IFSCC 2025 full paper (IFSCC2025-574)

"Exploring Flaxseed Mucilage's Potential in Cosmetic Applications"

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1. Introduction

In recent years there has been an increasing interest in exploring natural gums and mucilage, driven by consumer demand on natural alternatives to synthetic polymers. In plants, gums and mucilage are produced in various tissues, including bark, seeds, and roots, and are composed of a wide range of sugar monomers, resulting in unique chemical structures and properties [1].

Seed mucilage is a complex mixture of water-soluble polysaccharides which exudate and unravel to full extension upon the hydration of the seed, creating a translucent colloidal gel-like substance on the outer layer of the seed coat [2]. Among the numerous sources of mucilage, flax (Linum usitatissimum L.) seeds have gained significant attention due to their high mucilage content and their potential applications, especially in pharmaceutical and healthcare products [3]. Additionally, flaxseed mucilage (FM) is a by-product of industrial high quality flaxseed oil production [4,5] indicating that it can be commercially obtained in an industrial scale for further use, minimising waste and providing an additional source of income for flax farmers.

Although all seed mucilage is composed of polysaccharides, proteins, lipids, and minerals, the composition of the polysaccharides varies between species [6]. Flaxseed mucilage is considered a heteropolysaccharide composed of two polysaccharide types, a neutral arabinoxylan fraction and an acidic pectic-like rhabdogalacturonan fraction [7]. A number of studies have suggested that flaxseed mucilage has potential for cosmetic applications but a minimal number of them has further explored this [8-10]. This project aimed to explore the use of flaxseed mucilage in cosmetic applications, by examining its stability when combined with active ingredients, specifically sodium hyaluronate, compared to other conventional natural and synthetic gelling materials.

2. Materials and Methods

2.1 Mucilage extraction and dehydration

The method was adapted from Cui et al. [7] with some modifications. Flax seed (Whole Food Earth, China) was added to distilled water, at 1:13 seed to water ratio, which was previously

adjusted to pH 6.5 using 0.2M NaOH or HCl. The mixture was heated on a magnetic hotplate at 70-75˚C for 3 hours and magnetically stirred at 600 rpm. The mixture was then cooled down to 40˚C and the crude mucilage was filtered using a cheesecloth. To prepare the dehydrated mucilage, crude mucilage was spread onto a tray and dried at 50˚C in a dehydrator (Buffalo Appliances, UK) for 18 hours. The dried mucilage was then ground to a fine powder.

2.2 Hydrocolloid dispersion preparation using rehydrated flaxseed mucilage

To prepare the 4% flaxseed mucilage hydrocolloid dispersion, deionised water was heated to 60°C and mucilage powder was added slowly under continuous stirring at 800 rpm for 1 hour to ensure full dissolution.

2.3. Control gels preparation using guar gum and carbomer

1% (w/v) Guar Gum and 0.1% (w/v) carbomer dispersions were prepared as control solutions. Deionised water was heated to 60°C using a magnetic hotplate and the water was magnetically stirred at 800rpm. The Guar Gum powder was slowly sifted with a 40-mesh screen sieve into the water vortex to avoid clumping. The dispersion was magnetically stirred for 1 hour to ensure complete dissolution. The carbomer was pre-dispersed with a small amount of water to create a slurry. The slurry was added in aliquots into the water vortex and was magnetically stirred for 1 hour to allow the dispersion to be fully dissolved. The pH was adjusted with NaOH/HCl to 6.5, the same pH as mucilage dispersion.

2.3. Preparation of gels with hyaluronic acid

Hydrocolloid suspensions of crude flaxseed mucilage (CFM), 4% rehydrated flaxseed mucilage (FMH), guar gum (GGH) and carbomer (CPH) were prepared as per Methods 2.1, 2.2 and 2.3. The gels were heated to 60°C and stirred magnetically at 800 rpm on a magnetic hotplate for 1 hour. Sodium hyaluronate at a concentration of 0.3% (w/v) was sifted in stages using a 40-mesh screen sieve and then magnetically stirred for 30 minutes until full dissolution. The gels were stored overnight in sealed containers in the fridge at 4°C, to ensure complete gelation and settling of structure. The pH of the gels was measured at 21°C in triplicate.

2.4 Rheology

All rheology measurements were performed using HAAKE MARS iQ Air Rheometer (Thermo Fisher Scientific, UK) with a 35 mm diameter parallel plate geometry (P35 Ti15 SE spindle) and a 1 mm gap spacing. Duplicate measurements were done at 21±1°C for each mucilage using separate samples. Results were obtained using HAAKE RheoWin Data Manager software (Thermo Fisher Scientific, UK) and were presented as the means of the two measurements.

