NUTRAVA® Citrus Fiber can Stabilize Oil-in-Water Emulsions even in the Presence of up to 4% Salt

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ABSTRACT

There is a strong consumer trend for more nature-based food, and as NUTRAVA® Citrus Fiber is a label-friendly ingredient produced from sustainably sourced citrus peels, a by-product of the fruit juice industry, it is an obvious choice for supporting dietary fiber intake and providing texture and stability in food applications.

One of the main application areas for NUTRAVA® Citrus Fiber is oil-in-water systems, where it provides texture and stability. There are several types of citrus fiber available, which can be categorized into three major subgroups, depending on raw material source and mechanical processing when producing the citrus fiber.

This investigation characterizes three different citrus fibers and it is seen that they have different properties and robustness towards providing stability and texture in oil-in-water emulsions containing salt and calcium. Especially NUTRAVA® Citrus Fiber can provide stability even in the presence of up to 4% NaCl as well as in the presence of Calcium.

INTRODUCTION

Food producers are looking for clean label possibilities[1], 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.

There are several commercial citrus fibers available on the market and they can be categorized into two main types, one type that is prepared from waste peel after juice production and one type prepared from waste peel after pectin production (spend peel).

NUTRAVA® Citrus Fiber (CF1) is prepared from waste peel after juice production and consists of ~40% insoluble fiber, primarily cellulose, and ~40% soluble fiber, pectin, which is retained during our patented process. Based on publicly available document, commercial Citrus Fiber 2 (CF2) is presumably prepared from spent peel, using a by-product from pectin production, and contains less amount of pectin and commercial Citrus Fiber 3 (CF3) is presumably prepared from peel waste after juice production.

Different types of citrus fiber (CF1, CF2 and CF3) can be used as ingredients in clean-label products and the combination of soluble and insoluble dietary fibers follows the market trend of functional ingredients for use in clean-label food products. One of the food applications in which citrus fiber can be used successfully is oil-in-water systems[2], however, a typical sauce can contain up to 4% NaCl which can impact the functionality of the citrus fiber. This study evaluates the three main types of citrus fiber and their ability to stabilize simple oil-in-water systems in the presence of calcium and in the presence of up to 4% NaCl.

METHODS AND MATERIALS

DOE-1 (Design of experiments, MODDE (version 16 from Sartorius Stedim Biotech, Goettingen, Germany) investigating the impact of Calcium:

Weigh out 100, 200 or 300 g rapeseed oil (kokkens catering, DK) into a 1000 ml plastic beaker, make a slurry by adding 6 g CF1 (NUTRAVA® Citrus Fiber peak (CP Kelco ApS, Lille Skensved, Denmark), CF2 or CF3 stirring at 500 rpm using a propeller mixer for 1minute. Add 1.5 ml 20% potassium sorbate (CAS no. 24634-61-5), 2 drops antifoaming agent (AFE-1510. dimethyl polysiloxane), de-ionized water (to reach 500 g), and 0 or 5 ml 0.5M $CaCl_2$ (CAS no. 10035-04-8) while continuing to mix for 1 minute at 500 rpm.

DOE-2 (Design of experiments), MODDE (version 16 from Sartorius Stedim Biotech, Goettingen, Germany) investigating the impact of NaCl:

Weigh out 50, 150 or 250 g rapeseed oil (kokkens catering, DK) into a 1000 ml plastic beaker, make a slurry by adding 4, 6 or 8 g CF1 (NUTRAVA® Citrus Fiber peak (CP Kelco ApS, Lille Skensved, Denmark) or CF2 stirring at 500 rpm using a propeller mixer for 1 minute. Add 1.5ml 20% potassium sorbate (CAS no. 24634-61-5), 2 drops antifoaming agent (AFE-1510. dimethyl polysiloxane), 0, 2 or 4% NaCl (CAS no. 7647-14-5) and de-ionized water (to reach 500 g), while continuing to mix for 1 minute at 500 rpm.

Preparing emulsions:

Continue mixing for an additional 5 minutes, while adjusting 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 301 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 measurement is started.

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

Stability test:

Add 10 g emulsion to a centrifuge tube, centrifuge at 4400g for 10 minutes on Hettich Rotana 460R (Andreas Hettich GmbH & Co., Tuttlingen, Germany). Visually evaluate if emulsions remain stable or separate, ranked from 5 = no visual separation to 1 = several layers, oil and water separation.

