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Formulation of Hair Gels Based on Mango Pectin and Coconut Oil

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Abstract

Alopecia, defined as thinning or loss of hair, is a condition affecting the hair follicle. Traction alopecia, which is traumatic in nature, mainly results from common hairstyling practices among black women. Conventional treatments, such as minoxidil, have many limitations. This study aimed to formulate hair gels based on pectin extracted from mango and coconut oil, in order to offer a natural alternative for the treatment of traction alopecia. The extracted raw materials were characterized. The pectin gel was prepared at 95°C under agitation at 800 rpm, then dispersed in the lipophilic phase under constant agitation at 1500 rpm for 10 minutes to obtain two formulations, B and B'. These homogeneous gels showed instability under centrifugation. Microscopic examination revealed coarse emulsions. Over 28 days, control showed a slight variation in pH, indicating satisfactory microbiological stability. In stability tests at different temperatures, gel B proved stable at 6 ± 2°C, 25 ± 2°C, and 40 ± 2°C, while gel B' remained stable only at cold and room temperatures. In terms of rheology, gel B exhibited shear thinning behavior, thixotropic behavior and viscoelastic properties, unlike gel B', which showed predominantly elastic behavior. These results are part of the development of innovative phytocosmetics for the management of traction alopecia in black women.

Keywords: Non-Scarring Alopecia, Gels, Pectin, Coconut Oil, Shear Thinning.

1. INTRODUCTION

Non-scarring alopecia is the result of a condition that reduces or slows down hair follicle growth. The causes are diverse and multifactorial (De Lorenzi C *et al*., 2018). Non-scarring alopecia includes traction alopecia (De Lorenzi C *et al*., 2018). Traction alopecia is a traumatic, non-scarring form of alopecia. It is characterized by hair loss following tension on the hair shafts. Several treatments exist and are used, including minoxidil, but they are poorly tolerated by patients due to numerous adverse effects (Kirakosyan R, 2018; Rossi A *et al*., 2012). To overcome these deficiencies, a better alternative is needed for the treatment of traction alopecia in black women. Several plants and plant-based formulations are traditionally used throughout the world, particularly in Africa (Bharti M *et al*., 2020). These include the coconut palm, *Cocos nucifera L.* (Arecaceae), whose seed oil is used in tropical countries to beautify long hair and protect it from damage such as brushing, excessive exposure to chemicals or heat (Rele AS *et al*., 2003). In addition, pectin is an antioxidant, biocompatible, biodegradable, and non-toxic polysaccharide found in the cell walls of plants, particularly *Mangifera indica L.* (Anacardiaceae) (Grant GT *et al*., 1973; Colodel C *et al*., 2010; Watts P *et al*., 2009; Ro J *et al*., 2015). Pectin allows hydrogels to be obtained in the presence of sucrose and divalent cations such as calcium (May CD, 1990; Morris ER *et al*., 1982; Chan SY *et al*., 2017), which ensure moisture retention to provide and maintain a moisturizing effect. Gels based on these different natural products could be an alternative in the treatment of traction alopecia. The objective of this study was to formulate hair gels based on mango pectin combined with coconut oil for the treatment of traction alopecia in black women.

2. MATERIALS

2.1 Plant Materials

The plant raw materials consisted of mango pectin, *Mangifera indica L.* (Anarcadiaceae), and coconut oil, *Cococ nucifera L.* (Arecaceae). Mangoes belonging to the Amélie variety were selected based on

their fibrous nature, their availability in Côte d'Ivoire, and their degree of maturity. The ripe mangoes were harvested in Korhogo by an agent from the National Agricultural Research Center (Korhogo branch) between December 2020 and January 2021. An initial identification was carried out on site. A second identification was then carried out at the National Center for Floristics in Abidjan with the herbarium number UCJ000983.

Mango peel waste was used for pectin extraction. Coconut oil was extracted from coconut fruits using a manual cold press.

2.2 Solvents and Reagents

Analytical grade solvents and reagents obtained from Sigma Chemical Co. (Steinheim, Allemagne) consisted of hydrochloric acid (batch no. 16C020516), sodium hydroxide (batch no. 1866402), potassium hydroxide (batch no. 472173), calcium chloride (batch no. 0000456696), citric acid monohydrate (batch no. A0388355), sodium chloride (batch no. 1665066), Potassium iodide (batch no. 472737), Sodium thiosulfate (batch no. 483827) and Phenolphthalein (batch no. F04950500). Osmosis water was used (ThermoFisher Scientific, France).