2.4.1 Shear Rate Sweep

Samples were tested over 120 s with a shear rate range of 0.1 s-1 to 200 s-1.

2.4.2 Three-step thixotropy

Samples were tested using a succession of low, high and low shear rates (10 s^{-1}, 200 s^{-1}, and 10 s^{-1}), with each step taking 60 seconds. The percentage recovery was calculated using the

viscosity at the end of the third step (average of 10 readings) in relative to that at the end of the first step (Eq. 1).

$$\%\ Recovery\ = \frac{Average\ viscosity\ at\ the\ end\ of\ the\ third\ step}{Average\ viscosity\ at\ the\ end\ of\ the\ first\ step} \times 100$$

Equation 1: Calculating percentage of recovery

2.5 Texture Analysis

Texture profile analysis of samples was conducted with the TA.XT Plus Texture Analyser (Stable Micro Systems, USA). Samples were placed in glass jars (30g) and ½-inch cylindrical probe was placed 1 inch above sample surface. An immersion/de-immersion penetration test was conducted on the sample using the Pectin Penetration protocol on the Exponent software (Stable Micro Systems, UK), with the test speed set at 2mm s^{-1}, trigger force at 1 g and distance at 8 mm.

3. Results

Sodium Hyaluronate (NaHA), the sodium salt of hyaluronic acid, was the selected cosmetic active. Hyaluronic acid is natural polysaccharide whose properties can be improved by the presence of other bioactive molecules such as saccharides and plant extracts [11]. The combination of plant seed polysaccharides with hyaluronic acid has promise, as a study by Yoon and Lee [12] demonstrated improved rheological properties of combining hyaluronic acid with 3-4% tamarind seed polysaccharide.

Rheology and texture analysis were conducted on the four hydrocolloid suspensions with and without the active: 4% flaxseed mucilage (FM) and with NaHA (FMSH), extracted crude FM (CFM) and with NaHA (CMSH), Guar Gum (GG) and with NaHA (GGSH), and carbomer (CP) and NaHA (CPSH). At the time of writing, this appeared to be the first study of its kind exploring the effect of NaHA on both crude and rehydrated FM physical properties.

The results revealed that sodium hyaluronate had minimal impact on pH for the rehydrated mucilage at 4% and the crude extract. The carbomer dispersion was the only gel where the incorporation of sodium hyaluronate notably decreased the pH (from 6.5 to 5.6).

3.1 Flow Rheology

A series of shear rate sweeps individually comparing the hydrogels with and without sodium hyaluronate are seen in Figure 1 below. The initial viscosity of FMSH (4024.3 mPa·s) was considerably higher than of 4% FM (2135.5 mPa·s). This also applied to CMSH which had a much greater initial viscosity (1413 mPa·s) to crude FM (205.74 mPa·s). The peak viscosity values followed a similar pattern between FMSH and 4% FM, as well as CMSH and crude FM.

