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Investigate the versatility and dependability of biopolymers in the production of an oil-water (O/W) emulsion

Marlène Lartigue ¹, Claire Dang ¹, Céline Sauré ¹, Séverine Sigurani ¹ and Alicia Roso ^{1,*}

¹ Seppic Research and Innovation, 127 Chemin de la poudrerie, Castres, France

1. Introduction

Synthetic polymers have been extensively studied and optimized for their ease of use [1-4]. These polymers are known for their flexibility in being introduced during the manufacture of an emulsion, without compromising the robustness of their properties. This criterion is still important for formulators as for economic and sustainability considerations a single ingredient can be used in multiple types of formulations and purposes. Many studies have been carried out in recent years to suggest natural polymers that can compete with synthetic polymers in terms of thickening, stabilisation, film formation, and sensory properties [5-8]. Nevertheless, their flexibility in the manufacturing process remains relatively undisclosed. As oil-in-water (o/w) emulsions are a significant component of skin and hair care formulations [9], this work aimed to investigate the impact of the manufacturing procedure of an O/W emulsion using biopolymers from different resources and with various properties (thickening and/or stabilising, film-forming or not).

2. Materials and Methods

2.1. Tested polymers and formulations

Four polymers were investigated in comparison to a synthetic polymer reference in a simple oil-in-water (O/W) emulsion. These included a well-established tara gum (INCI name: Caesalpinia Spinosa Gum), a co-processed stabilizer consisting of xanthan gum coated with acacia gum (INCI name: Acacia Gum and Xanthan Gum), an original glucomannan thickener extracted from the tubers of Amorphophallus muelleri, and an experimental modified polysaccharide (crosslinked xanthan gum). All polymers were in powder form and were incorporated into the emulsion at their respective average recommended dosages (Table 1). Unique identifiers were assigned to each emulsion for subsequent referencing throughout this manuscript.

Table 1. Tested formulations

Ingredients (%w/w)	E-AX	E-Tara	E-Gluc	E-CrossX	E-Ref
Water	Up to 100				
Caprylic/capric triglyceride	10.0	10.0	10.0	10.0	10.0
Glucolipid emulsifier*	3.0	3.0	3.0	3.0	3.0
Acacia Senegal Gum & Xanthan Gum	1.2	-	-	-	-
Caesalpinia Spinosa Gum	-	0.8	-	-	-
Glucomannan	-	-	1.0	-	-
Modified xanthan gum	-	-	-	0.5	-
Synthetic reference**	-	-	-	-	0.5

Preservative***	1	1	1	1
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* Arachidyl Alcohol (and) Behenyl Alcohol (and) Arachidyl Glucoside; ** Hydroxyethyl Acrylate/Sodium Acryloyldimethyl Taurate Copolymer. *** Phenoxyethanol (and) Ethylhexylglycerin.

To assess the versatility of incorporating biobased polymers, three distinct emulsion manufacturing processes were investigated, specifically excluding pre-dispersion. Direct addition of the polymer in powder form to the oil phase, eliminating the need for pre-gel preparation, results in time efficiency. Historical studies have demonstrated no significant impact on the various characteristics of the emulsion when using the studied synthetic polymer. Emulsion manufacturing was performed at a 200g laboratory scale using the same homogenizer. The pH was adjusted to between 5.5 and 7 as needed.

- *Manufacturing process 1, indirect:* The polymer was added to the hot oily phase, followed by the addition of the hot aqueous phase (80°C). Emulsification was done using a rotor-stator turbine at 4000 rpm for 4 minutes. The emulsion cooled at room temperature under an anchor stirrer at 100 rpm for 10 minutes, followed by 10 minutes in a cold water bath, and the preservative was added.

- *Manufacturing process 2, one pot:* All ingredients (oil, emulsifier, polymer) were sequentially added to the hot water (80°C), with mixing using a spatula after each addition. Emulsification was done using a rotor-stator turbine at 4000 rpm for 4 minutes. The emulsification and cooling steps were the same as in Processes 1 and 2.

