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Sustainable Marine Biopolymer Gels for Active, Biodegradable Film Formation

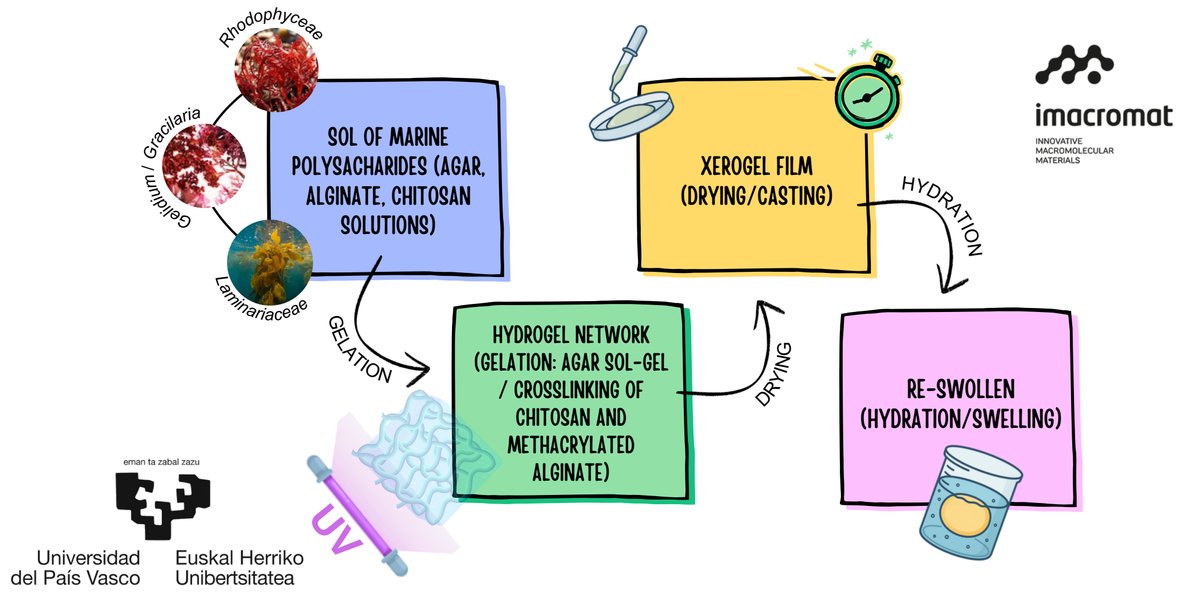
Andrés Felipe Vélez Linares (andresfelipe.velez@ehu.eus)[1], Beatriz Gutiérrez Portal (bgutierrez022@ikasle.ehu.eus)[1], Leire Ruiz Rubio (leire.ruiz@ehu.eus)[1,2], Leyre Perez Alvarez (leyre.perez@ehu.eus)[1,2], Jose Luis Vilas Vilela (joseluis.vilas@ehu.eus)[1,2]

1. Innovative Macromolecular Materials (Imacromat), Physic-Chemistry department, Basque Country University UPV/EHU, 48940 Leioa, Spain

2. Basque Center for Materials, Applications and Nanostructures, UPV/EHU Science Park, 48940 Leioa, Spain

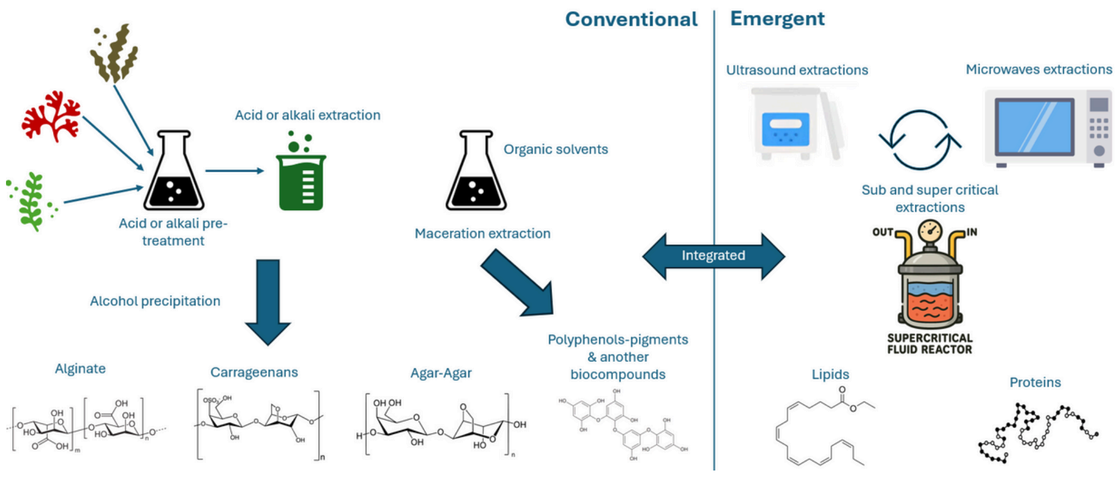
INTRODUCTION & AIM

Replacing fossil-based plastics in the food sector demands sustainable materials with tunable structure–function relationships. Marine polysaccharides (agar, alginate, carrageenan, and chitosan) form hydrogels that, upon drying, yield functional xerogels (films) capable of re-swelling and regaining gel-like behavior. Our approach integrates sustainable extraction, methacrylation, and β-cyclodextrin inclusion to tailor network architecture and add functionality. Aim: to establish a gel→xerogel pathway to active, biodegradable films, validating network structure by FTIR/DSC and re-hydration behavior by swelling assays, as a foundation for food-packaging applications.