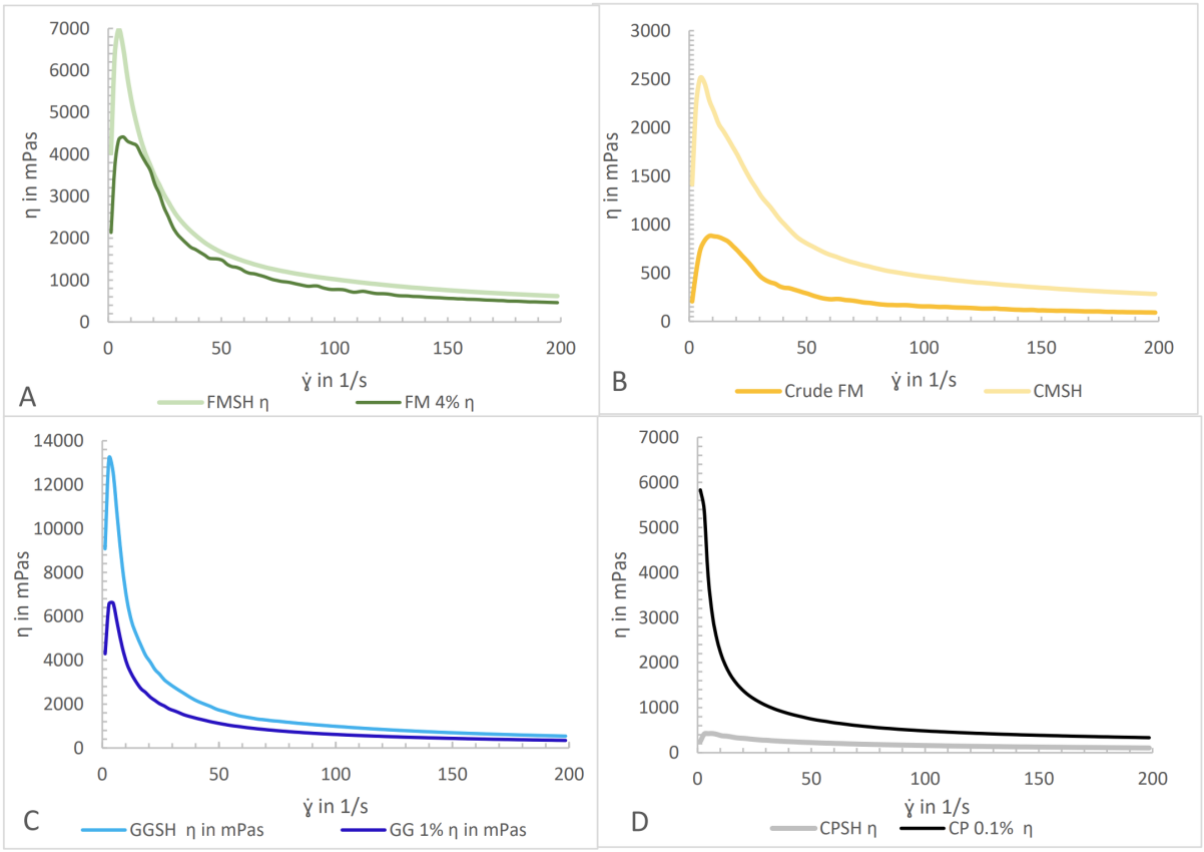

Figure 1. Shear Rate Sweep results (0.1-200s^{-1}) comparing the hydrogels with and without NaHA as function of viscosity against shear rate for (A) FMSH and FM; (B) CMSH and CFM; (C) GGSH and GG 1%; (D) CPSH and CP 0.1%.

3.2 Three-step Thixotropy

Three step graphs in Figure 2 compare the thixotropy of the hydrocolloids with active against the gelling ingredient on its own. The carbomer gel's thixotropic nature was not altered with the addition of sodium hyaluronate, as the recovery was still nearly 100%, but its viscosity at 10s^{-1} had changed. The 4% FM and FMSH had similar viscosity in the 1st and 2nd shear step, but the addition of the active increased recovery to 77.2%, much higher than the 55.1% of FM alone. This was similar for the crude FM, where CMSH had a higher recovery value at 64.9% than the crude extract (37%).

