Novel NUTRAVA^{TM} Citrus Fiber and its ability to Stabilize Emulsions

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ABSTRACT

Novel NUTRAVA^{TM} Citrus Fiber, ("CF" or "Citrus Fiber") developed by a patented process,[1] shows unique properties and can stabilize water-in-oil emulsions even when subjected to centrifugation. The emulsions appear visually stable and maintain structure (G') and viscosity when evaluated over several months.

NUTRAVA^{TM} Citrus Fiber works via dual function, using the combination of the emulsifying properties of the inherent pectin together with the stabilized fiber matrix to provide stable oil-in-water emulsions. It was possible to prepare stable emulsions with 20, 40 or 60 % rapeseed oil and 1-1.6 % Citrus Fiber.

INTRODUCTION

Food producers are looking for clean label possibilities[2], including "good for you" benefits, lower number of ingredients, simpler, more consumer-friendly and sustainably sourced ingredients for their products, while still getting the required functionalities like stabilization, suspension, texture/mouthfeel, taste and flavour perception.

We have developed NUTRAVA^{TM} Citrus Fiber (CF) which combines providing an ingredient for use in clean labelling products with functionality. CF is a combination of soluble and insoluble dietary fibers, and fits into the market trend of functional ingredients for use in clean label food products.

By nature, citrus peel has high pectin content, which is retained during our patented process[1]. In the following, we refer to this as *inherent pectin*. During the CF manufacturing process, the cellulose is "opened up" by mechanical defibrillation, which reduces the pore size, providing large surface area. With large surface area, cellulose fibers tend to self-associate, leading to a decrease in surface area and thereby reduced end-use functionality, but the inherent pectin in CF helps inhibit the self-association of the cellulose fiber, thereby ensuring a functional CF.

Furthermore, pectin is known to have emulsifying properties[3] and the combination of inherent pectin and the cellulose matrix was investigated for its ability to stabilize emulsions.

METHODS AND MATERIALS

<u>1.0-1.6 % CF in 20, 40 or 60 % oil-in-water emulsion:</u>

Weigh off 200, 400 or 600 g Rapeseed oil (Kokkens Catering, DK) into a 2000 ml plastic beaker, make a slurry by adding 10, 12, 14 or 16 g NUTRAVA^{TM} Citrus Fiber (CP Kelco ApS, Lille Skensved, Denmark) stirring at 500 rpm using a propeller mixer for 1 minute. Add 3 ml 20 % potassium sorbate (CAS no. 24634-61-5), 4 drops antifoaming agent (AFE-1510. dimethyl polysiloxane), and cold deionized water (to reach 1000 g) while continuing to mix for 1 minute at 500 rpm. While mixing for an additional 5 minutes, adjust pH to 4.2 (+/- 0.2) using 50 % w/v citric acid (CAS no. 5949-29-1) solution.

Move the beaker to Silverson L5M-a (Silverson Machines, Inc., MA, USA) shear at 7500 rpm for 10 minutes and fill into containers.

Rheological test:

Structure (G' & G") and viscosity of the samples were measured at 23ºC with an Anton Paar MCR 101 rheometer (Anton Paar GmbH, 8054 Graz, Austria) equipped with a cylindrical geometry (CC27).

After loading the sample into the rheometer, allow 2 minutes of waiting time before measuring is started.

Time sweep: time = 300 s, strain = 0.5 %, frequency = 1 Hz (10 measuring points, measuring point duration = 30 s), G' after 300 s is extracted. Flow curve: Shear rate 0.1 … 1000 s^{-1} (log distributed), 21 measuring points (linearly distributed), Shear rate 1000 … 0.1 s^{-1} (log distributed), 21 measuring points (linearly distributed).

Stability test:

Add 10 g emulsion to centrifuge tube, centrifuge at 4400 g for 10 minutes on Hettich Rotana 460R (Andreas Hettich GmbH & Co., Tuttlingen, Germany). Visually evaluate whether emulsions remain stable or separate, Figure 1.

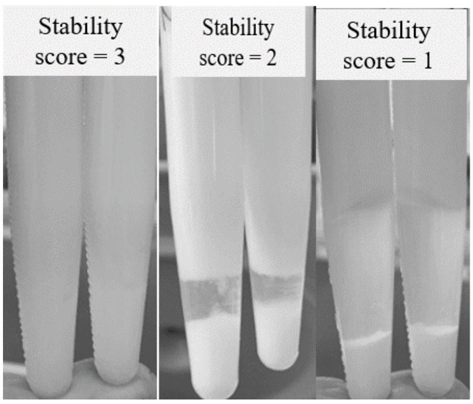
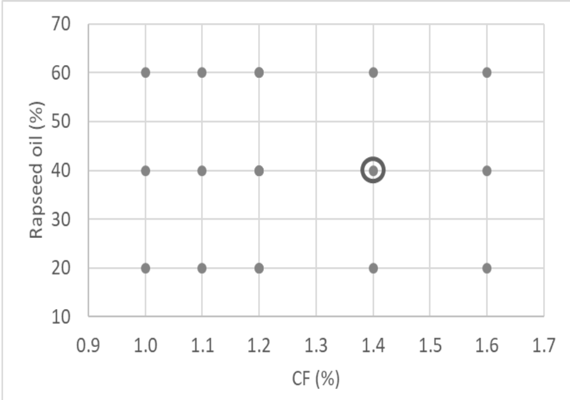


Figure 1. Visually evaluated stability after centrifugation (no separation = 3).

