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Formulation of Hair Gels Based on Mango Pectin and "*Makoré*" Butter

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Abstract

Non-scarring alopecia results from a loss of hair or reduction in hair follicle growth. Among its forms, traction alopecia is the most common in black women and women of African descent. Although several treatments exist, their use remains limited due to the associated adverse effects. This study aimed to develop hair gels based on mango pectin combined with "*makoré*" butter as a natural alternative for the management of this condition. The extracted raw materials were characterized. The pectin gel was prepared at 95 °C under stirring at 800 rpm, then dispersed in the lipophilic phase under constant agitation at 1500 rpm for 10 minutes to obtain two formulations, D and D′. The gels were homogeneous, free from air bubbles and with a characteristic odor. They were unstable under centrifugation. Microscopic analysis revealed coarse emulsions, with a better distribution of globules in gel D. Over 28 days, the preparations exhibited minimal variation in pH, reflecting good microbiological stability. Both gels remained stable at 25 ± 2°C. Rheological evaluation showed shear thinning, viscoelastic, and thixotropic behavior. Overall, gel D demonstrated beneficial properties for hair, confirming its potential use for the development of phytocosmetic formulations intended for the management of traction alopecia in black women.

Keywords: Gels, "*Makoré*" Butter, Non-Scarring Alopecia, Pectin, Shear Thinning.

1. INTRODUCTION

Alopecia is a disorder that affects the hair follicle and is defined as a partial or total loss of hair. It has a negative impact on self-esteem, mental health, and overall quality of life (Kumar N *et al.*, 2012). Two main types of alopecia are recognized: scarring and non-scarring alopecia. Among non-scarring forms, traction alopecia occupies a prominent place (De Lorenzi C *et al.*, 2018). Traction alopecia is a traumatic form of alopecia that results from hair grooming practices. It is the most prevalent type among African and African-descendant women. According to a South African study, its prevalence was 17.1% among students aged 6 to 21 years and 31.7% among women aged 18 to 86 years (Khumalo NP *et al.*, 2008). Although several treatments exist, their use in the management of traction alopecia remains controversial due to the occurrence of various adverse effects, as exemplified by minoxidil (Kirakosvan R *et al.*, 2018; Rossi A *et al.*, 2012). Given these undesirable effects, a natural and well-tolerated therapeutic approach appears to be a promising alternative for managing traction alopecia in Black women. Across the world, and particularly in Africa, numerous plants and plant-based formulations are traditionally used for hair care (Bharti M *et al.*, 2020). The butter obtained from the kernel of *Thieghemella heckelii* A.Chev. (Sapotaceae), commonly known as "*makoré*" butter, is among them. It is traditionally used in West Africa (notably in Côte d'Ivoire, Liberia, and Nigeria) as a skin and hair care pomade (*Tieghemella heckelii* (PROTA)). In addition, pectin is an antioxidant, biocompatible, biodegradable, and non-toxic polymer found in the cell walls of plants, particularly *Mangifera indica* L. (Anacardiaceae) (Grant GT *et al.*, 1973; Colodel C *et al.*, 2019; Watts P *et al.*, 2009; Ro J *et al.*, 2015). Pectin can form hydrogels in the presence of sucrose and divalent cations such as calcium (May CD, 1990; Morris ER *et al.*, 1982; Chan SY *et al.*, 2017), enabling moisture retention and conferring humectant properties. Therefore, gels based on these natural substances could represent an innovative alternative for the management of traction alopecia. The present study aimed to formulate hair gels based on mango pectin combined with "*makoré*" butter 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., Anacardiaceae) and "*makoré"* butter (*Thieghemella heckelii*, Sapotaceae). The mangoes used belonged to the "Amélie" variety, selected for their fibrous nature, availability in Côte d'Ivoire, and degree of ripeness. Mature fruits were harvested in Korhogo by a technician from the National Center for Agronomic Research (Korhogo branch), between May and June 2021. An initial botanical identification was performed on site, followed by confirmation at the National Floristic Center of Abidjan, where the specimen was registered under the herbarium number UCJ000983. Mango peel waste was used for pectin extraction, while "*makoré"* butter was obtained by cold manual pressing of "*makoré"* fruits.