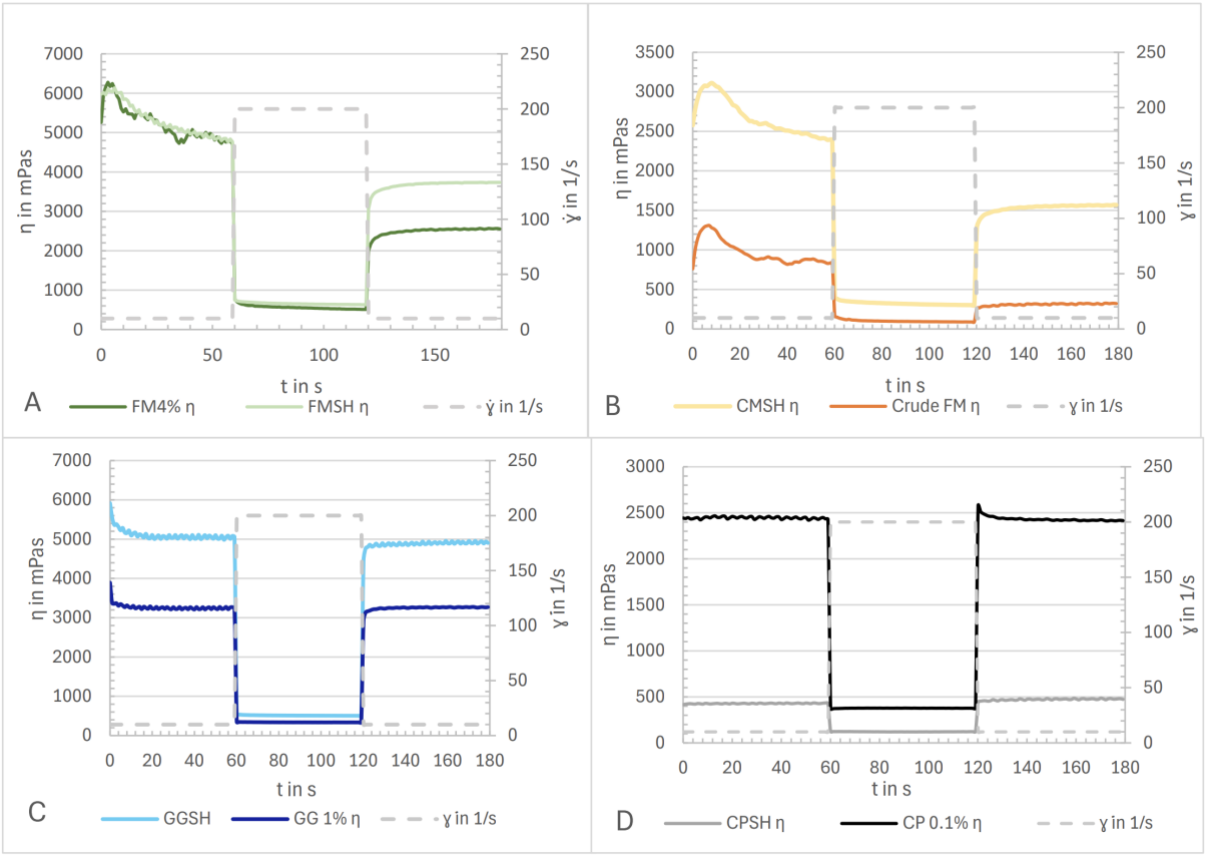

Figure 2. Stepwise shear rate of $10s^{-1}$, $200s^{-1}$,$10s^{-1}$ comparing the recovery of hydrogels with and without NaHA for (A) FMSH and 4%FM; (B) CMSH and CFM; (C) GGSH and GG 1%; (D) CPSH and CP 0.1%. The recovery indicates the level of thixotropy, with higher recovery suggesting lower thixotropic character.

3.3 Texture Analysis

Texture analysis (Table 1) showed that NaHA seemed to have a slight effect on the parameters of the hydrogels as the values had small differences. The FMSH compared to 4% FM had lower firmness, consistency and cohesiveness values, but similar brittleness. The CMSH compared to the crude FM had lower firmness, but higher values for all the other parameters. The GGSH had higher firmness and brittleness, but decreased consistency and cohesiveness. The CPSH values were less than CP across all parameters.

Table 1. Texture analysis parameters firmness or rupture strength, brittleness, consistency, work of cohesion and cohesiveness with ± standard deviation for hydrogels with and without sodium hyaluronate

Sample	Firmness (g)	Brittleness (mm)	Consistency (g/sec)	Cohesiveness (g)
FM	5.11 ± 0.14	7.97 ± 0.04	16.89 ± 0.31	-2.47 ± 0.04
FMH	3.80 ± 0.02	7.90 ± 0.05	13.33 ± 0.05	-1.93 ± 0.01
CFM	4.16 ± 1.51	7.24 ± 0.88	12.13 ± 4.57	-1.46 ± 0.52
CFMH	3.44 ± 0.04	7.99 ± 0.02	13.95 ± 0.25	-1.48 ± 0.03
GG	2.89 ± 0.08	7.19 ± 0.70	13.20 ± 0.60	-1.60 ± 0.83
GGH	3.78 ± 1.12	7.61 ± 0.44	12.45 ± 3.70	-1.04 ± 0.08
CP	4.66 ± 0.13	7.45 ± 0.58	13.35 ± 0.96	-2.04 ± 0.04
CPH	2.84 ± 0.47	4.17 ± 3.20	12.09 ± 4.51	-1.20 ± 0.24

4. Discussion

Hyaluronan has been investigated with other plant-derived polysaccharides such as corn fibre gum, Galactomannans and tamarind seed, revealing synergistic interactions between polymer chains which may have influenced the viscosity and rheology [13-15]. Calculation of synergism index would need to be conducted to confirm synergism between FM polysaccharides and NaHA [16].