RESULTS

The emulsions were prepared according to the DOE 1 and DOE 2, listed in **Tab. 1**, together with the measured responses G' after 300s, viscosity at 10s-1, and stability score. Data were analysed in MODDE and models for the three responses were found. The contour plot of the responses for DOE 1 is shown in **Fig. 1**, here CF type, oil% and calcium level was investigated. It is seen that although the structure of CF2 is highest, the stability score and viscosity are highest for CF1. Unfortunately, Citrus Fiber type CF3 was not able to stabilize any of the emulsions, irrespective of oil% or Ca-level.

In general, increasing oil% results in higher structure, higher viscosity, and more stable emulsions for CF1 and CF2, whereas increasing Ca-level results in slightly lower structure for CF1 and lower structure for CF2. Viscosity is in general decreased as Ca-level increases, and the same is seen for the stability score.

As CF3 was not able to stabilize any emulsions prepared in DOE 1, the CF type was not included in DOE 2, **Tab.1**. This investigation looked at the impact of citrus fiber use level, CF type (CF1 and CF2), oil% and salt% (NaCl).

TABLE 1: DOE 1 – Factors: CF type, oil% and Ca (ppm) and DOE 2 – Factors: CF type, CF%, oil% and salt%. Responses measured on the prepared emulsions are structure (G'), viscosity at 10s-1 and stability score.

DOE 1					
type	**oil%**	**Ca**	**Structure G' (Pa)**	**visc10 (mPas)**	**Stability Score**
CF1	20	0	55	1048	3
CF1	20	200	32	629	2
CF1	40	0	109	2912	5
CF1	40	200	122	2611	5
CF1	60	0	493	11358	5
CF1	60	200	399	9151	4
CF2	20	0	133	1147	2
CF2	20	200	29	255	1
CF2	40	0	297	2386	3
CF2	40	200	155	1281	2
CF2	60	0	697	6797	5
CF2	60	200	310	2161	1
CF3	20	0	3	81	1
CF3	20	200	2	37	1
CF3	40	0	10	312	2
CF3	40	200	9	180	1
CF3	60	0	38	944	1
CF3	60	200	22	556	1

DOE 2						
CF type	**CF%**	**oil%**	**salt%**	**Structure G' (Pa)**	**Visc10 (mPas)**	**Stability score**
CF1	1.2	30	2	63	1530	3
CF2	1.6	50	4	524	4419	1
CF2	1.6	10	0	221	1539	1
CF1	1.6	50	0	466	11222	5
CF1	1.6	10	4	53	1153	4
CF1	0.8	50	4	161	2625	2
CF1	1.2	30	2	61	1463	2
CF1	0.8	50	0	139	2939	4
CF2	1.2	10	4	40	281	1
CF2	0.8	50	4	173	746	1
CF1	1.2	30	2	55	1393	3
CF2	0.8	10	0	28	283	1
CF1	1.2	10	0	38	813	1
CF2	1.6	30	2	282	1866	1
CF2	1.2	50	0	563	4375	2
CF1	1.6	50	4	555	12090	5
CF1	0.8	10	4	7	153	1

The emulsions were prepared according to the DOE 1 and DOE 2, listed in **Tab. 1**, together with the measured responses G' after 300s, viscosity at 10s-1, and stability score. Good models are found for structure (G') and viscosity at 10s-1 as seen in **Fig. 2** and all factors, CF type, oil% and calcium level impact the models significantly. The stability score is not easily modelled, although CF type is significant, oil and calcium level are barely significant, and model fit is ok, R2 = 0.73, but the model is not very robust as Q2 is only 0.50.