3. METHODS

3.1 Pectin Extraction

Extraction was performed according to the procedure described by Chaiwarit *et al.*, (Chaiwarit, T *et al*., 2020). 20 g of mango peel powder was added to 600 ml of acidified water. The mixture was heated in a microwave oven at 550 watts for 20 minutes, then filtered. The filtrate was centrifuged at 4800 rpm for 20 minutes; the supernatant was collected, then precipitated with two volumes of 95.5% pure ethanol and stored for one hour to obtain wet pectin; the wet pectin obtained was dried in an oven at 50°C for 48 hours, then ground to obtain a powder. An extraction yield (%) after two tests was calculated.

3.2 Characterization of the Pectin

The characteristics investigated were: organoleptic characteristics, moisture content, and degree of esterification determination.

3.3 Characterization of Coconut Oil

The characterization consisted of performing physical and chemical characterization (organoleptic properties, determination of moisture content, determination of chemical indexes: saponification index, acid index, iodine index, peroxide index).

3.4 Formulation and Characterization of Gels

3.4.1 Formulation

Gels consisted of two phases: a hydrophilic phase (mango pectin gel) and a lipophilic phase (coconut oil). The proportions of coconut oil varied in the different compositions. These compositions listed in Table I.

Table I: Composition of mango pectin and coconut oil gels

Composition (%) Different gels	Pectin gel	Coconut oil	Propylparaben
Gel B	94.8	5	0.2
Gel B’	89.8	10	0.2

The mango pectin gel was gradually introduced into the lipophilic phase under constant stirring at 1500 rpm for 10 minutes at 25°C ± 2°C. The final gels were stored for 48 hours at 4 ± 2°C to eliminate air bubbles before characterization.

3.4.2 *Gels Characterization*

Organoleptic Characterization

Appearance, odor, and color of the gels were evaluated. Appearance was performed by spreading the gel to check for the presence or absence of lumps and air bubbles.

Microscopic Characterization

The analysis consisted of measuring the size of 300 globules at least using an optical microscope equipped with a micrometric scale at 10x magnification.

Physicochemical Characterization

- **pH Determination:** The pH was measured using a HI 2211, electronic pH meter with a glass electrode. After calibration, the electrode was wiped and immersed in a beaker containing a 10% solution of the gel. The test was performed three (03) times.
- **Determination of Emulsion Type:** The type of emulsion was determined using a dyeing method with two dyes: one water soluble (methylene blue) and the other fat-soluble (Sudan III). This involved spreading a small amount of gel on a watch glass and then placing a small amount of a 1% methylene blue solution and a drop of a 1% Sudan III solution on the surface. The type of emulsion was determined based on the affinity of the dyes for the continuous and dispersed phases.
- **Rheological Characterization:** The rheological properties of the gels were evaluated using a Kinesus rotary rheometer (Malvern Instruments, Massy, France) equipped with a plane-plane geometry, 1 mm of gap and a cover to limit sample drying. The Linear Viscoelastic Region (LVR) was determined by oscillatory deformation scanning (1 Hz, 0.01-10 Pa), in accordance with Doucet *et al.* (Doucet *et al*., 2001). Each test was repeated three times on approximately 10 g of gel, and the averages

were analyzed using rSpace software. The measurements focused on the evolution of shear stress as a function of shear rate (0.01-100 s^{-1}) at 25 ± 1 °C and 37 ± 1 °C, allowing the typology and thixotropy of the formulations to be identified according to the Herschel-Bulkley model (Gilbert, 2012): $\tau = K \gamma^n + \tau_0$, where τ is the shear stress (Pa), γ is the shear rate (s^{-1}), n is the flow index, K is the consistency index ($Pa \cdot s^n$), and τ_0 is the critical stress (Pa). The viscoelastic parameters were studied as a function of temperature varying from 5 to 90°C at a frequency of 1 Hz and a strain of 1%. These parameters were also evaluated in oscillatory mode over a frequency range of 0.01 to 100 Hz at low strain (1%) and performed at 25 ± 1°C.

- **28-day Stability Tests:** Stability tests were performed over 28 days (D_0, D_1, D_2, D_3, D_7, D_{14}, D_{21}, D_{28}) on gels previously stored for 24 hours in a refrigerator. Centrifugation and temperature stability tests were performed at 2000 rpm for 10 minutes and at 6 ± 2°C, 25 ± 2°C and 40 ± 2°C, respectively. Color, odor, appearance and pH were noted.