Although, the viscosity obtained from a preliminary shear rate sweep revealed low initial viscosity (not shown). This highlighted that the combination of FM with the active, produced higher viscosity than the individual polymers alone, indicating cooperative interactions. Perhaps, the intermolecular interactions, such as hydrogen bonding between polymers chains, influenced the viscosity increase of the hydrogels and their more pronounced shear-thinning behaviour, as seen Figure 2A and 2B. Additionally, the increase in dilation zones of FMSH and CFSH, compared to the hydrogels alone, could be due to interactions of NaHA with other compounds present in FM. One study suggested that interactions with protein contaminants within the mucilage with sodium hyaluronate contributed to the viscoelastic behaviour [17]. As both the crude and rehydrated FM were expected to contain proteins, the interactions of them with the active ingredient could have also influenced the viscosity and rheology of the mixture.

A study [18] has demonstrated synergistic effects of guar gum and NaHA combined, as the mixture had a higher viscosity than both polymers individually. The authors suggested that the synergic associations for galactomannans, were derived from the molecular structure rather than its weight. The findings of the present study seem to align with the literature, as evidenced by the significantly higher viscosity and viscosity curves of GGSH compared to guar gum alone, as shown in Figure 1.

The combination of carbomer (CP) with NaHA had an opposite effect compared to the other hydrogels, as indicated in Figure 1D with a significant decrease in viscosity and less pronounced shear-thinning behaviour. A study [19] investigated the relationship between carbomer and hyaluronic acid and suggested that any deviation from the carbomer and HA ratio of 10:1 (0.1%:0.2%) could lead to antagonistic interactions between structures.

The recovery values of both FMSH and CFMH were higher than the mucilage alone, which implied a decrease in thixotropic behaviour with the addition of NaHA. Moreover, the findings are consistent with prior research showing a reduction in thixotropy upon adding HA to various formulation types. This is likely attributed to the interactions between hydrogel polymer and NaHA chains, which are intensified with high molecular weight NaHA [20, 21]. The controls' non-thixotropy behaviour did not alter with addition of NaHA, but it did increase the low shear viscosity. Research has demonstrated the non-thixotropic nature of hyaluronic acid, indicating that the deformation and recovery of structure occurred in the same intermediate states [22].

Texture analysis is widely employed in food, cosmetics and pharmaceuticals research to quantify mechanical properties and relate them to sensorial properties of products [23,24]. Tafuro et al. [24] highlighted that polysaccharide interactions modify textural properties, and further revealed that specific ratios of two polymers produced values that were intermediate to the individual polymers on their own. FMSH, compared to FM without actives, had lower values for most parameters, apart from brittleness, with its results being negligible in difference. Additionally, the CSMH firmness was lower than crude FM on its own. The results seemed contradictory to the rheology findings of both FM hydrogels previously discussed, as lower firmness implies lower viscosity. Furthermore, the decrease of all parameter values with the addition of NaHA to rehydrated mucilage, indicated that the active reduced viscoelastic properties of the mucilage. This conflicted with the oscillatory results which suggested that NaHA had enhanced the gel network and intermolecular associations. The GGSH findings indicated a lower elasticity to GG, which opposed the conclusions from the oscillatory rheology. CPSH was the only finding where the textural parameters aligned with previous instrumental tests conducted. Overall, the findings seemed to indicate that NaHA interacted with the hydrogels in a manner that increased the viscous and liquid-like properties. However, the differences discussed were very slight, which may be due to the low concentration of the active used. Therefore, the possibility of any sensorial difference being perceived by the consumer is not likely since the variations in parameters were marginal. Nevertheless, the discrepancies within the study indicated the need for further testing.

Future work will aim to gather further data through oscillatory rheology to investigate the mucilage's behaviour further and with other common active ingredients used in cosmetics, as well as dyes used in colour cosmetics. Further, the mucilage's emulsion stabilising properties in cosmetic formulations will be investigated.

5. Conclusion

This section should summarize the main findings and conclusions drawn.
Flaxseed mucilage was shown to exhibit promise for cosmetic applications. The use of crude mucilage provided an interesting comparison for the dried FM, but it was lacking the capability to finetune its physical properties that the dried mucilage had.
The mucilage's increase in viscosity observed with the addition of NaHA suggests favourable interactions between FM and NaHA, potentially indicating synergistic effects between the polymers. This finding opens possibilities for FM to interact positively with other hydrophilic cosmetic actives in topical applications. Texture analysis results implied favourable interactions between NaHA and FM - the decreased firmness and consistency for FMSH indicates better spreadability and application on the skin, as it signifies a lower force required

to reach a set deformation. Further work is needed to asses the mucilage's properties and measure its interactions with other common cosmetic active ingredients.

6. References

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