2.2 Solvents and Reagents

Analytical-grade solvents and reagents were purchased from Sigma Chemical Co. (St. Louis, MO, USA) and included: 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 (batchos no. F04950500). Osmosis water was used for all experimental procedures (ThermoFisher Scientific, France).

3. METHODS

3.1 Pectin Extraction

Pectin extraction was performed following the procedure described by Chaiwarit *et al.* (Chaiwarit, T *et al*., 2020). 20 g of powdered mango peels were 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 and precipitated with two volumes of absolute ethanol and stored for one hour to obtain wet pectin. The wet pectin was dried in an oven at 50 °C for 48 hours and ground into powder. The extraction yield (%) was calculated after two replicates.

3.2 Characterization of the Pectin

Organoleptic characteristics, moisture content, and the degree of esterification of the extracted pectin were determined.

3.3 Characterization of "*Makoré*" Butter

Characterization of "*makoré*" butter included both organoleptic and physicochemical analyses.

3.4 Formulation and Characterization of Gels

3.4.1 Formulation

The gels were composed of two phases: a hydrophilic phase (mango pectin gel) and a lipophilic phase ("*makoré*" butter). The proportions of "*makoré*" butter varied among formulations, as shown in Table I.

Table I: Composition of mango pectin and "*makoré*" butter gels

Composition (%) Different gels	Mango pectin gel	"*makoré*" butter	Propylparaben
Gel D	94.8	5	0.2
Gel D'	89.8	10	0.2

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

3.4.2 Gels Characterization

Organoleptic Characterization: Organoleptic characterization assessed appearance, odor, and color of the gels. The appearance was evaluated by spreading a small amount of gel to check for the presence or absence of lumps and air bubbles.

Microscopic Characterization: A drop of gel was placed between a slide and a coverslip, and the size of at least 300 dispersed globules was measured using an optical microscope equipped with a micrometric scale (magnification ×10).

Physicochemical Characterization

- **pH Determination:** The pH was determined using a HI 2211, electronic pH meter equipped with a glass electrode. After calibration, the electrode was wiped and immersed in a beaker containing a 10% gel solution. Each measurement was repeated three times.
- **Determination of Emulsion Type:** The emulsion type was determined using two dyes: methylene blue (hydrosoluble) and Sudan III (liposoluble). A small quantity of gel was spread on a watch glass, and a drop of each dye solution (1%) was added. The emulsion type was identified according to the affinity of the dyes for the continuous and dispersed phases.
- **Rheological Characterization:** The rheological properties of the gels were evaluated using a Kinexus rotational rheometer (Malvern Instruments, Massy, France) equipped with a plane-plane geometry, 1 mm of gap and a cover to prevent drying of samples. The Linear Viscoelastic Region (LVR) was determined by oscillatory strain sweep (1 Hz, 0.01–10 Pa) following Doucet *et al* (Doucet D *et al*., 2021). Each test was repeated three times on approximately 10 g of gel, and the averages were analyzed using rSpace software. Measurements were performed to assess the variation of shear stress as a function of shear rate (0.01–50 s^{-1}) at 5 ±

1 °C and 37 ± 1 °C, identifying typology and thixotropy 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). Viscoelastic parameters were assessed as a function of temperature (5–90 °C) at a frequency of 1 Hz and strain of 1%, and also under oscillatory mode in the frequency range 0.01–100 Hz at 25 ± 1 °C.

28-Day Stability Tests

Stability tests were conducted over 28 days (days 1, 2, 3, 7, 14, 21, and 28) on gels stored for 24 hours in the refrigerator. Centrifugation and thermal stability tests were performed at 2000 rpm for 10 minutes and at 6 ± 2 °C, 25 ± 2 °C, and 40 ± 2 °C, respectively. Changes in color, odor, appearance and pH were recorded.

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 Characterization of Pectin

After extraction, the mango pectin appeared greenish-brown, had a faint odor, and a characteristic taste. The moisture content was 7.10 ± 0.10%, and the degree of esterification was 38%, below 50%, confirming that the extracted pectin was low-methoxyl pectin (LMP).