4. RESULTS

4.1 Extraction and Pectin Characterization

4.1.1 Mango Pectin Extraction Yield

The extraction yield containing mango peel pectin (MP) obtained using microwave-assisted extraction was 17.55% ± 4.04%.

4.1.2 Pectin Characterization

After extraction, the mango pectin observed was brownish-green in color, with a faint odor and characteristic taste. The moisture content was 7.10 ± 0.10%. The degree of esterification determined was 38%, less than 50%, indicating that the extracted pectin was low methylated pectin (LMP).

4.2 Coconut oil Characterization

4.2.1 Organoleptic Characterization

Coconut oil was ivory white, viscous, with a characteristic coconut odor.

4.2.2 Physicochemical Characterization of Coconut Oil

The moisture, iodine, peroxide, saponification, and acid values of the coconut oil were determined. The moisture levels were relatively low (Table II). The peroxide value was in line with the Codex Alimentarius standard (less than 15 mEq O2/kg). The acid value was in line with the Codex Alimentarius (less than 4 mg KOH/g). The iodine value was in line with the Codex Alimentarius (6.3-10.6 g I_2 /100 g for coconut oil). The coconut oil had a saponification index lower than that of the Codex Alimentarius (248-265 mg KOH/g). These index values are listed in Table II.

Table II: Physicochemical analysis of coconut oil (n= 3)

Raw materials	Residual moisture (%)	Peroxide value (mEq O_2/ Kg)	Acid value (mg KOH/g)	Iodine value (g d'I_2/ 100 g)	Saponification index (mg KOH/g)
Coconut oil	0.35 ± 0.1	1.00 ± 0.0	1.45 ± 0.1	7.16 ± 0.1	192.6± 2.1

4.3 Formulation and Characterization of Gels

4.3.1 Organoleptic Characterization

The gels were homogeneous, without air bubbles, and had a characteristic coconut odor. The coconut oil and pectin-based gels were dark brown in color (Figure 1).

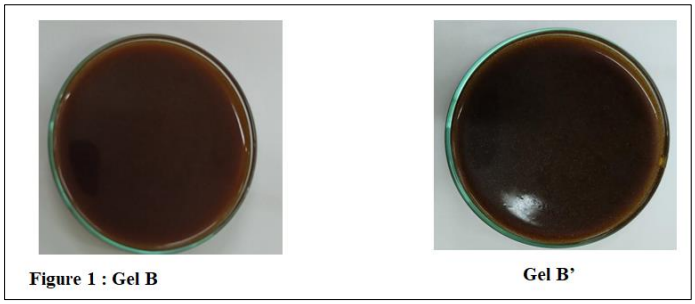

Figure 1: Coconut oil and pectin-based gels B (94.8% pectin gel + 5% coconut oil) and B': (89.8% pectin gel + 10% coconut oil)

4.3.2 Microscopic Characterization

The dispersed globules of coconut oil in gels B and B' are shown at ×10 magnification in Figure 2 below.

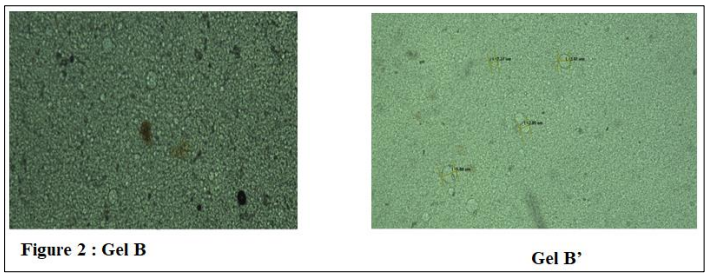

Figure 2: Coconut oil globules in gels B and B' at ×10 magnification

The median diameter of the globules was 1.51 ± 0.60 µm, meaning that gel B corresponded to a coarse emulsion (Le HIR, 2016). The tenth decile (D10) was 0.96 µm and the ninety-first decile (D90) was 2.28 µm, with an interdecile ratio (D90/D10) of 2.38 (Figure 3). For gel B', the median diameter of the globules was 2.24 ± 1.02 µm, confirming that gel B' was a coarse emulsion. The tenth decile (D10) was 1.15 µm and the ninetieth decile (D90) was 3.66 µm, with an interdecile ratio (D90/D10) of 3.18 (Figure 3). The distribution of globules was better in the low-fat gel (B) compared to the high-fat gel (B'). The interdecile ratio of gel B was closer to 1 compared to gel B'.

