: The samples were rinsed with warm water, and dried using a hairdryer.

iii) Testing several application parameters like temperature, dilution, times and hydrogen peroxide volume.

All the tests described above, utilizing bleaching powder with 1% L-Lysine or 3% Rice Starch containing 1% Zeolite, were conducted while varying different parameters such as temperature, time, volume of hydrogen peroxide, and dilution. Here are the tests performed:

- Two tests were conducted for each active ingredient, changing the temperature from 30°C to room temperature. The test results were compared with each other.
- Two tests were conducted for each active ingredient, altering the developing time from 45 minutes to 90 minutes. The test results were compared with each other.
- Two tests were conducted for each active ingredient, changing the volume of hydrogen peroxide: hydrogen peroxide at 20 volumes and 30 volumes were used. The test results were compared with each other.
- Two tests were conducted for each active ingredient with a different dilution ratio, ranging from 1:2 to 1:1. The test results were compared with each other.

Results

i) Applying bleaching powders with zeolite (1%, 2%)

The study indicates that, in all trials, 1% zeolite enhances bleaching more effectively than 2%, when compared with powders enriched with traditional chelating agents. This performance is attributed to its microporous aluminosilicate structure, which allows ions absorption and exchange.

Bleaching powder A + Zeolite (1%, 2%)	Bleaching powder B + Zeolite (1%, 2%)
Parameters for fig a) dil.1:2, 40 vol, 30°C, 45'	*Parameters for fig b): dil.1:2, 40 vol, 30°C, 45'*

Fig. 1: Effect of 1% and 2% Zeolite in Different Bleaching Powders. Images show the results of bleaching experiments using two bleaching powders with 1% and 2% zeolite (fig. 1a) (fig 1b). The images illustrate that the 1% zeolite enhances the bleaching process more effectively than the 2% zeolite. Bleaching powders enriched with traditional chelating agents were less effective than those containing zeolite.

ii) Experimenting new actives (1% L-lysine, 3% Rice Starch) for improving sensoriality

Some decrease in the sensorial properties was noticed. Two actives, L-Lysine (texturizing) and Oryza Sativa (Rice) Starch (conditioning, restructuring, protective) were selected to counteract such drawbacks. Tests combining one of these two actives with 1% zeolite in bleaching formulations showed consistent results, enhancing hair softness and brightness, without significant impact on the bleaching effects.

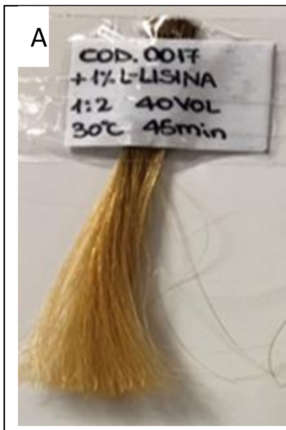
A 	B 	C 	D 
*Bleaching Powder + Active (**1% L-Lisina**)* ***Parameters:** dil.1:2, 40 vol, 30°C, 45'*	*Bleaching powder + Active **(1% L-Lisina)** + 1% Zeolite* ***Parameters:** dil.1:2, 40 vol, 30°C, 45'*	*Bleaching Powder + Active (**3% Rice Starch**)* ***Parameters:** dil.1:2, 40 vol, 30°C, 45'*	*Bleaching Powder + Active (**3% Rice Starch**) + **1% Zeolite*** ***Parameters:** dil.1:2, 40 vol, 30°C, 45'*

Fig. 2. Experimenting new actives (1% L-lysine, 3% Rice Starch) for improving sensoriality – result tests *(a) bleaching powder combined with 1% L-Lysine; (b) bleaching powder combined with 1% L-Lysine containing 1% zeolite; (c) bleaching powder combined with 3% Rice Starch ; (d) bleaching powder combined with 3% Rice Starch) containing 1% zeolite. Tests combining one of these two actives with 1% zeolite in bleaching formulations showed consistent results, enhancing hair softness and brightness, without significantly impacti on the bleaching effects.*

iii) Testing several application parameters like temperature, dilution, times and hydrogen peroxide volume.

Temperature, time, hydrogen peroxide volume, and dilution, as are known, impact the bleaching process. Lowering temperature from 30°C to room conditions reduced the bleaching effect, while increased development times and higher volumes of hydrogen peroxide enhanced it. Dilution at 1:1 ratio also improved bleaching but resulted in a challenging, pasty application.

Developing Time Variation Test: *Bleaching powder + Active (1% L-Lisina) + 1% Zeolite*

Parameters for fig 3a): dil.1:2, 40 vol, **45'**

Parameters for fig 3b): dil.1:2, 40 vol, 30°C, **90'**

Fig. 3. The results of the developing time variation tests conducted for the L-Lysine active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that increasing development time from 45' (fig. 3a) to 90' (fig. 3b) enhanced the bleaching effect.

Hydrogen Peroxide Volume Variation Test: *Bleaching powder + Active (1% L-Lisina) + 1% Zeolite*

Parameters for fig 4a): dil.1:2, **20 vol**, 30°C, 45'

Parameters for fig 4b): dil.1:2, **30 vol**, 30°C, 45'

Fig. 4.: The results of the hydrogen peroxide variation tests conducted for the L-Lysine active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that increasing hydrogen peroxide volume from 20 vol. (fig. 4a) to 30 vol (fig. 4b) enhanced the bleaching effect.