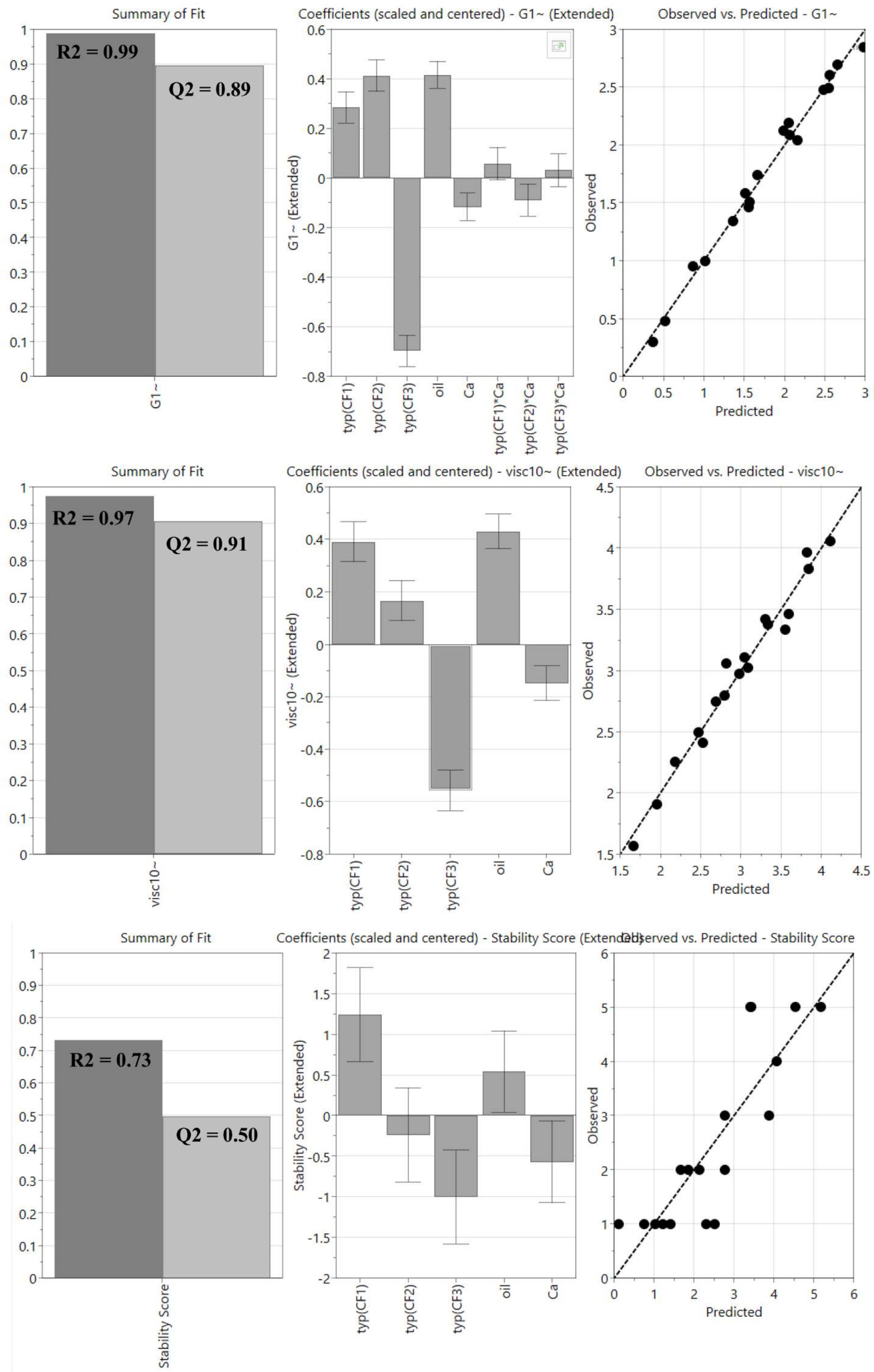

FIGURE 1: Summary of Fit of the model, showing R2 and Q2 of the model, coefficients (scaled and centered factors with error bars) and the observed response and predicted response are plotted for the structure (G') (Model fit R2 = 0.99; robustness Q2 = 0.89) of the emulsion's top plots, and the viscosity at 10s-1 (Model fit R2 = 0.97; robustness Q2 = 0.91) middle plots. Stability score is modelled (Model fit: R2= 0.73; robustness Q2 = 0.50).