- *Manufacturing process 3, reduction of water heating:* The polymer was added to the hot oily phase (80°C), followed by the addition of two-thirds of the hot water. Emulsification was done using a rotor-stator turbine at 4000 rpm for 4 minutes. The emulsion cooled at room temperature under an anchor stirrer at 100 rpm for 10 minutes, then the remaining water was added, followed by homogenization using a rotor-stator turbine at 1500 rpm for 2 minutes.

2.2. Emulsion characterization

Emulsion characteristics were assessed: pH, viscosity, stability, and microscopic examination. Rheology experiments were conducted on the different formulations, seven days after manufacturing, using a rotational Controlled stress/strain hybrid rheometer (DHR2; TA Instruments; cone 40 mm/2°).

- Flowing experiments were conducted at 25°C to determine the formulation's yield stress and flow behavior. Yield stress, defined as the minimum stress required to initiate flow (measured in Pascals, Pa), was quantified by applying a shear stress ramp from 0 to 200 Pa over 60 seconds using a steady-state protocol (onset point analysis: (determination by tangent intersection)). The flow profile was characterized by applying a linear shear rate ramp from 0 to 1200 s⁻¹ over 120 seconds, maintaining the maximum shear rate for 60 seconds, and then decreasing the shear rate back to 0 over 120 seconds. The measurements were performed in duplicate. For simplified analysis, the results were presented as viscosity variations as a function of shear rate. Shear thinning behavior was quantified using the Herschel-Bulkley model for formulations exhibiting a yield stress, and the Carreau-Yasuda model for those without a yield stress (a gradient index closer to zero indicates more pronounced shear-thinning behavior, while an index closer to 1 suggests behavior approaching that of a Newtonian fluid).

- Oscillatory rheology experiments were conducted to characterize the viscoelastic properties of the formulations and to assess the influence of the manufacturing process on their microstructure. Initially, the structural properties were evaluated at 25°C under low shear stress conditions (0 to 100 Pa at a fixed oscillation frequency of 1 Hz). For formulations exhibiting significant viscoelastic behavior, supplementary temperature ramp experiments

were performed (from +5°C to 80°C at a fixed 1 Hz frequency, using an anti-evaporation cap) to determine the thermal stability of the structure. The storage modulus G' (representing elastic behavior) and the G'/G" ratio (average value within the linear domain, where G" is the loss modulus representing viscous behavior) were monitored as indicators of the formulation's structure. A higher G'/G" ratio indicates a more structured product. Results were expressed as average value in the linear domain (from duplicate measurements).

3. Results

3.1. Impact of processing conditions on emulsion texture, viscosity, and particle size

The texture (Figure 1) and viscosity (Figure 2) of the emulsions depended on the selected polymer, as suggested by their nature and different dosage, but no significant differences were observed between the manufacturing processes tested. Emulsions exhibiting the most consistent texture were produced with glucomannan (E-Gluc) and the synthetic reference (E-Ref), correlating with their respective viscosity values.

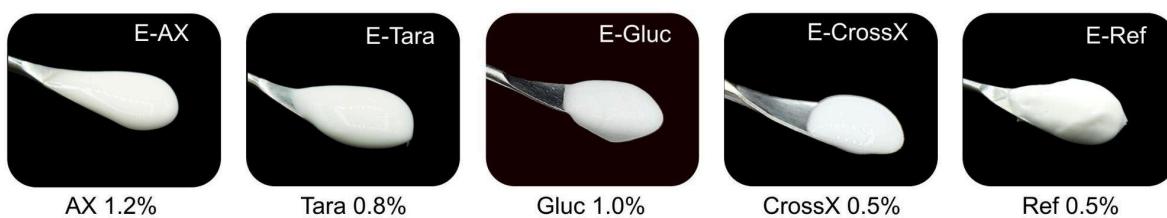


Figure 1. Emulsions texture, example with manufacturing procedure n°1.