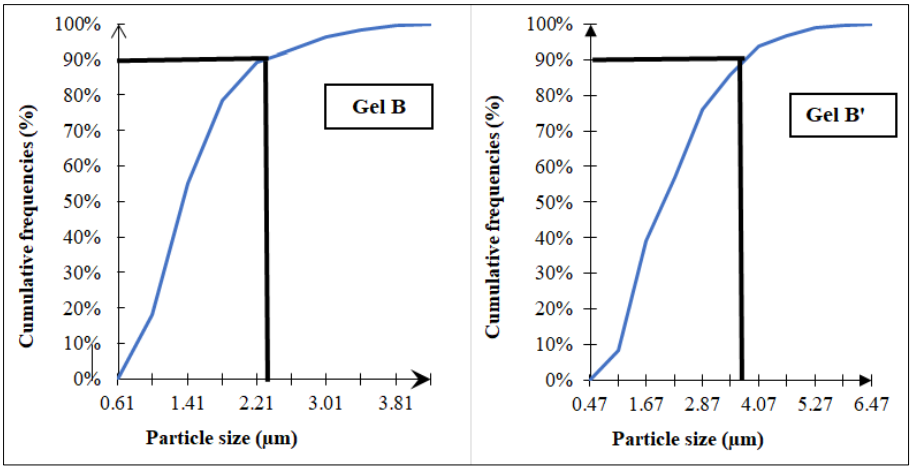

Figure 3: Distribution of cumulative frequencies according to size for gels B: and B'

4.3.3 Physicochemical and Stability Tests

pH and Emulsion Type

The pH of the gels was 4.91±2. These gels were Lipophilic/Hydrophilic (L/H) type.

Rheological Characterization

- **Evaluation of Fluid Typology at Different Temperatures**

❖ **Flow Curves at 25°C:** Gels B and B' exhibited high viscosity at low shear rates. Gels viscosities decreased as a function of shear rate, demonstrating their shear thinning behavior. Indeed, for shear rates ranging from 0.01 to 1 s^{-1}, the viscosity of gels B and B' decreased by 97% (Figure 4).

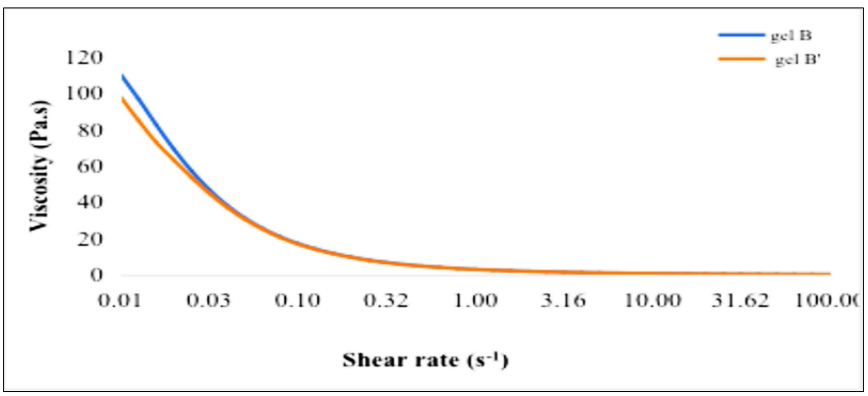

Figure 4: Viscosity of gels as a function of shear rate at 25°C ± 1°C (Gels B and B')

Figure 5 showed 3 phases forming a hysteresis cycle demonstrating thixotropic behavior of the gels at 25°C.