4.2 Characterization of "*Makoré*" Butter

4.2.1 Organoleptic Characterization

The butter exhibited a light-yellow color**,** solid consistency at 25 ± 2 °C, and a characteristic odor of "*makoré*" fruit.

4.2.2 Physicochemical Characterization

Physicochemical parameters assessed included moisture content, then iodine, peroxide, and saponification values of "*makoré*" butter. Moisture levels were relatively low (Table II). The peroxide value complied with Codex Alimentarius standards (below 15 mEq O_2/kg). The iodine value was similar to that of shea butter (30–75 g I_2/100 g), while the saponification value was nearly identical to that of coconut oil (248–265 mg KOH/g), as reported in the Codex Alimentarius. The moisture content and index values are listed in Table II.

Table II: Physicochemical analysis of "*makoré*" butter (n= 3)

Raw material	Residual moisture (%)	Peroxide value (mEq O_2/ Kg)	Iodine value (g d'I_2/ 100 g)	Saponification index (mg KOH/g)
"M*akoré*" butter	0.3 ± 0.00	1.8 ± 0.3	39.6 ± 0.6	248.5 ± 0.8

4.3 Formulation and Characterization of Gels

4.3.1 *Organoleptic Characterization*

The gels were homogeneous, free of air bubbles, and had a characteristic odor of "*makoré*" fruit. The gels containing "*makoré*" butter presented a light brown color (Figure 1).

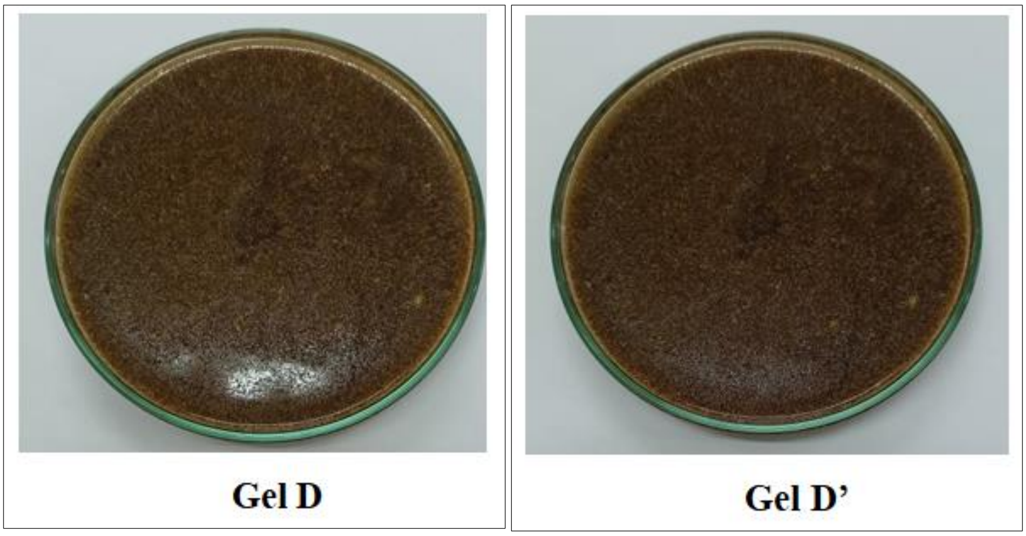

Figure 1: *Makoré butter and pectin based gels* D: (mixture of 94.8% pectin gel + 5% Makoré butter) D': (mixture of 89.8% pectin gel + 10% Makoré butter)

4.3.2 Microscopic examination

The dispersed globules of "*makoré*" butter in gels D and D′ observed under ×10 magnification are shown in Figure 2.