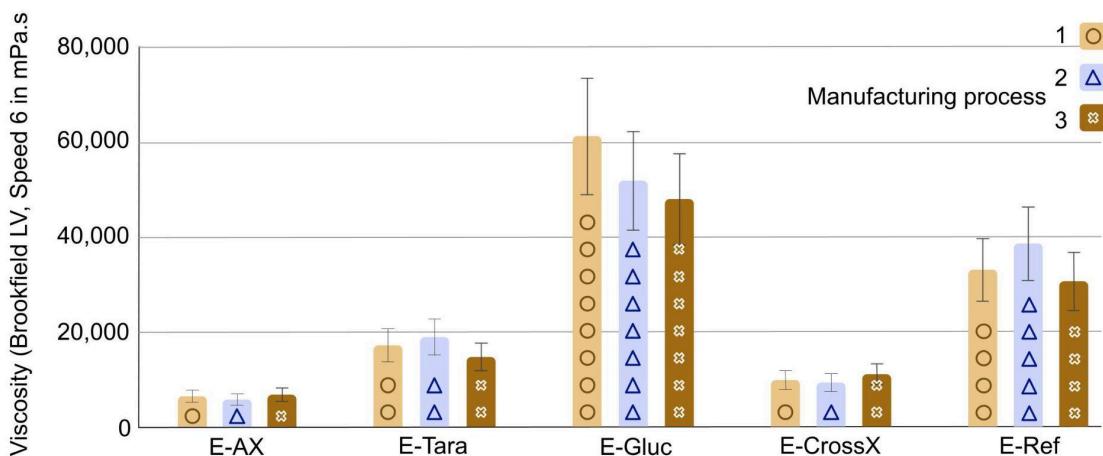


Figure 2. Viscosity of emulsions as a function of polymer and manufacturing procedure.

All the emulsions were stable after one month of storage at room temperature and 45°C and did not impact the formation of liquid crystals visualized by maltese cross under polarized light (Figure 3). Emulsions produced with tara gum (E-Tara) and with the synthetic control (E-Ref) showed similar particle size with the different manufacturing processes. For the other natural polymers, emulsions were more or less thin depending on the process and the polymer nature, associated with variations in the liquid crystals visualization. As expected, thinner particles related to less visible liquid crystals as maltese cross represent bilayers around the oil droplets. The emulsions produced with the co-processed acacia gum/xanthan gum (E-AX) were thinner with the two processes in which the polymer is introduced in the oil (process 1 and 3). For glucomannan, the emulsions (E-Gluc) were thinner with process 1 (indirect) and 2 (One pot) whereas for the experimental crosslinked xanthan gum, process 2 (One pot) and 3 (step with concentrated emulsion) provided thinnest emulsions (E-Cross X).

