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Microfluidized hydrogels and emulgels structured with dietary fiber: influence of processing on physical properties

D. Mammolentia, E. Brunoa, F.R. Lupia, N. Baldinoa, M.C. Garcíab, J. Muñozb, D. Gabrielea

^aDepartment of Information, Modeling, Electronics and Systems, (DIMES) University of Calabria, Via P. Bucci, Cubo 39C, 87036, Rende, Italy beginning begin

INTRODUCTION & AIM

The usage of dietary fibers from food processing by-products is receiving a growing interest because of its health benefits, texturing and stabilization ability [1]. Under mechanical shear stress, citrus fiber aqueous suspension typically gives rise to (particle) hydrogel or emulgel, if oil phase is present [2]. Technology and process condition affects rheology, microstructure and stability of such system [2]. Microfluidization is a very attractive and effective homogenization technique for processing food systems. The effects microfluidization channel types (Z-z chamber series and y-Z chamber series) and number of cycles (1 and 2) on rheology and microstructure of both hydrogel and emulgels was faced. In particular, rheological properties connected to strength, deformation and consistency were evaluated by dynamic oscillatory shear tests and particle and droplets size distribution were determined by laser diffraction. Morphology was investigated by optical and confocal microcopies. Finally, physical stability was assessed by multiple light scattering.

METHOD

High Shear Homogenization (4000 rpm, 1 min) + Microfluidization (172 MPa)



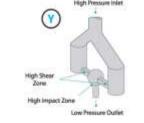
Rotational rheology - Haake Mars 40 (Thermo Scientific, USA):

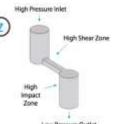
- ➤ Stress sweep test (0.01 100 Pa, 1 Hz);
- ➤ Frequency sweep test (0.01 10 Hz, LVR).

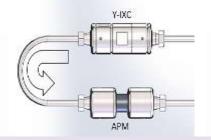
Microstructure:

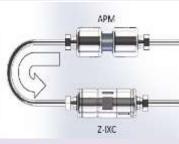
- Laser diffraction-Mastersizer 2000 (Malvern, USA);
- Optical microscopy Axio Scope A1 (Carl Zeiss, Germany);
- CLSM Stellaris 8 (Leica microsystem, Germany).

Sample ID	n° of cycles	Channel configuration	Channel diameters (µm)	
H_C1	1	APM-z	200-87	
H_C2	2	APM-z + APM-z		
H_C3	1	APM-z	75-200	
H_C4	2	APM-z + APM-z		
E_C1	1	APM-z	200-87	
E_C2	2	APM-z + APM-z		
E_C3	1	APM-z	75.000	
E_C4	2	APM-z + APM-z	75-200	









$$\sigma_L = \left. au \right|_{G' = 0.95 \, G'_{LIN}}$$
 Limit stress

$$\gamma_L = \gamma \Big|_{G'=0}$$

Limit strain (-)

Critical strain (-)

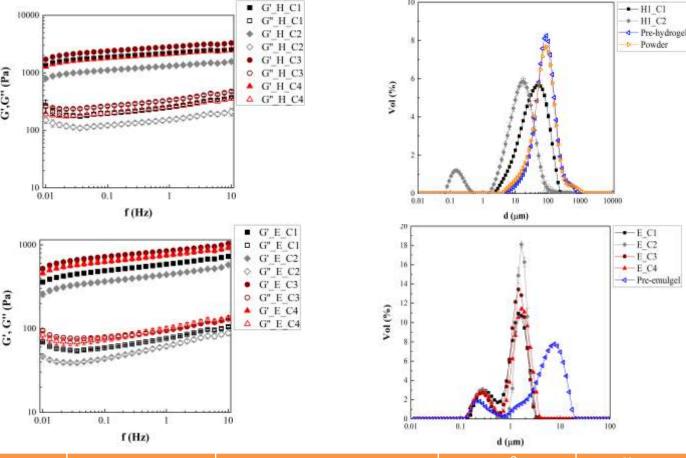
$$au_c = au \Big|_{G' = G''}$$
 Critical stress (Pa) $au_c = au \Big|_{G' = G''}$

G''_{min}

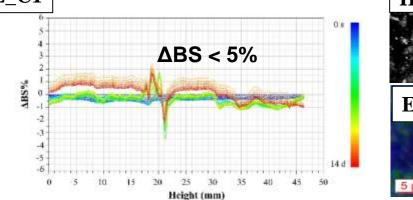
Minimum viscous modulus (Pa)

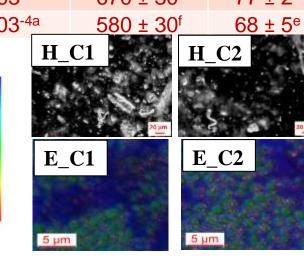
RESULTS & DISCUSSION

- Hydrogels and emulgels showed weak gel behavior.
- The plateau condition is located at approximately 0.315 Hz;
- Particle size distribution is notably affected by processing cycles whereas droplets size distribution is scarcely affected.



Sample	$ au_L$	γ_L	G_N^{0}	$G^{\prime\prime}_{min}$
ID	(Pa)	(-)	(Pa)	(Pa)
H_C1	22.1 ± 0.5^{a}	0.01138 ± 0.00001 ^a	1720 ± 20 ^a	178 ± 9 ^a
H_C2	14 ± 1 ^b	0.012 ± 0.001 ^a	1020 ± 40 ^b	110 ± 9 ^b
H_C3	$30.33 \pm 0.02^{\circ}$	0.0120 ± 0.0004 ^a	2060 ± 90°	230 ± 10 ^c
H_C4	11 ± 2 ^b	0.010 ± 0.001a	1550 ± 50 ^d	176 ± 7 ^a
E_C1	5 ± 1 ^{de}	0.010 ± 0.002^{a}	403 ± 10 ^e	46 ± 1 ^d
E_C2	2.5 ± 0.2^{e}	0.0092 ± 0.0007^{a}	330 ± 20^{e}	39 ± 1 ^d
E_C3	6.889 ± 0.002^d	0.009 ± 0.003^{a}	670 ± 30^{f}	77 ± 2 ^e
E C4	3.6 ± 0.2^{e}	0.010 ± 0.0003 ^{-4a}	580 ± 30^{f}	68 ± 5 ^e
E C1		н	С1 Н	C2





CONCLUSION

- A second cycle generates a second population of small fiber particles (microfibrils) in both hydrogel and emulgel.
- $\succ \tau_L$, G_N^0 and G''_{min} decrease with a second cycle.
- \triangleright γ_L is independent from process conditions and type of systems.
- For all sample, the second cycle yields to weaker systems but with the same limit strain.
- The decrease in strength (τ_L) and elasticity (G_N^0) found for hydrogel and emulgel can be associated to the hindering action of microfibrils and fragments of microfibrils toward fiber network.

REFERENCES

- [1] Pathania S.; Kaur N., Utilization of fruits and vegetable by-products for isolation of dietary fibres and its potential application as functional ingredients, Bioactive Carbohydrates and Dietary Fibre 27 (2022) 100295.
- [2] Bruno, E.; Lupi, F.R.; Mammolenti, D.; Baldino, N.; Gabriele, D. Development and Rheological Modeling of Dietary Fiber and Policosanol Plant-Based Bigels for Potential Food Applications. Food Hydrocoll. 2024, 150, 109733