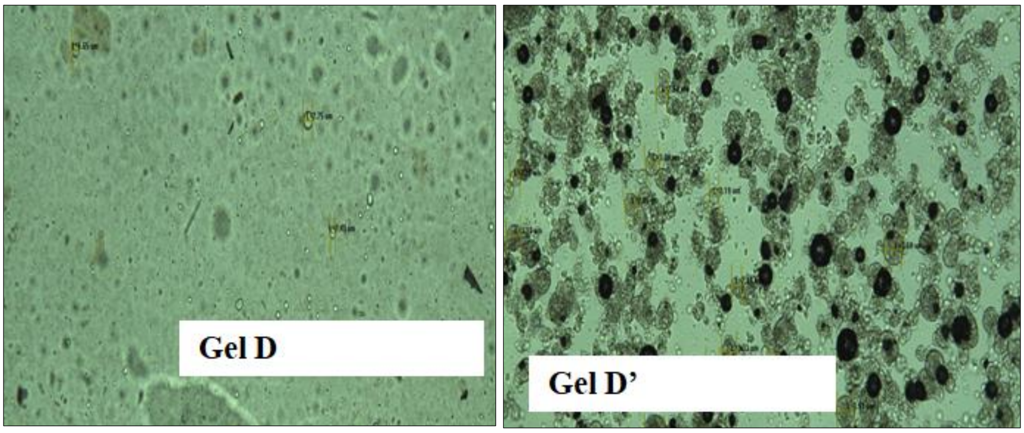

Figure 2: Dispersed globules of makore butter in gels (Gel D et D’)

The median globule diameter in gel D was 2.29 ± 0.68 µm, indicating a coarse emulsion. The 10th decile (D10) was 1.43 µm and the 90th decile (D90) was 3.30 µm, with an interdecile ratio (D90/D10) of 2.31. For gel D′, the median globule diameter was 2.03 ± 0.98 µm, also corresponding to a coarse emulsion, with D10 equal to 1.03 µm and D90 equal to 3.33 µm, resulting in an interdecile ratio of 3.23. The distribution of globules was better in the gel with a lower lipid content (gel D) compared to gel D′. The closer the interdecile ratio is to 1, the more uniform the dispersion, confirming the greater homogeneity of gel D.

4.3.3 Physicochemical and Stability Tests

pH and Emulsion Type

The pH of the gels was 4.89 ± 2, and they were classified as Lipophilic/Hydrophilic (L/H) type emulsions.

Rheological characterization

- **Flow Behavior at Different Temperatures**

❖ **Flow Curves at 25 °C**

Both gel D and gel D′ exhibited high viscosity at low shear rates (Figure 3). The viscosity decreased with increasing shear rate, confirming their shear-thinning behavior. Between shear rates of 0.01 and 1 s^{-1}, viscosity decreased by 98% (Figure 3).

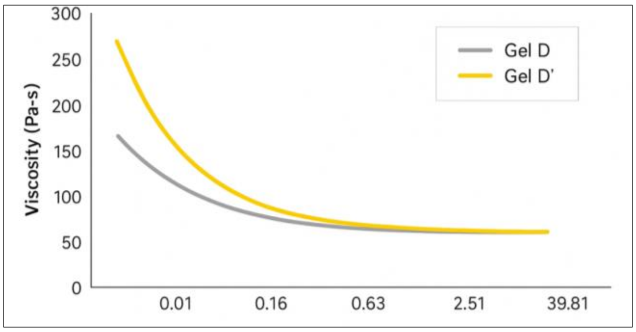

Figure 3: Viscosity of gels as a function of shear rate at 25 ± 1 °C

Figure 4 showed 3 phases forming a hysteresis cycle demonstrating thixotropic behavior of the gels at 25°C.

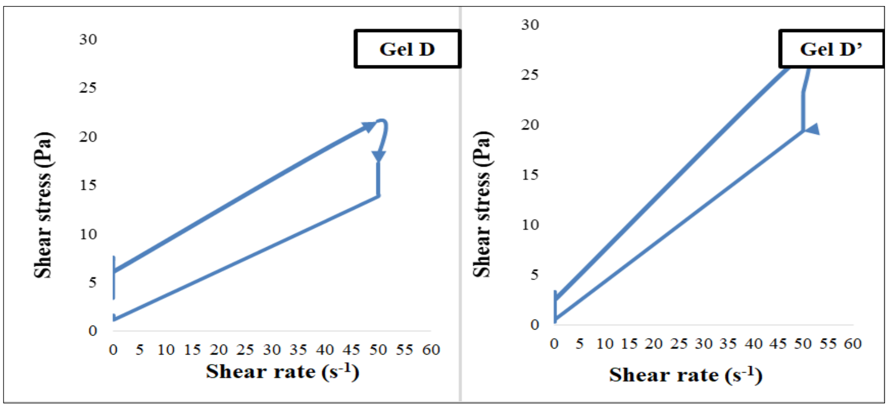

Figure 4: Shear stress as a function of shear rate at 25°C ± 1°C

❖ **Flow Curves At 37 °C**

A similar shear-thinning behavior was observed at 37 °C (Figure 5). Viscosity decreased by 97.6% between 0.01 and 1 s^{-1}. The rise in temperature caused a thermofluidification effect, reducing viscosity.