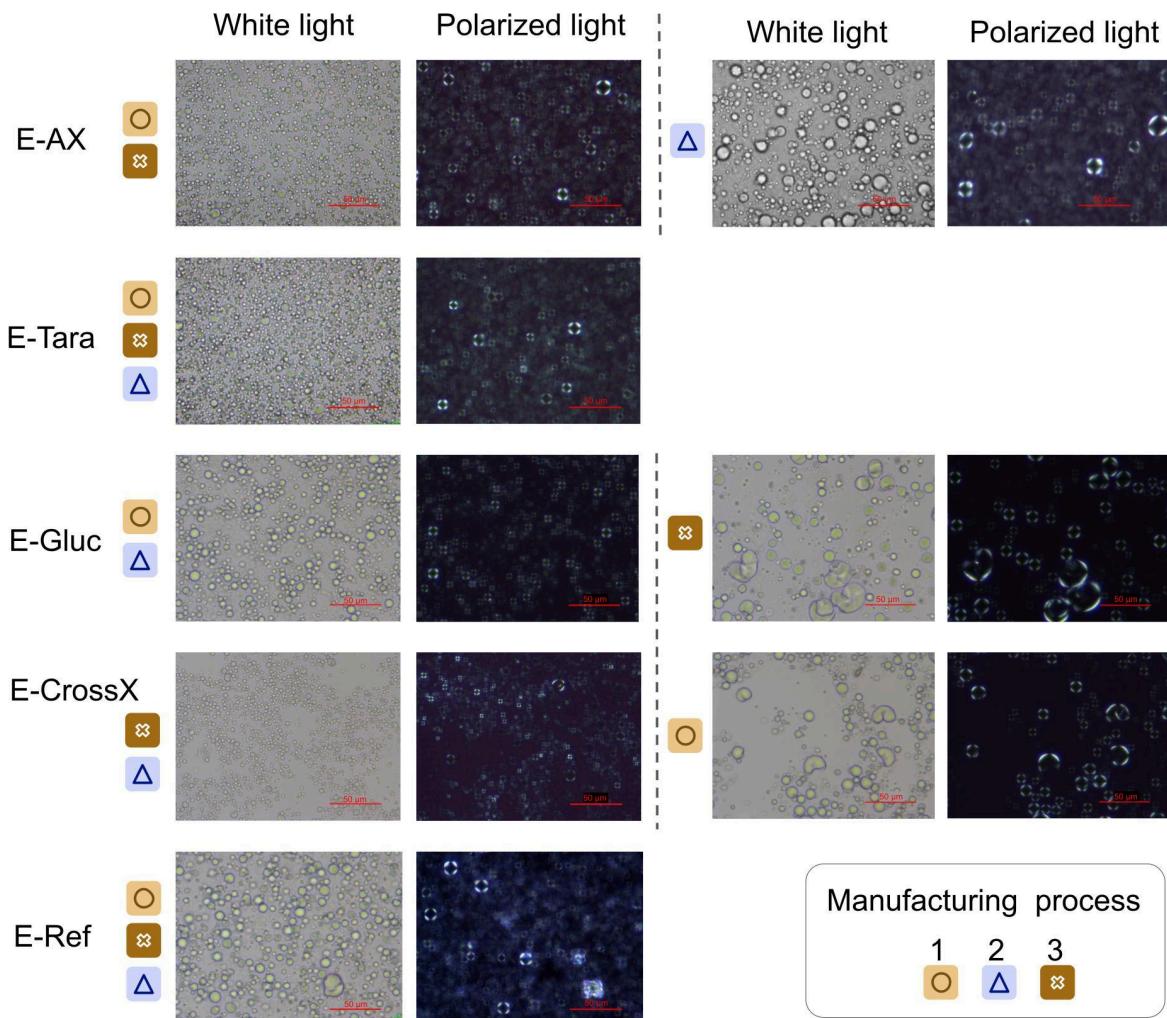


Figure 3. Microscopic appearance of emulsions as a function of polymer and manufacturing procedure (white light and polarized light at 40x magnification).

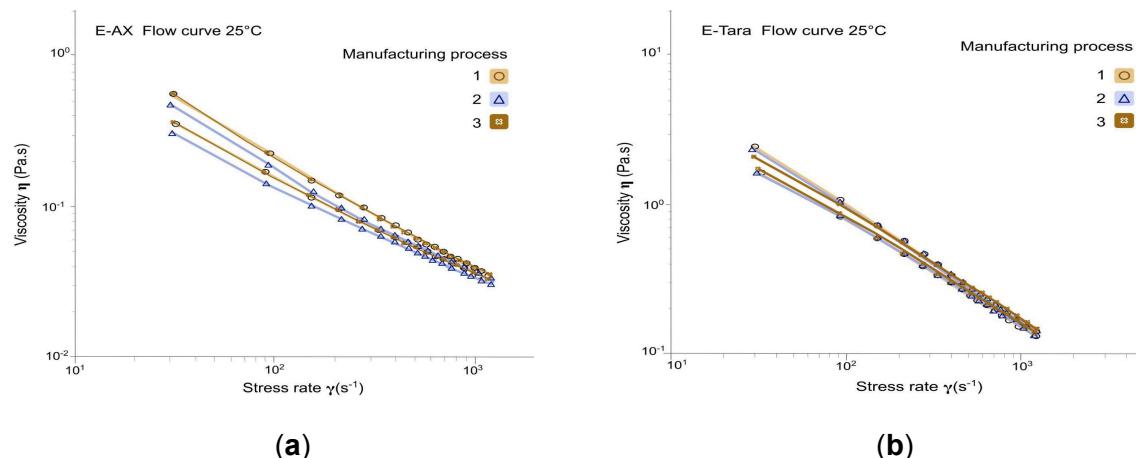
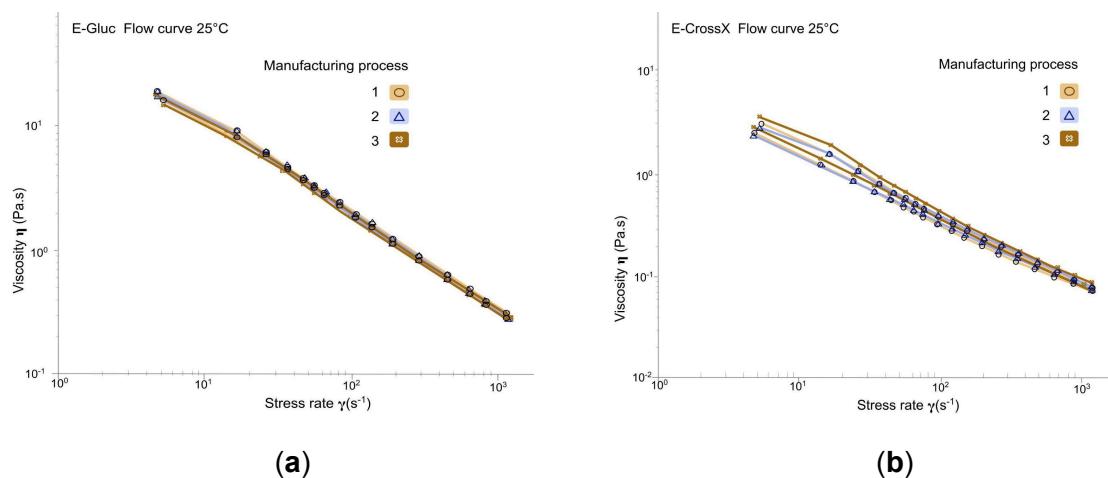
3.2. Impact of processing on emulsion flow properties