MODDE design:

MODDE (version 12.1 from Sartorius Stedim Biotech, Goettingen, Germany).



MODDE design illustrated in Figure 2.

Figure 2. MODDE design showing the combination of CF use level and oil content in the emulsion (dots), center point illustrated by circle.

RESULTS

When using CF, it is important to activate the CF; different high shear approaches can be used. For these emulsions we use a Silverson equipped with an emulsion screen both to activate the CF and to create the emulsion. As seen in Table 1, it is possible to prepare 40 % water-in-oil emulsions stabilized with 1.2 % CF reproducibly when activating with a high-speed mixer.

Table 1. The structure (G' & G") and viscosity at a shear rate of 100 s^{-1} of 40 % oil-in-water emulsions prepared with 1.2 % CF.

	G' (Pa)	G" (Pa)	visc. (mPas)	Stabil. Score
1	133	56	906	3
2	127	54	914	3
3	132	54	949	3
Average	130	55	923	3
Stdv	3	1	23	0
%stdv	2.5	1.9	2.5	0

The elastic modulus of 40 % oil-in-water emulsions stabilized with 1.2 % CF is 130 Pa and higher than G” (55 Pa) clearly showing that the CF creates a network of particulates that spans the container giving this pronounced elastic structure.

In order to evaluate the stability of the emulsions they are subjected to centrifugation. A stable 1.2 % CF in 40 % oil-in-water emulsion is shown in Figure 3. It is evident that even when strained the emulsion can keep the oil droplets entrapped in the fiber network.

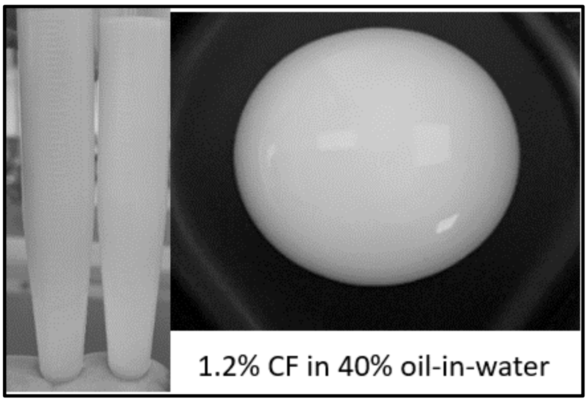


Figure 3. 1.2% CF in 40 % oil-in-water, a stable emulsion (photographed) and tube after centrifugation (stability score = 3).

The emulsions are primarily showing elastic properties as seen in Figure 4 where G’ and G” are shown for the time sweep for selected emulsions. Emulsions are very stable and only insignificant increase in structure is observed over the measured time period.

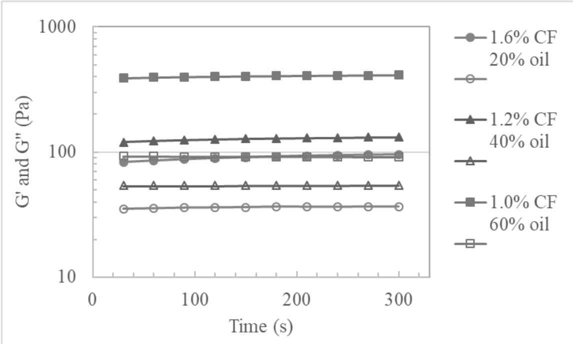


Figure 4. Time sweep of G’ (filled markers) and G” (open markers) of 1.6% CF in 20% oil, 1.2% CF in 40% oil and 1.0% CF in 60% oil-in-water emulsions.

Table 2. The structure (G’ & G”), viscosity at a shear rate of 100s^{-1} and particle size D[4,3] of the emulsions prepared.

CF (%)	Oil (%)	G' (Pa)	G" (Pa)	Visc., (mPas)	Stabil. score
1.4	60	551	141	3050	3
1.0	40	100	37	690	2
1.2	60	524	127	2425	3
1.2	40	133	56	906	3
1.1	40	123	49	811	3
1.1	20	60	19	288	1
1.0	60	411	91	1755	3
1.2	40	127	54	914	3
1.2	20	62	21	336	1
1.4	40	177	75	1300	3
1.0	20	43	14	240	1
1.2	40	132	54	949	3
1.1	60	462	105	2135	3
1.4	20	90	33	486	2
1.2	20	50	19	315	1
1.2	60	545	101	1965	3
1.6	20	96	36	565	3
1.6	40	280	89	1307	3
1.6	60	864	220	4148	3

The emulsions are showing shear thinning behaviour; however, the structure is almost fully recovered as shear is reduced again, as seen in Figure 5.