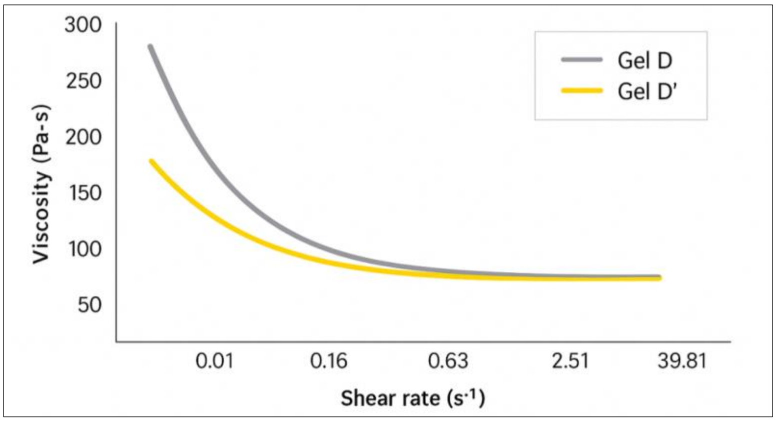

Figure 5: Viscosity of gels as a function of shear rate at 37 ± 1 °C

Figure 6 showed 3 phases forming a hysteresis cycle demonstrating thixotropic behavior of the gels at 37°C.

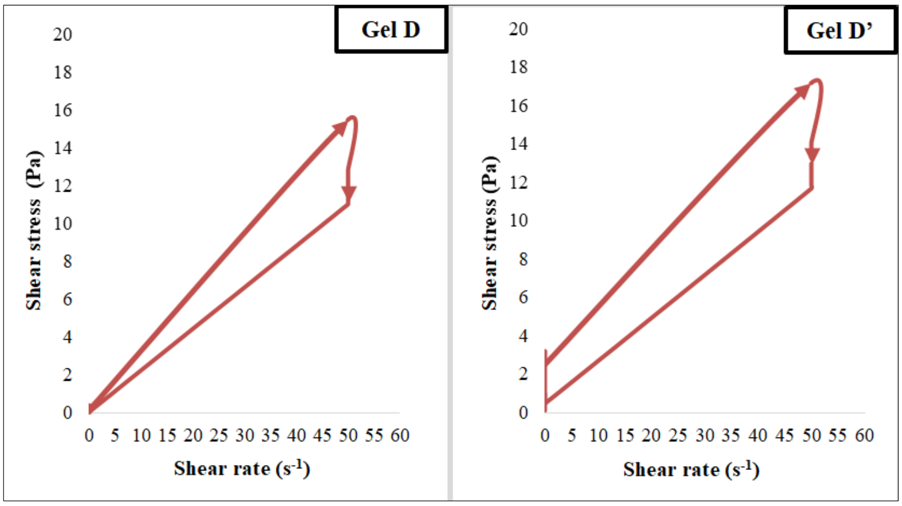

Figure 6: Shear stress as a function of shear rate at 37°C ± 1°C

- **Viscoelasticity Study**

❖ **Frequency Sweep**

The storage (G′) and loss (G″) moduli were determined over a frequency range of 0.01–100 Hz at 25 ± 1 °C (Figures 7 and 8).