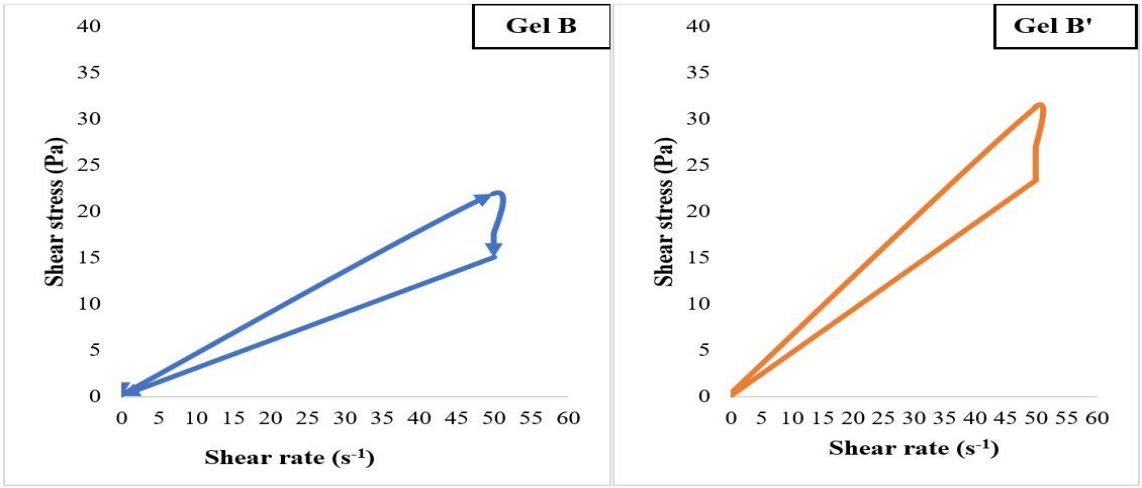

Figure 5: Shear stress as a function of shear rate at 25°C ± 1°C (Gels B and B')

❖ **Flow Curves at 37°C:** Gels viscosities decreased as a function of shear rate, demonstrating their shear thinning behavior. For shear rates ranging from 0.01 to 1 s^{-1}, the viscosity of gels B and B' decreased by 97.3% (Figure 6). At 37°C, viscosity of the gels decreased, leading to thermofluidification of these gels.

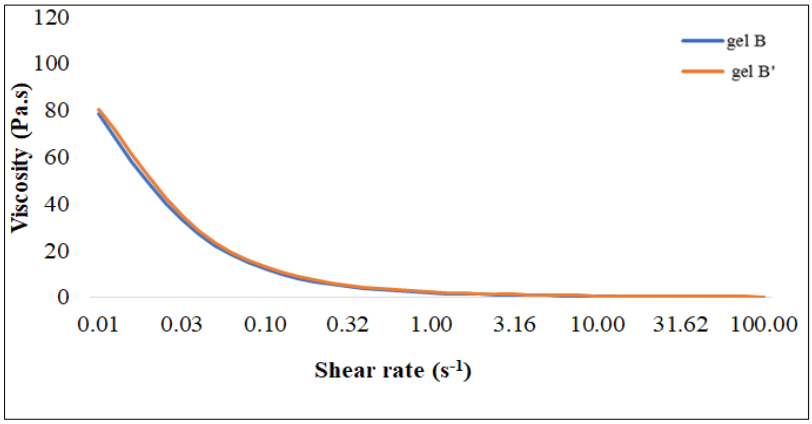

Figure 6: Viscosity of gels B and B' as a function of shear rate at 37°C ± 1 °C

Figure 7 showed 3 phases forming a hysteresis cycle demonstrating thixotropic behavior of the gels at 37°C.

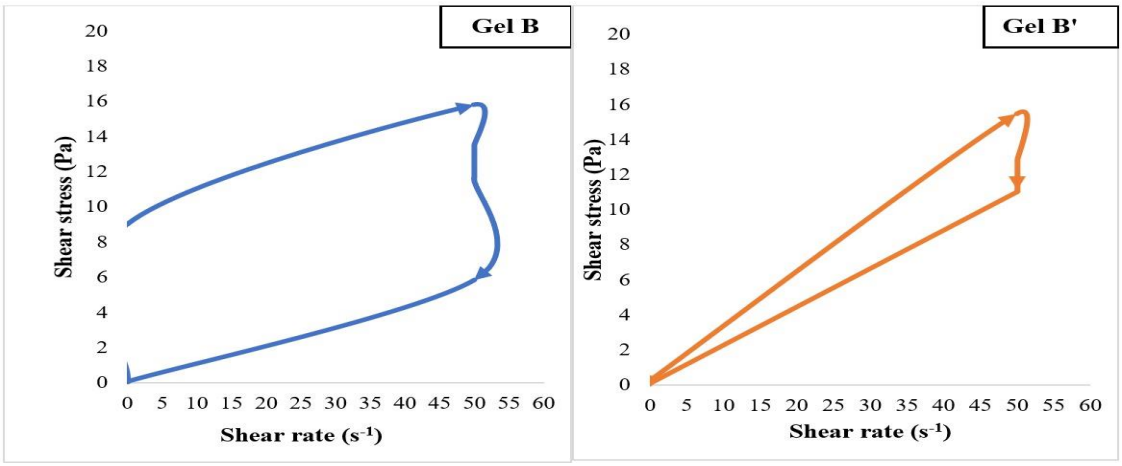

Figure 7: Shear stress as a function of shear rate at 37°C ± 1°C(gel B and B')

- **Gels Viscoelasticity Properties**

❖ **Measurement of Gel Viscoelasticity and Oscillatory Test:** The storage (G') and loss (G") moduli were determined by varying the frequency between 0.01 and 100 Hz at 25 ± 1°C (Figures 8 and 9).