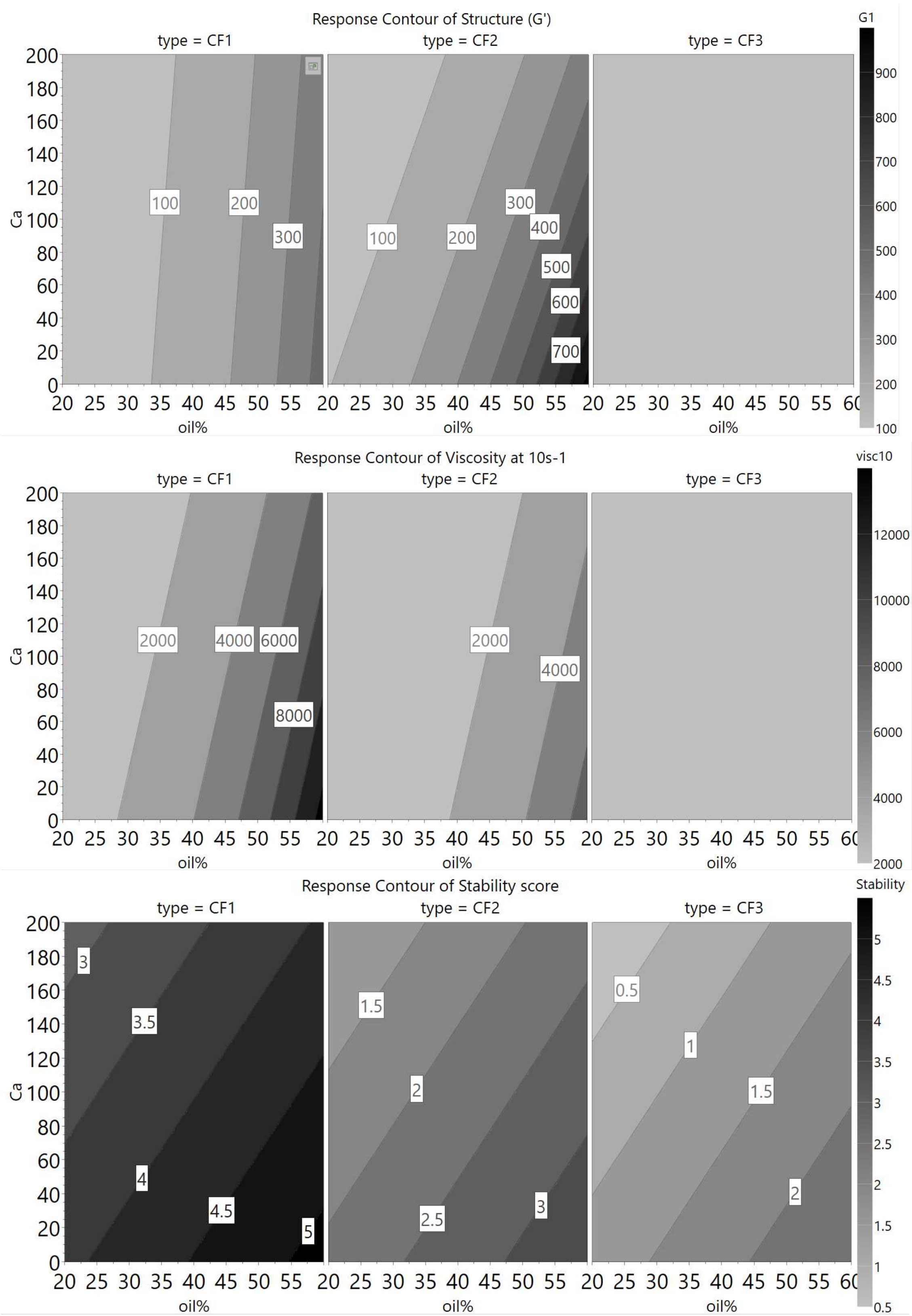

FIGURE 2: Response Contour plot of G' (structure), Viscosity at 10s-1, and Stability Score for emulsions prepared with different Citrus Fibers (CF1, CF2 and CF3) at different Ca-level (0 – 200ppm) and oil content (20 – 60%).

The response contour plot is shown in **Fig. 2**, and it is seen that none of the emulsions prepared with CF3 were stable. The stability score is below 2 indicating that samples separated when subjected to centrifugation. Furthermore, low structure and viscosity were seen and often the samples even separated when left undisturbed. The other CF types (CF1 and CF2) were able to stabilize the emulsions prepared and all appeared visually stable. It is seen that functionally the emulsions are different, where CF1 provides the highest viscosity and stability score, CF2 provides a higher initial structure (G'), but as the emulsion is sheared the structure is ruptured and the emulsion becomes more shear sensitive, which also results in lower stability score than CF1.