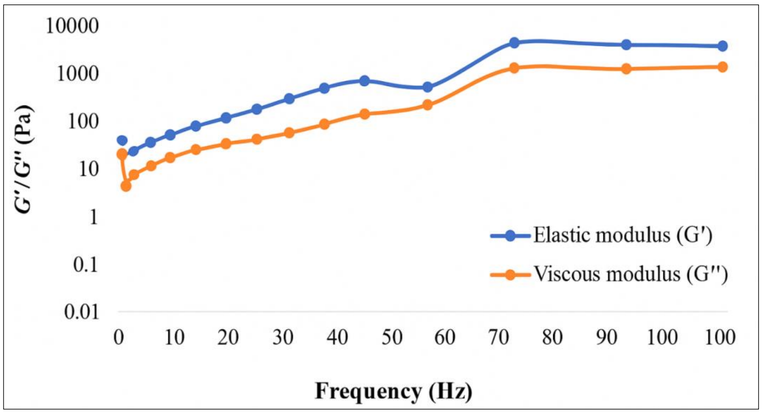

Figure 7: Evolution of storage (G′) and loss (G″) moduli of gel D as a function of frequency at 25 ± 1 °C

Over the tested frequency range, both gels generally exhibited G′ > G″, with a loss angle (δ) between 0° < δ < 45°, indicating elastic-dominant behavior (Figure 7).

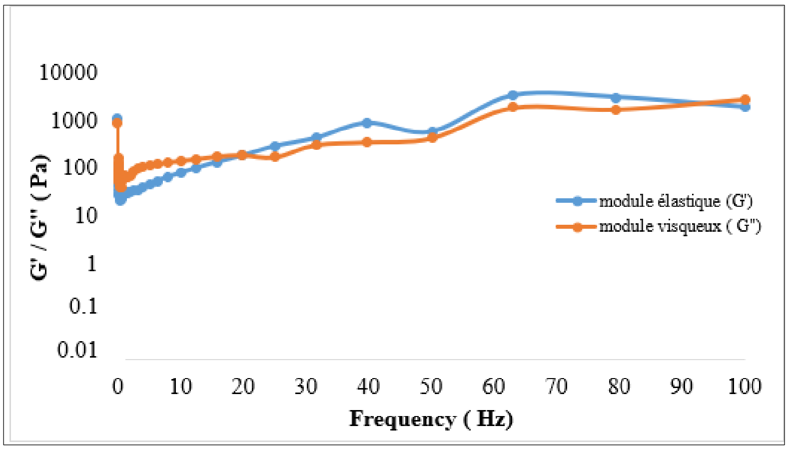

Figure 8: Evolution of storage (G′) and loss (G″) moduli of gel D′ as a function of frequency at 25 ± 1 °C

Gel D′ showed viscoelastic behavior, being more viscous at low frequencies and more elastic at higher frequencies (Figure 8).

❖ **Effect of Temperature on Viscoelasticity**

Figures 9 and 10 illustrate the viscoelastic response of gels D and D′ over a temperature range of 5–90 °C. Both exhibited viscoelastic but non-thermosensitive behavior.

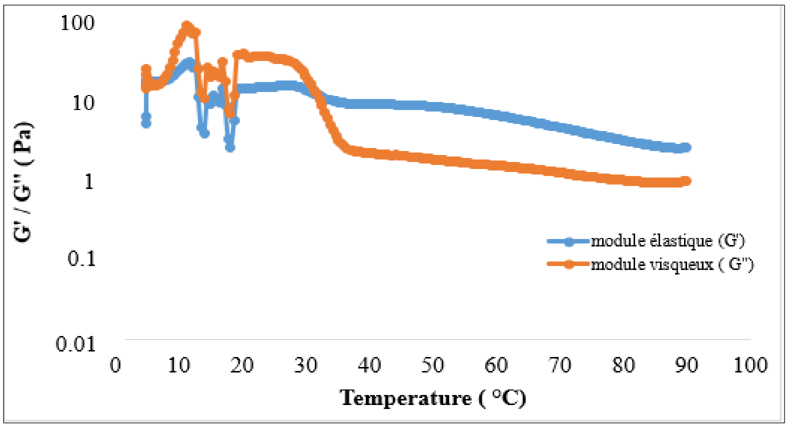

Figure 9: Evolution of storage (G′) and loss (G″) moduli of gel D as a function of temperature

Below 32 °C, G″ exceeded G′, while above 32 °C, G′ became predominant. Both moduli remained linear across temperatures, indicating thermal stability and non-thermosensitive nature.