The superposition of flow curves for emulsions obtained with different manufacturing processes for each polymer (Figures 4 to 6) demonstrated that the emulsion flow behavior was not significantly affected by the process, similar to the synthetic polymer reference, with the exception of emulsions containing the experimental crosslinked xanthan gum (E-CrossX). In the latter case, processes 2 (one-pot) and 3 (step with concentrated emulsion) resulted in slightly reduced yield stress and an increased shear-thinning characteristic.

Regardless of the manufacturing process, the shear-thinning behavior of the emulsion and the presence or absence of a yield stress varied depending on the chosen polymer. Emulsions obtained with the co-processed acacia gum/xanthan gum (E-AX) and glucomannan (E-Gluc) exhibited shear-thinning behavior similar to the emulsion with the synthetic polymer, but they had a nearly non-existent yield stress. The emulsion produced with tara gum (E-Tara) showed a much more pronounced shear-thinning behavior, while the emulsion with the crosslinked xanthan gum (E-CrossX) exhibited a more Newtonian tendency. With the exception of the glucomannan-based emulsion (E-Gluc), all tested emulsions demonstrated slight thixotropic behavior, evidenced by the hysteresis observed between the ascending and descending viscosity-shear rate curves.

Table 2. Impact of manufacturing process on flow curve depending on the polymer nature

Formula	Flow curve analysis	Process 1	Process 2	Process 3
E-AX	Yield stress (Pa)	3	3	3
	Gradient index	0.5	0.6	0.6
E-Tara	Yield stress (Pa)	3	3	3
	Gradient index	0.1	0.1	0.1
E-Gluc	Yield stress (Pa)	None	None	None
	Gradient index	0.8	0.8	0.8
E-CrossX	Yield stress (Pa)	23	12	16
	Gradient index	0.7	0.4	0.5
E-Ref	Yield stress (Pa)	11	17	14
	Gradient index	0.4	0.4	0.4

**Figure 4.** Flow curves across manufacturing processes: (a) Emulsions with co-processed acacia gum/xanthan gum: E-AX; (b) Emulsions with tara gum: E-Tara.**Figure 5.** Flow curves across manufacturing processes: (a) Emulsions with glucomannan: E-Gluc; (b) Emulsions with crosslinked xanthan gum: E-CrossX.