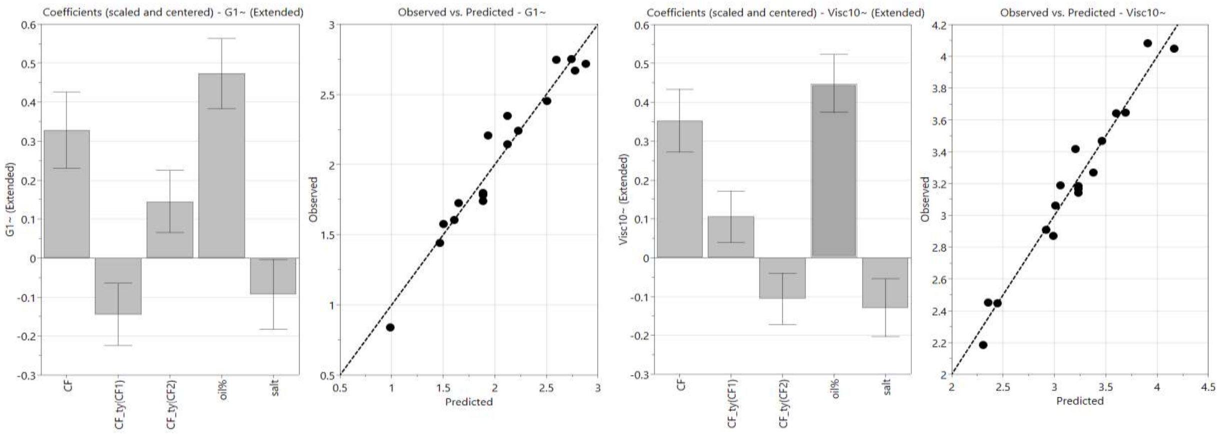

FIGURE 3: Coefficients (scaled and centered factors with error bars) and the observed response and predicted response are plotted for the structure (G') (Model fit R2 = 0.95; robustness Q2 = 0.88) of the emulsion's left plots, and for the viscosity at 10s-1 (Model fit R2 = 0.96; robustness Q2 = 0.91) right plots.

The calcium level only slightly impacts the structure of CF1, but the viscosity does decrease as calcium increases. However, for CF2 both structure and viscosity decrease as calcium level increases. Stability typically drops one level when increasing the calcium up to 200 ppm.

The effect of preparing an emulsion in a high salt environment (4% NaCl) was investigated in DOE2. For the structure, G' a good and robust model (R2 = 0.95; Q2 = 0.88) is found where CF%, CF type and oil% significantly impacted the structure of the emulsion, whereas the salt level was barely significant, **Fig. 3**. A good and robust model (R2 = 0.96; Q2 = 0.91) to predict viscosity at $10s^{-1}$ was found where all factors were significant.