- Gel B

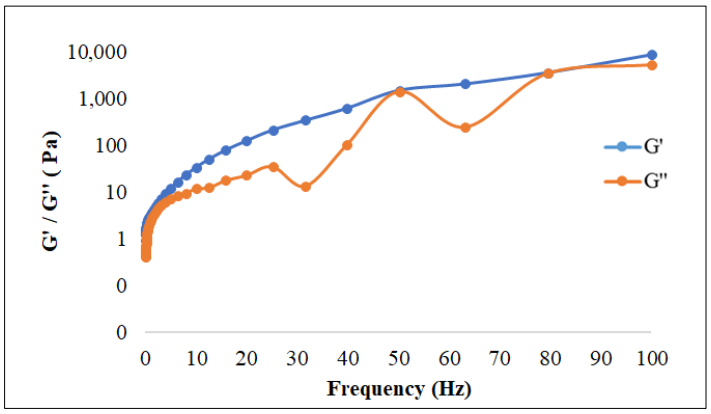

Figure 8: Storage and loss moduli gels evolution as a function of frequency at 25°C ± 1 °C

- Gel B'

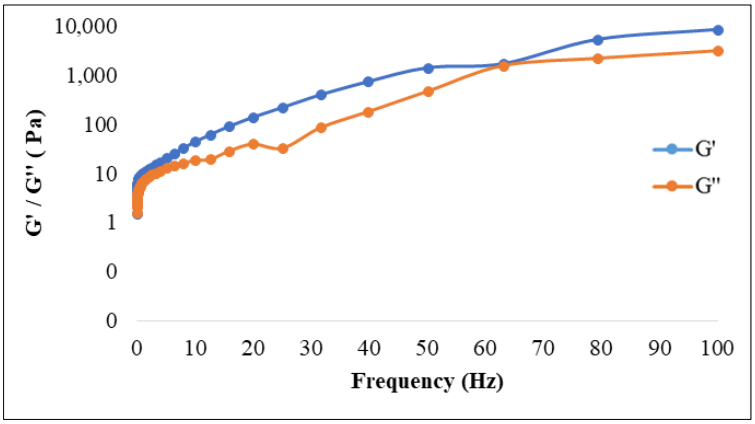

Figure 9: Storage and loss moduli gels evolution as a function of frequency at 25°C ± 1 °C

Over the frequency range from 0.01 to 100 Hz, gels B and B' generally exhibited storage modulus greater than loss modulus, indicating dominant elastic behavior of these gels (Figures 8 and 9).

❖ **Influence of Temperature on Gels Viscoelasticity**

Figures 10 and 11 show the behavior of gels submitted to a temperature range of 5 to 90°C. These gels were viscoelastic and not thermosensitive.

- Gel B

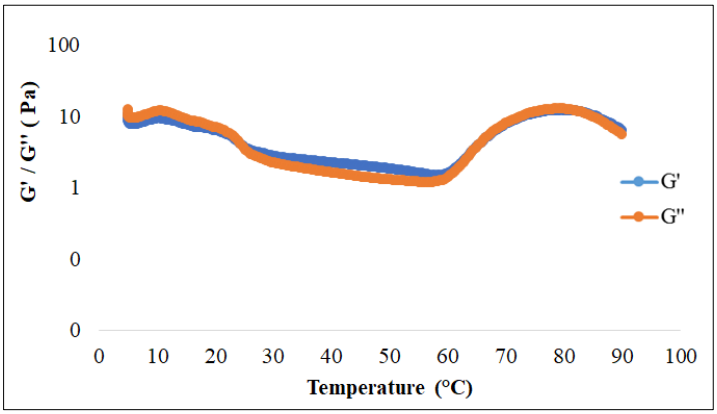

Figure 10: Storage and loss moduli gels evolution as a function of temperature (Gel B)

- Gel B’