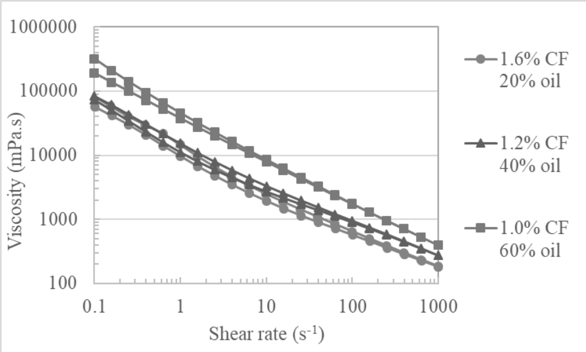


Figure 5. Viscosity as a function of shear rate for 1.6 % CF in 20 % oil, 1.2 % CF in 40 % oil and 1.0 % CF in 60 % oil in oil-in-water emulsions.

The data was modelled in MODDE. Both structure (G') and viscosity (100 s^{-1}) could be predicted by the oil and CF contents and because the overall picture was similar, only viscosity is shown, Figure 6. A very good (R2=0.99) and robust (Q2=0.99) model was able to predict the viscosity.

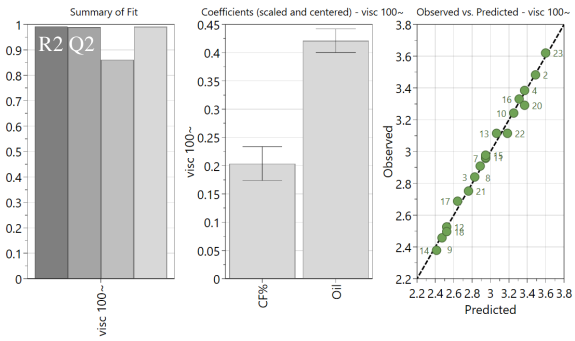


Figure 6. Summary of fit, coefficients (scaled and centered) and observed vs. predicted values shown for viscosity at 100 s^{-1} of the emulsions. CF and oil % both add to the viscosity of the emulsions.

The viscosity increases as CF and oil % increases as seen in the contour plot, Figure 7.

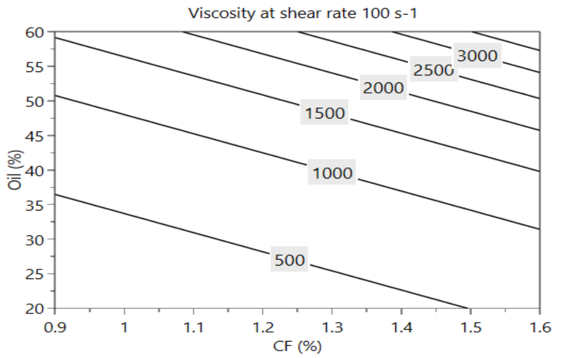


Figure 7. Contour plot of the viscosity (100 s^{-1}) for the emulsions.

The Stability score, Figure 8, follows the same trend as the viscosity, clearly showing that a certain viscosity will help with stabilizing the emulsion. In Table 3 stable emulsions are listed for 20, 40 and 60 % oil together with structure (G') and viscosity and a photo of the emulsion.

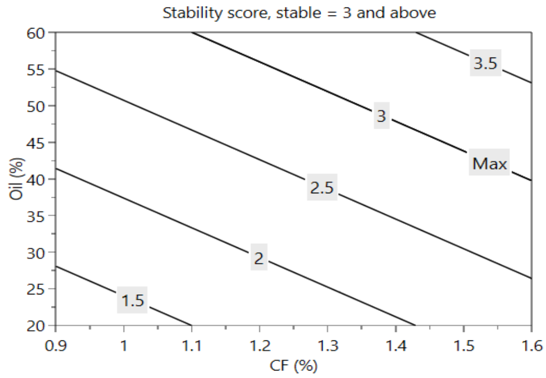
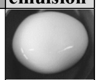
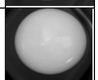
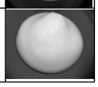


Figure 8. Contour plot of stability score where 3 is required for a stable emulsion.

Table 3. Typical stable oil-in-water emulsions.

CF (%)	Oil (%)	G' (Pa)	Visc. (mPas)	Stability score	Photo of emulsion
1.6	20	96	565	3	
1.2	40	132	949	3	
1.0	60	411	1755	3	

CONCLUSION

NUTRAVA™ Citrus Fiber is able to stabilize oil-in-water emulsions at 20 – 60% rapeseed oil by increasing viscosity and creating a fiber network stabilized by the inherent pectin present inside the fiber matrix.

REFERENCES

1. Staunstrup, J. *et. al.* “Methods of producing activated pectin-containing biomass compositions” US10287366B2.
2. Hotchkiss, S. and Trius, A. “Functional Food Fibers and their use in healthier fat reduced formulations”, Agro. Food Industry Hi Tech. vol 27, 26-29, 2016.
3. Ngouemazong, E. D. et al., The emulsifying and Emulsion-Stabilizing Properties of Pectin: A Review”, Comprehensive Reviews in Food Science and Food Safety, vol. 14, 705-718, 2015.