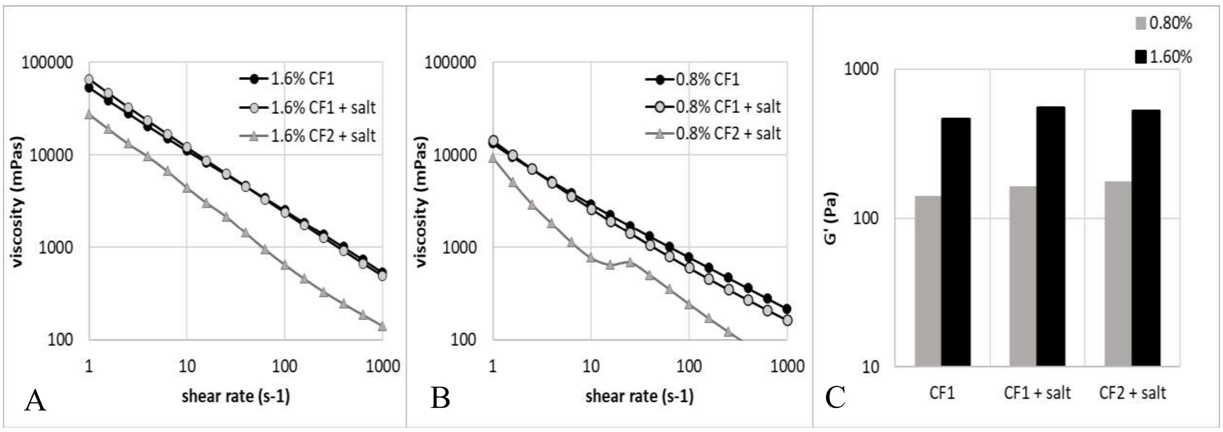

FIGURE 4: Viscosity versus shear rate for selected 50% oil in water emulsions using 1.6% CF plot A, 0.8% CF plot B and the structure (G') for the emulsion are shown in plot C.

The trend is that increasing salt level results in slightly lower structure and viscosity. However, this trend is related to the CF type, and in general the viscosity of CF2 appears to be more impacted by increased salt-levels, as seen in **Fig. 4**. Whereas the structure is only slightly influenced by the salt level, the greatest impact is seen for the stability score, no emulsions appeared stable for CF2 whereas emulsions prepared at high use level of CF1 and in oil concentrations between 30-60% oil could withstand centrifugation, **Fig. 5**.

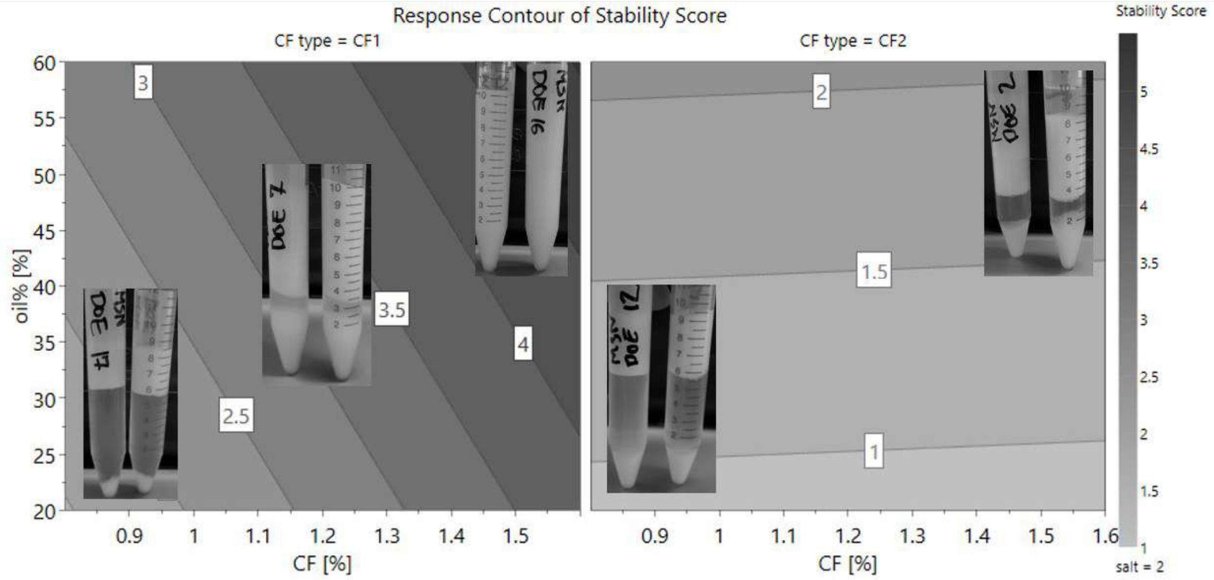

FIGURE 5: Response contour plot showing stability score as a function of CF% and oil% for the two CF types, this data is for intermediate salt level (2% NaCl) but the overall trend is the same for all salt levels.

CONCLUSION

The use of citrus fiber as a clean label-friendly ingredient produced from sustainably sourced citrus peels for condiments is an obvious choice. However, it is important to select the citrus fiber that fits the need, as it is seen that different citrus fibers have different functionality when used for stabilizing oil-in-water emulsions.

Three different citrus fibers that have been produced in different ways have different properties and robustness towards providing stability and texture in oil-in-water emulsions containing salt and calcium. Especially NUTRAVA® Citrus Fiber (CF1) can provide emulsion stability even in the presence of up to 4% NaCl as well as in the presence of Calcium.

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