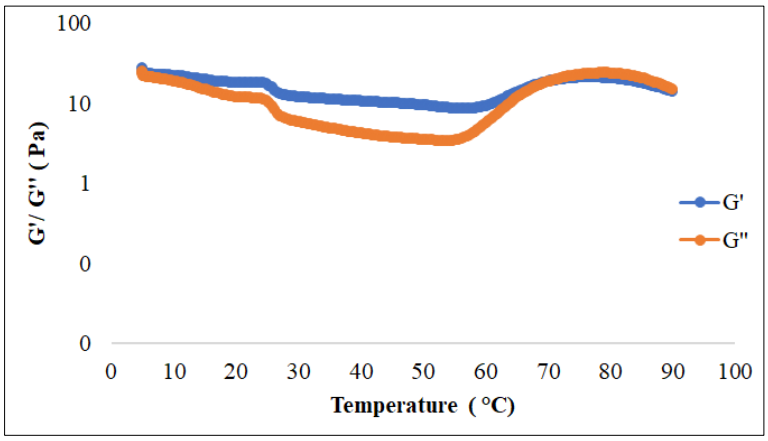

Figure 11: Storage and loss moduli gels evolution as a function of temperature (Gel B’)

Stability Tests over 28 days

The pH was 4.91 ± 2 at D_0 and 4.83 ± 1 at D_{28} for gels B and B'. However, they were unstable at 2000 rpm. This instability was characterized by phase separation for gels B and B'. Gel B stored at 25 ± 2°C, 6 ± 2°C and 40 ± 2°C for 28 days was stable and homogeneous. In fact, we observed no change in odor or color. Likewise, no phase separation or sedimentation was observed. Gel B' was stable and homogeneous at 25 ± 2°C and 6 ± 2°C but unstable at 40 ± 2°C, with phase separation leading to a loss of homogeneity and a change in appearance from day 7 onwards. At cold temperatures and at room temperature, we observed no change in color or odor.

5. DISCUSSION

At the end of the extraction process, the yield was 17.55 ± 4.04%, which was higher than the yield obtained by Sommano *et al.*, which was 10.45% (Sommano SR *et al*., 2018). The yield of extraction can be improved by applying the optimal condition, but it also depends on the mango cultivar and the efficacy of the extraction technique and equipment. The pectin obtained was green-brown in color, with a faint odor and a characteristic taste. These results were similar to those of Malviya *et al.*, (Malviya R *et al*., 2012). The pectin obtained was a low methylated pectin because the DE was less than 50%. Low-methylated pectin have the ability to form gels in the presence of sucrose and divalent cations such as calcium in an acidic environment (May CD, 1990; Chan SY *et al*., 2017). Organoleptic characterization showed that the coconut oil was ivory white, viscous, and had a characteristic odor. These results are similar to those of Tuo Kouassi *et al.* (Tuo-Kouassi AN *et al*., 2020). The different index of coconut oil are listed in Table II. The peroxide index of coconut oil was in accordance with the Codex Alimentarius (< 15 meq O2/kg), indicating a low risk of rancidity (OMS *et al*, 1999). The acid index of coconut oil was in accordance with Codex Alimentarius (< 4 mg KOH/g). Coconut oil will therefore have a longer shelf life. The iodine value of coconut oil was in line with Codex Alimentarius (6.3-10 g I_2/100g of coconut oil), confirming the presence of unsaturation in the oil's chemical structure. The saponification index is fat identification factor according to the Codex Alimentarius; its high value would reflect the length of the carbon chain of fatty acids in triacylglycerols (Abiodun *et al*., 2014). The higher the saponification index, the richer the fat is in short-chain fatty acids. Coconut oil had a saponification index lower than the Codex Alimentarius (248-265 mg KOH/g). This value would reflect that coconut oil had a shorter carbon chain of fatty acids and was less rich in short carbon chain fatty acids. The moisture content of coconut oil (Table II), was higher than the value obtained by Suryani *et al*., which was 0.1% (Suryani S *et al*., 2020). This low water content would be beneficial for the preservation of coconut oil. Two gels, B and B', were prepared from “Amélie” variety pectin gel and coconut oil (Table I). These gels were obtained from a dispersion of coconut oil in a mango pectin gel. At J_0, they were homogeneous, without air bubbles, with a characteristic coconut smell and a dark brown color (Figure 1). These emulsified gels with a continuous aqueous phase could help improve the hydration of black hair, which is dry and fragile (Martini MC, 2011). Thanks to its film-forming properties, pectin could coat the hair and make it more resistant to traction. The median diameter of the globules dispersed in these gels was between 1 and 5 µm, meaning that these were coarse emulsions. The interdecile ratio of gel B (2.28) was lower than that of gel B' (3.18), indicating a better distribution of globule size in gel B (Figure 3). In fact, an interdecile ratio close to 1 was an indicator of good globule distribution. Increasing the proportion of coconut oil in the gel appeared to affect globule distribution. Gel B was therefore more homogeneous than gel B'. These gels were unstable when centrifuged at 2000 rpm. This instability corresponded to phase separation, which could be explained by the stress applied. The pH of the different gels varied very little. In 28 days, it had changed from 4.91 on day 0 to 4.83 on day 28 for gels B and B', which was close to the pH of the scalp (4.5< pH < 5.5). Such pH values allow the