METHOD

Beachcast macroalgae were collected by tidal action and processed within 48 h; the initial phase used red algae (Gelidium sesquipedale), with planned expansion to brown/green macroalgae and microalgae. Polysaccharides were recovered via alkaline pretreatment followed by hot-water extraction or ultrasound-assisted maceration; phenolic fractions were isolated by ethanolic maceration and quantified by Folin–Ciocalteu. Agar, alginate, and chitosan were methacrylated to tailor network formation. Films were prepared by inducing gelation (agar sol–gel cooling; network setting in methacrylated matrices) and drying to obtain xerogels. Structure and complexation were assessed by FTIR and DSC (including β-cyclodextrin complexes with curcumin/carvacrol), and water interaction by swelling assays.



Biomass was rinsed with deionized water, dehydrated at 40 °C, and milled (≤2–5 mm) prior to extraction. Working polymer solutions (agar, alginate, chitosan; native or methacrylated) were prepared in water with glycerol as plasticizer and, when indicated, with β-cyclodextrin–curcumin/carvacrol inclusion complexes obtained by complexation and solvent evaporation. Methacrylation proceeded via persulfate-initiated radical chemistry and was verified by FTIR; DSC verification is underway. Film formulations were cast onto leveled trays, gelation was induced (agar sol–gel cooling; network setting in methacrylated matrices), and controlled drying yielded xerogels of uniform thickness. Swelling assays are ongoing and will be quantified as mass/area gain after immersion with standardized blotting; structural characterization will be complemented with DSC (thermal transitions and inclusion evidence) in the next stage.

RESULTS & DISCUSSION

Extraction

Ethanolic maceration yielded polyphenolic extracts (Fig. a); the chromatogram (Fig. b) confirms a defined profile, with Folin–Ciocalteu values of 0.0925 mg GAE·g^{-1} (early batch) and 10.49 mg·L^{-1} (later protocol; R^2 = 0.992). Conventional agar extraction reached 0.80 g from 5 g dry biomass vs 0.50 g by ultrasound; the ultrasound agar showed slight green coloration (Fig 1. c), to be mitigated via clarification/purification during scale-up.

Figure 1. From left to right: a) Polyphenols extracted using the conventional method. b) Chromatogram of polyphenols. c) Agar extracted using the conventional method and assisted by ultrasound (green coloration).

Film formation

The gel→xerogel route yielded uniform agar films and improved handling/cohesion for methacrylated alginate and chitosan. FTIR across the three methacrylated matrices shows the C=O band at ~1720–1740 cm^{-1} and changes within 1100–1300 cm^{-1} (C–O), confirming network modification. Visually, films exhibit good surface integrity; methacrylated systems better withstand handling and release after drying.

Figure 2. From left to right: a) Methacrylated agar film with lignin as UV protection. b) Agar film with lignin and glycerol as plasticizer. c) Methacrylated agar film with glycerol as plasticizer.

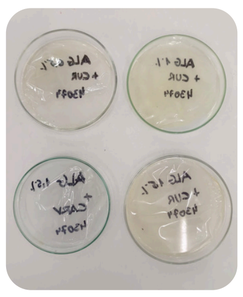


Figure 3. Methacrylated alginate films at different concentrations with betacyclodextrin-curcumin complex as an additive.

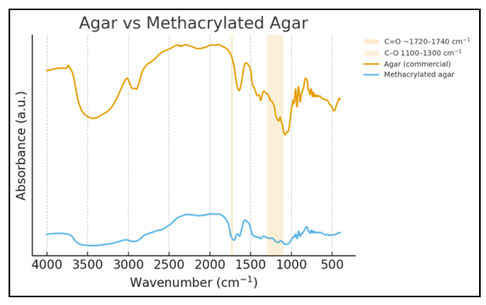


Figure 4. Comparison between commercial agar and methacrylated agar.

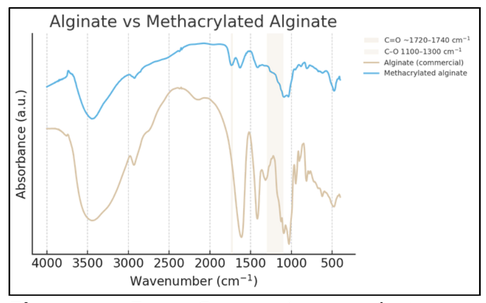


Figure 5. Comparison between commercial alginate and methacrylated alginate.

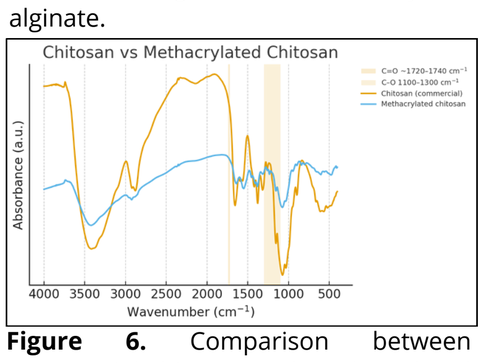


Figure 6. Comparison between commercial chitosan and methacrylated chitosan.

CONCLUSION

We validated a gel→xerogel pathway from marine polysaccharides, yielding structurally robust, re-swelling films. FTIR confirmed network tailoring (methacrylation) and β-cyclodextrin inclusion. Initial extractions and yields were reproducible. Next steps will quantify barrier and mechanical properties, alongside bioactivity and biodegradability, toward active, biodegradable food-packaging applications.

ACKNOWLEDGEMENTS

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