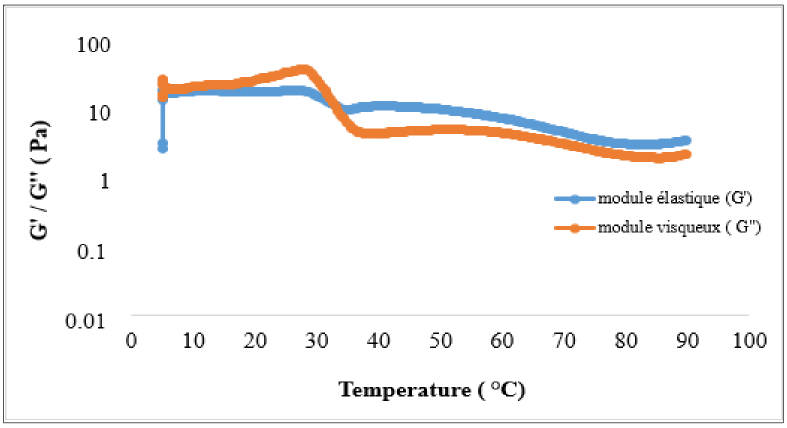

Figure 10: Evolution of storage (G′) and loss (G″) moduli of gel D′ as a function of temperature

Stability Tests Over 28 Days

At day 0 (J0), the pH of gels D and D′ was 4.89 ± 2, which slightly decreased to 4.83 ± 1 by day 28 (J28). However, both formulations were unstable under centrifugation (2000 rpm), showing phase separation. Gels D and D′ remained stable and homogeneous at 25 ± 2 °C. At ambient temperature, no color or odor changes were observed.

5. DISCUSSION

Following the extraction process, the average yield was 17.55 ± 4.04%, a value higher than that obtained by Somamo *et al.*, which was 10.45% (Sommano SR *et al.*, 2018). This difference could be attributed to the variation in mango varieties used for the extraction, then efficiency of the extraction technique and equipment. The pectin obtained was green-brown in color, had a mild odor, and exhibited a characteristic taste. These findings are consistent with those reported by Malviya et *al*. (Malviya R *et al*., 2012). The extracted pectin was identified as low-methoxyl pectin, as its degree of esterification (DE) was below 50%. Low-methoxyl pectins are capable of forming gels in the presence of sucrose and divalent cations such as calcium under acidic conditions (May CD, 1990; Chan SY *et al*., 2017). The evaluation of organoleptic properties showed that "*makoré*" butter was light yellow, solid at room temperature, and possessed a characteristic "makoré" fruits aroma. The peroxide value of "*makoré*" butter complied with the Codex Alimentarius standard (15 meq O_2/kg), indicating a low risk of rancidity (OMS *et al.*, 1999). The iodine value was nearly identical to that of shea butter, as reported in the Codex Alimentarius (30–75 g I_2/100 g) (Table II), confirming the presence of unsaturation in the chemical composition of "*makoré*" butter. The saponification value is an identifying factor for fats according to the Codex Alimentarius; a high value indicates a longer fatty acid chain in triacylglycerols. The higher the saponification value, the richer the fat is in short-chain fatty acids. "*makoré*" butter exhibited a saponification value close to that of coconut oil reported in the Codex Alimentarius (248–265 mg KOH/g) (Table II), reflecting the fatty acid chain length and the presence of short-chain fatty acids. The moisture content of "*makoré*" butter was 0.32 ± 0.03% (Table II), higher than that reported by Sylvie *et al*., which was 0.06% (Sylvie O, 2019). This low water content is advantageous for the preservation of "*makoré*" butter. Two gels, D and D', were prepared using pectin and "*makoré*" butter (Table I). These gels were obtained by dispersing "*makoré*" butter into the mango pectin gel. At day 0 (J0), the gels were homogeneous, free of air bubbles, had a characteristic "*makoré*" fruit odor, and were light brown in color (Figure 1). These emulsified gels with an aqueous continuous phase could contribute to the hydration of black hair, which is typically dry and fragile (Martini MC, 2011). Thanks to its film-forming properties, pectin could coat the hair shaft and improve its resistance to traction. "*makoré*" butter, through its emollient and nourishing properties, could promote maintenance and growth of the hair shaft (Tharanathan RN *et al.*, 2006).