cuticular cells to tighten and strengthen the protection of the hair fiber (Diallo M *et al*., 2016). In addition, the pH of the scalp (4.5< pH < 5.5)". There is no significant difference between the pH values, so we can conclude that increasing the oil proportion did not have a significant influence on the pH. pH stability over time is an indicator of good microbiological preservation of the different preparations (Rosso L *et al*., 1995). Gel B stored at 25 ± 2°C, 6 ± 2°C, and 40 ± 2°C for 28 days remained stable. No change in odor or color was observed, and the gel was homogeneous. This stability could be explained by the emulsifying properties of pectin and the thickening properties of sucrose. The emulsifying property of pectin was demonstrated by Akhtar *et al*., (Akhtar M *et al*., 2002), while its stabilizing potential was demonstrated by Jung *et al.* (Jung J *et al.*, 2012). Gel B' remained stable over time at 25 ± 2°C and 6 ± 2°C but was unstable at 40 ± 2°C. Phase separation leading to a loss of homogeneity and a change in appearance from day 7 onwards was observed at 40 ± 2°C. This instability could be explained by the high temperature causing forced degradation. The various gels presented shear thinning behavior. Viscosity decreased as shear rate increased, regardless of the working temperature, at 25°C or 37°C (Figures 4 and 6). This behavior would facilitate good spreading of the gel on the scalp, a better contact surface with the hair and therefore better penetration of the active substances. This shear thinning behavior of pectin gels has been demonstrated by Marcotte *et al.*, Koubala *et al.*, (Marcotte M *et al*., 2001; Koubala BB *et al*., 2009). However, gels B and B' at a temperature of 37°C (Figure 6) had lower viscosities than those at 25°C (Figure 4). This result reflects a thermofluidification of the gels at 37°C due to the disentanglement of macromolecules under the influence of the increase in temperature.

All gels were thixotropic at 25 and 37°C (Figures 5 and 7). Indeed, the different rheograms showing the evolution of the shear stress as a function of the shear rate described a hysteresis cycle, corresponding to a reversible and time-dependent viscosity": However, the thixotropy of gel B is less pronounced than that of gel B'. There was a destructuring of gels by induced shear, then a progressive restructuring after removal of the shear. These results indicate that at 25°C, gels will present ability to easily get out of its packaging, while gels at 37°C, will easily spread on the scalp and hair, then will regain its structure and flow with difficulty (Coussot P *et al*., 2002).

Gels viscoelasticity tests at 25°C in frequency variation made it possible to determine the behavior of the different gels. Thus, gels B and B' were mainly elastic ($0° < \delta < 45°$). Regardless of frequency, the storage modulus was generally higher than the loss modulus over most of the temperature range (Figures 8 and 9). The gels behaved essentially as elastic fluids, confirming the firmness of these gels (Han *et al*., 2017) as mango pectin gels, due to their interchain entanglements of the pectin molecule (Piermaria *et al*., 2008). This result is similar to the results obtained for agarose gels as strong polysaccharide gels, due to the storage modulus in these polysaccharides being greater than the loss modulus at all frequencies (Ross-Murphy, 1995). Over the entire temperature range, gels B and B' were viscoelastic and non-thermosensitive, as the storage and loss moduli were observed simultaneously and were almost linear. No gel-sol transition point was observed (Figures 10 and 11).

6. CONCLUSION

The objective of this study was to formulate hair gels based on mango pectin combined with coconut oil to treat traction alopecia in black women. Organoleptic, microscopic, physicochemical, and rheological characterization of gels were performed. Two formulations were developed based on mango pectin gel and coconut oil (B and B'). They were homogeneous, without air bubbles, and had a characteristic coconut odor. They were lipophilic/hydrophilic type and unstable when centrifuged at 2000 rpm, causing phase separation. At the microscopic level, they all corresponded to coarse emulsions. The distribution of globules was better in the gel with a low-fat content (Gel B). Gel B was stable at hot, cold, and room temperatures for 28 days. In terms of rheological characterization, all gels were almost non-thermosensitive, then presented shear thinning behavior, thixotropic behavior and elastic behavior at all frequencies. Ultimately, gel B had the best macroscopic, microscopic, physicochemical and rheological characteristics. The results of this work are encouraging for the development of phytocosmetics for the treatment of traction alopecia in black women.

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