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<td colspan="2">Dilution Ratio Variation Test: Bleaching powder + Active (1% L-Lisina) + 1% Zeolite
Parameters for fig 5a): dil.1:2, 40 vol, 30°C, 45’
Parameters for fig 5b): dil.1:1, 40 vol, 30°C, 45’</td>
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<td colspan="2">Temperature Variation Test: Bleaching powder + Active (1% L-Lisina) + 1% Zeolite
Parameters for fig 6a): dil.1:2, 40 vol, 45’, 30°C
Parameters for fig 6b): dil.1:2, 40 vol, 45’, Room temperature (23°C)</td>
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<td colspan="2">Fig. 5. The results of the dilution ratio variation tests conducted for the L-Lysine active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that lowering the dilution ratio from 1:2 (fig. 4a) to 1:1 (fig. 4b) enhanced the bleaching effect.</td>
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<td colspan="2">Fig.6. The results of the temperature variation tests conducted for the L-Lysine active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that lowering the temperature from 30°C (fig. 6b) to room temperature (fig. 6a) reduced the bleaching effect .</td>
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Developing Time Variation Test: *Bleaching powder + Active (3% Rice Starch) + 1% Zeolite* Parameters for fig 7a): dil.1:2, 40 vol, **45'** Parameters for fig 7b): dil.1:2, 40 vol, 30°C, **90'**		***Hydrogen Peroxide Volume Variation Test:*** *Bleaching powder + Active (3% Rice Starch) + 1% Zeolite* Parameters for fig 8a): dil.1:2, **20 vol**, 30°C, 45' Parameters for fig 8b): dil.1:2, **30 vol**, 30°C, 45'
Fig. 7. The results of the time variation tests conducted for the Rice Starch active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that increasing development time from 45' (fig. 7a) to 90' (fig. 7b) enhanced the bleaching effect.		**Fig. 8. The results of the Hydrogen Peroxide variation tests conducted for the Rice Starch active ingredient on the bleaching formulation with 1% zeolite.** The figures illustrate that increasing hydrogen peroxide volume from 20 vol. (fig. 8a) to 30 vol. (fig. 8b) enhanced the bleaching effect.

Dilution Ratio Variation Test: Bleaching powder + Active (3%Rice Starch) + 1% Zeolite Parameters for fig. 9a): **dil.1:2**, 40 vol, 30°C, 45’ Parameters for fig. 9b): **dil.1:1**, 40 vol, 30°C, 45’		***Temperature Variation Test:*** Bleaching powder + Active (3% Rice Starch) + 1% Zeolite Parameters for fig 10a): dil.1:2, 40 vol, 45’, **30°C** Parameters for fig. 10b): dil.1:2, 40 vol, 45’, **Room temperature (23°C)**
Fig. 9. The results of the dilution ratio variation tests conducted for the Rice Starch active ingredient on the bleaching formulation with 1% zeolite. The figures illustrate that lowering the dilution ratio from 1:2 (fig. 9A) to 1:1 (fig. 9B) enhanced the bleaching effect.		**Fig. 10. The results of the temperature variation tests conducted for the Rice Starch active ingredient on the bleaching formulation with 1% zeolite**. The figures illustrate that lowering the temperature from 30°C (fig. 10b) to room temperature (fig. 10a) reduced the bleaching effect

Discussion.

The study establishes zeolite as a promising and versatile ingredient in hair bleaching formulations, providing an eco-friendly solution for metal chelation. This is attributed to zeolite's ion exchange properties, absorption capacity, and its role in stabilizing formulations. The incorporation in bleaching powders of some active ingredients, such as lysine or rice starch, contributes to sensorial performances of hair fibers, acting as conditioning agents.

During hair bleaching, disulfide bond cleavage under alkaline conditions produces sulfonates, including cysteic acid. This promotes interactions with lysine residues, potentially influencing hair protein structure and enhancing fibers stability. Additionally, rice starch acts as hair conditioner, due to its content of amylose and amylopectin, polysaccharides forming a thin film onto the hair's surface. This film seals the cuticles, reducing their porosity. Future research exploring the synergistic potential of zeolite combined with other actives is pivotal for expanding the landscape of eco-friendly and effective hair care formulations.

Conclusion.

In conclusion, the findings of this study highlight the significant potential of natural zeolite as an eco-friendly alternative for metal chelation in hair bleaching formulations. The use of zeolite at a concentration of 1% proved to be more effective than at 2%, demonstrating its ability to enhance the bleaching process while maintaining consistent results across all trials. This efficacy can be attributed to the microporous aluminosilicate structure of zeolite, which facilitates ion absorption and exchange. Moreover, the incorporation of active ingredients such as L-Lysine and Rice Starch contributed to improved sensorial effects, including enhanced softness and brightness of the hair. However, variations in parameters such as temperature, time, hydrogen peroxide volume, and dilution were found to impact the bleaching process, emphasizing the importance of optimizing these factors. Overall, this research underscores the promising role of zeolite and other active ingredients in developing environmentally sustainable and effective hair care products. Further exploration of synergies between zeolite and additional actives holds considerable potential for advancing the field of eco-friendly hair care formulations.

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Conflict of Interest Statement.

The author declares no conflict of interest.

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