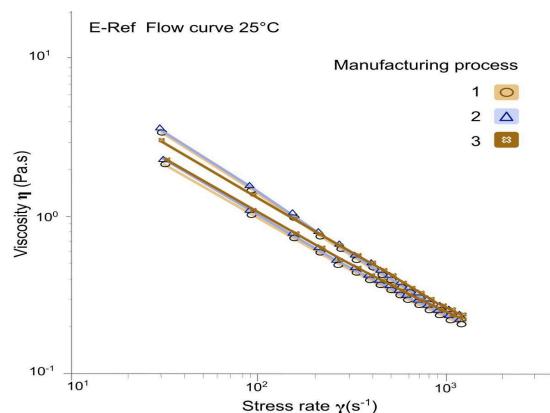


Figure 6. Flow curves of synthetic polymer emulsions across manufacturing processes.

3.2. Impact of processing on emulsion viscoelastic properties

To facilitate comparison of manufacturing process influences, the viscoelastic properties of the emulsions were characterized by the average storage modulus (G') and the G'/G'' ratio within the linear viscoelastic region. Higher values for both parameters indicate a more pronounced elastic character of the emulsion. The combined analysis of these parameters allowed for relative comparisons (Figure 7). A G'/G'' ratio exceeding 2 suggests a dominant elastic character, which is further substantiated by the absolute value of G' .

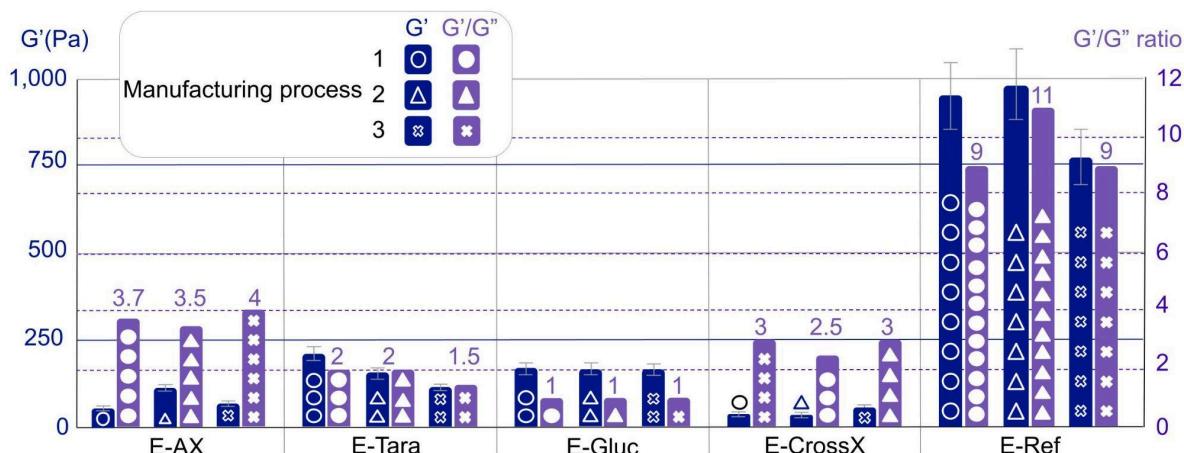


Figure 7. Viscoelastic properties across manufacturing processes and polymers.

Overall, the process variations did not change the emulsion structure and its response to low oscillatory stress. Emulsions containing glucomannan (E-Gluc) did not display a distinctive elastic character, regardless of the manufacturing process, which is consistent with its function as a thickener in aqueous dispersions. Emulsions with tara gum (E-Tara) showed a similar trend, although with slightly elevated G'/G'' ratios.

Although not reaching the same level as the synthetic polymer reference (E-Ref), emulsions with co-processed acacia gum/xanthan gum (E-AX) and crosslinked xanthan gum (E-Cross X) imparted dominant elastic character and thus brought structure to the emulsion. However, the curves for the E-AX emulsion indicated that manufacturing process 2 (one-pot) should be avoided due to the observed reduction in the linear viscoelastic region (Figure 9), which may compromise its stabilizing efficacy, particularly in applications requiring particle suspension.

Notably, E-CrossX exhibited an elastic character comparable to E-AX, despite being used at half the polymer concentration.