The median diameter of dispersed droplets in these gels ranged from 1-5 µm, indicating coarse emulsions. The decile ratio of gel D (2.31) was lower than that of gel D' (3.23), reflecting a more uniform droplet size distribution in gel D, and thus greater homogeneity. Indeed, a decile ratio close to 1 is an indicator of good droplet distribution. Increasing the proportion of "*makoré*" butter in the gel appeared to affect droplet distribution. Therefore, gel D was more homogeneous than gel D'. These gels were unstable under centrifugation at 2000 rpm, corresponding to phase separation likely caused by the applied stress.

The pH of the gels showed minimal variation. Over 28 days, the pH changed from 4.89 at J_0 to 4.83 at J_{28} for both gels D and D', close to the physiological pH of the scalp (4.5 < pH < 5.5). Such pH values allow the cuticular cells to tighten, enhancing hair fiber protection (Diallo M *et al.*, 2016). Moreover, the stability of pH over time indicates good microbiological preservation of the preparations (Rosso L *et al.*, 1995). Gels D and D' remained stable and homogeneous at 25 ± 2°C. At room temperature, no change in color or odor was observed.

All prepared gels exhibited shear-thinning behavior. Viscosity decreased with increasing shear rate, regardless of working temperature, at 25°C or 37°C (Figures 3 and 5). This behavior facilitates even spreading of the gel on the scalp, maximizing surface contact with hair and enhancing active ingredient penetration. The shear-thinning property of pectin gels has been demonstrated by Marcotte *et al*., and Koubala *et al*., (Marcotte M *et al.*, 2001; Koubala *et al.*, 2009). However, gels D and D' at 37°C (Figure 5) showed lower viscosities compared to 25°C (Figure 3), indicating thermofluidization at higher temperature due to the disentanglement of macromolecules. All gels were thixotropic at 25 and 37°C (Figures 4 and 6). 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. 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).

Viscoelasticity studies at 25°C under frequency variation mode revealed the gels' mechanical behavior. Gel D was predominantly elastic, with the storage modulus higher than the loss modulus across all frequencies (Figure 7). The gel behaved primarily as an elastic fluid, confirming its firmness (Han W *et al.*, 2017), similar to mango pectin gels, due to interchain entanglements of molecular pectin (Piermaria J *et al.*, 2008). This result aligns with findings on agarose gels as strong polysaccharide gels, where the storage modulus exceeds the loss modulus at all frequencies (Ross-Murphy SB, 1995). Conversely, gel D' exhibited viscoelastic behavior, being viscous at low frequencies and elastic at higher frequencies (Figure 8). The high proportion of "*makoré*" butter (10%) in gel D' likely influenced its behavior, possibly affecting the gel's porous network, as Shukla *et al.,* reported that increasing frequency reduces the viscous modulus (G'') of fat while increasing its elastic modulus (G') (Shukla A *et al.*, 1995). Across all temperatures, gels D and D' demonstrated viscoelastic but non-thermosensitive behavior, with storage and loss moduli observed simultaneously and almost linear. A gel-to-sol transition point was noted around body temperature (33°C) (Figures 9 and 10) (Strigo IA *et al.*, 2000).

5. CONCLUSION

The objective of this study was to formulate hair gels based on mango pectin combined with "*makoré*" butter to trait traction alopecia in black women. The formulated hair gels were characterized macroscopically, microscopically, physicochemically, and rheologically. Two formulations were developed using mango pectin gel and coconut oil (gels D and D'). They were homogeneous, free of air bubbles, and had the characteristic odor of "*makoré*" fruit. They were oil-in-water emulsions and unstable under centrifugation at 2000 rpm, leading to phase separation. Microscopically, all gels were coarse emulsions. Droplet distribution was better in the gel with lower fat content (gel D). Gels D and D' remained stable at room temperature for 28 days. Rheologically, gel D was almost non-thermosensitive, then presented shear thinning behavior, thixotropic behavior and elastic behavior at all frequencies. Both gels were viscoelastic across the temperature range, with a gel-to-sol transition observed near body temperature. Overall, gel D exhibited the best macroscopic, microscopic, physicochemical, and rheological characteristics. These results are encouraging for the development of phytocosmetic products for managing traction alopecia in black women.

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