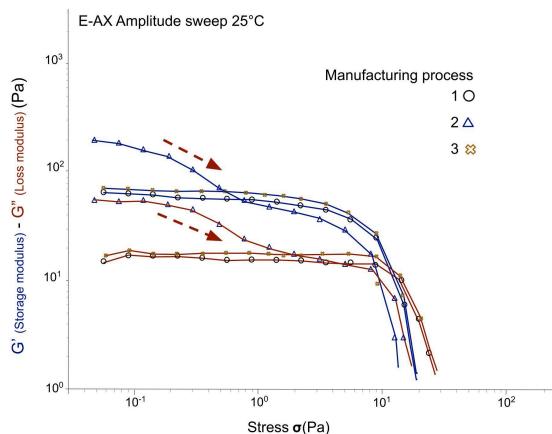


Figure 8. Impact of manufacturing process 2 on the linear viscoelastic region.

To assess structural stability, temperature ramp experiments were performed from 5°C to 80°C on emulsions exhibiting dominant elastic character (E-AX, E-CrossX, and E-Ref). All tested emulsions maintained structural integrity up to 55-60°C (Figures 9 and 10), supporting their thermal stability. This temperature range aligns with the initial melting point of the waxy emulsifier above 60°C. Figure 9a confirmed that process 2 is less favorable for the co-processed acacia/ xanthan gum.

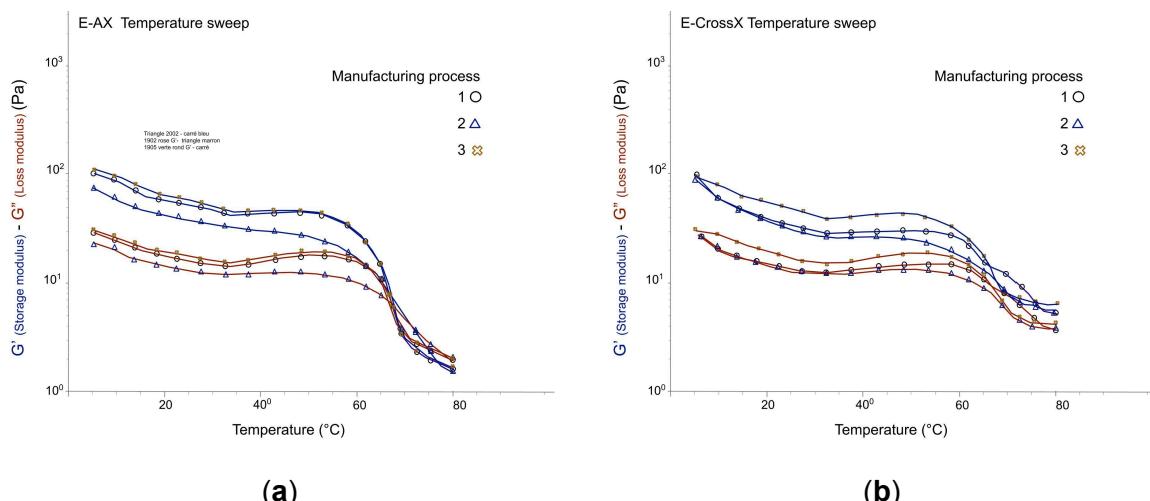


Figure 9. Stability of the elastic structure across temperature variations from 5°C to 80°C: (a) Emulsions with co-processed acacia gum/xanthan gum: E-AX; (b) Emulsions with crosslinked xanthan gum: E-CrossX.

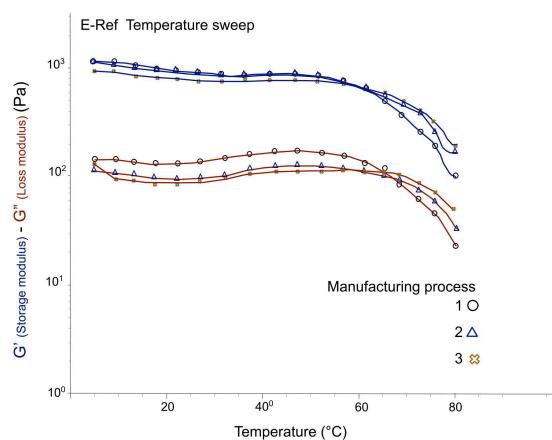


Figure 10. Stability of the elastic structure across temperature variations from 5°C to 80°C: emulsions with the synthetic control.

4. Discussion

Although traditional manufacturing processes for emulsions often involve dispersing natural polymers in water or polar co-solvents like glycols before emulsification [10-12], this study demonstrates that alternative manufacturing processes can be employed with minimal impact on emulsion characteristics. While certain processes yielded finer microscopic appearances, a direct correlation with emulsion stability could not be conclusively established at this stage. Further long-term stability testing is necessary.

Oscillatory rheology experiments provide an indication of potential emulsion stability, with established correlations for O/W emulsions [13-15]. Despite the level of emulsion structuring being generally lower compared to the synthetic polymer reference, oscillatory rheological data suggests no significant stability differences between the tested manufacturing processes, with the exception of the acacia/xanthan gum co-processed emulsion prepared using process 2 (E-AX). In this specific case, a coarser microscopic appearance was associated with reduced emulsion structuring and diminished resistance of the microstructure to temperature variations, potentially leading to decreased long-term stability.

Flow rheological profiles also offer insights into emulsion behavior under stress conditions, such as those encountered during packaging filling or application to the skin [16-19]. For the experimental crosslinked xanthan gum, processes 2 and 3 resulted in thinner emulsions with lower yield stress and higher shear thinning characteristics, which could be advantageous for facilitating pouring from packaging with narrow apertures. For the other biopolymers, the observed lack of difference between manufacturing processes suggests minimal impact. However, real-world usage tests are required to validate these conclusions.

These findings offer a promising approach for developing more sustainable manufacturing processes using biopolymers. Notably, manufacturing process 3, which involves partial heating of the aqueous phase, has been previously proposed as a method for reducing energy consumption [20]. Heating and cooling phases were reported to account for 95% of the total energy utilized during emulsion manufacturing, whereas mechanical homogenization and mixing only comprise 5%. A recent study from 2023 [21] further demonstrated through energy calculations that this process (called the “hot-cold” method) can save approximately 82% of thermal energy for oil-in-water emulsions, significantly reducing the carbon footprint of the final product. Previous work has shown the effectiveness of this method with xanthan gum as a natural polymer; the current study expanded the interest to other biopolymers. The one-pot process, process 2, has primarily been described in the synthesis of various materials [22], such as nanocrystals, nanoparticles, microcapsules, and microspheres, as

well as in the preparation of Pickering emulsions and high internal phase emulsions [23-24]. However, the simplicity and potential sustainability of this approach, particularly its ability to eliminate the separate heating of the oily phase in oil-in-water emulsions, has been highlighted. From an economic perspective, this manufacturing protocol has the potential to decrease emulsion processing duration and may enable the use of simplified industrial equipment, avoiding the need for auxiliary melting vessels.

This study was conducted at a laboratory scale with limited quantities. Larger-scale emulsion trials are necessary to confirm these findings and verify industrial feasibility. Future work could investigate the applicability of these manufacturing procedures to cold processable emulsions using appropriate liquid emulsifiers. Future research directions could also encompass the investigation of more complex formulations incorporating active ingredients or cleansing surfactants.

5. Conclusion

This study demonstrated globally the flexible use of the tested biopolymers in emulsion formulations. Alternative manufacturing processes, including one-pot and reduced-heating methods, can be effectively employed with these biopolymers without significantly compromising emulsion characteristics, supporting sustainability goals. While some minor differences in microscopic appearance and rheological properties were observed depending on the polymer and process, the overall impact on emulsion stability was expected as minimal, with the exception of the co-processed acacia gum/xanthan gum in the one-pot process. The rheological data of the emulsions revealed a variety of profiles, including varying degrees of shear thinning, elastic behavior, and the presence or absence of yield stress. This diversity can be used to identify the most suitable biopolymer or combination of biopolymers to achieve desired texture and application properties. Larger-scale tests are needed to confirm industrial viability of these biopolymer-based emulsion manufacturing methods.

6. References

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