International Journal of Chemical, Material and Environmental Research 2015, 2 (4): 52-62

Research article

IJCMER
www.ijcmer.org

**INTERNATIONAL JOURNAL OF CHEMICAL,
MATERIAL AND ENVIRONMENTAL RESEARCH**
An International Open Free Access, Peer Reviewed Research Journal

ISSN 2410-5945
2015, Vol. 2, No. 4:
pg. 52-62

Modelling the Effect of Emulsion Components on the Properties of Oil-in-Water Emulsion Stabilized with Gelatinized Bambara Groundnut Flour Using Response Surface Methodology

Oladayo Adeyi[1, 3]*, Daniel I.O. Ikhu-Omoregbe[1], Victoria A. Jideani[2]

[1]Department of Chemical Engineering, Cape Peninsula University of Technology, Cape Town 7530, South Africa
[2]Department of Food Technology, Cape Peninsula University of Technology, Cape Town 7530, South Africa
[3]Department of Chemical Engineering, Landmark University, Omu-aran, Nigeria

(Received: November 04, 2015; Accepted: December 16, 2015)

Abstract

The unending demand for natural food product by consumers has increased research in food emulsion technology towards finding natural emulsifiers and stabilizers to improve the properties of oil-in-water food emulsions. Bambara groundnut although abundant in Africa has been grossly underutilized despite its admiring physicochemical properties. This work therefore explored the use of bambara groundnut flour (BGNF) as the sole natural stabilizer in food emulsion system. The contribution of the main emulsion components viz BGNF and sunflower oil (SFO) on the emulsion stability parameter (droplet size ($d_{3,2}$ and $d_{4,3}$)) and time-dependent rheological properties of BGNF-stabilized emulsions for formulation purposes was investigated. D-optimal response surface methodology was used for the experimental design. Droplet size and rheological characterizations were carried out using image analysis and a shear rate controlled rheometer respectively. Both BGNF and SFO had significant influence on the droplet size and time dependent behaviour of the emulsion. Quadratic polynomial relationships were found between the emulsion main components (BGNF and SFO), droplet sizes ($d_{3,2}$ and $d_{4,3}$) and time dependent properties. The interaction effect of BGNF and SFO was positive while the linear effect was negative on the time-dependent properties of the emulsions. Emulsion formulated with 7% (w/w) BGNF and 40% (w/w) SFO possessed highest structural interaction, stability and manifested as high viscosity.

Keywords: Bambara, groundnut, emulsion, rheology, stability

1. Introduction

An emulsion is a biphasic, metastable coarse dispersion of two immiscible materials, usually liquids (typically oil and water), that produces a semisolid [1]. Emulsion stability and rheological behaviour are among important properties of emulsion systems. Several industrial systems such as food emulsions, personal care and cosmetics, pharmaceuticals and paints consist of emulsions. Generally, emulsions are thermodynamically unstable and have tendencies to break down over time due to the difference in specific gravity between the oil droplets and medium usually water [2, 3]. Emulsions can destabilize by creaming, droplet flocculation, coalescence and Ostwald ripening or combination of two or more phenomena.

---

* Corresponding author:
adeyioladayo350@yahoo.com

In order to make emulsions kinetically stable, emulsifiers are added which keep the dispersed phase in the form of droplets suspended in a continuous phase. Particle size determination of emulsion is probably the most important way of emulsion characterization [4]. This is because particle size influences other properties of emulsions such as texture, creaming velocity and rheology [5], tastes, appearance, etc. Creaming stability of emulsion is largely dependent on the particle size. The larger the oil droplet the higher the creaming rate.

The knowledge of the rheological properties of food dispersions cannot be over emphasized for numerous reasons. Rheological data of food products are needed in shelf life testing, process engineering calculations, determination of ingredient functionality, quality control and in sensory evaluations [6]. Rheological data are also useful during food product development stage [7] and could address the industrial production of food (stirring, pumping, dosing, dispersing and spraying), home based cooking as well as consumption of food (oral perception, digestion and well-being) [8]. From a technological point of view, the rheology of food dispersions is fundamental mainly due to its relationships with emulsion stability [9]. Time-dependent rheological behavior is therefore very important for understanding the products changes that occur during processing [10]. Reliable and accurate characterizations of time-dependent properties of foodstuffs are necessary for the quality control, product optimization, texture, shelf-life and for the design of processing equipment [6].

One of increasingly growing area in food emulsion technology research is finding new natural alternatives for improving the stability and rheological properties of emulsions. This is because consumers are demanding more natural product. Therefore finding natural emulsifiers and stabilizers that have required functionalities in food systems has remained a significant interest [11] and challenge in food industries. The nutritional composition of bambara groundnut (BGN) indicates its potential as a natural food emulsifier/stabilizer. BGN contained carbohydrate contents of 49-63.5%, protein content of about 15-25%, fat contents of about 4.5-7.4%, fibre content of 5.2-6.4, ash of 3.2-4.4% and 2% mineral [12]. The high percentage of protein and carbohydrate contents as well as the probable protein-carbohydrate interactions during gelatinization and emulsion preparation made BGN to have high potential as main emulsifier/stabilizer in food emulsions [3].

Therefore, the objectives of this study are to determine the relationship between the emulsion components/ingredients, oil droplet sizes (which determines emulsion stability) and time dependent rheological properties of the gelatinized BGN stabilized emulsions. This is for the purpose of understanding the contributions (both individual and interactive) of the ingredients (sunflower oil (SFO) and BGN) to the intrinsic properties of the emulsions which are useful for the formulation purposes.

2. Materials and methods

Dried Bambara groundnut (BGN) seeds of brown variety were purchased from Triotrade Gauteng CC, South Africa. The seeds were washed, and dried at 50^{o}C for 48 hr by using cabinet drier (Model: 1069616). The dried seeds were milled into flour using a hammer mill and screened through 90 µm sieve to give bambara groundnut flour (BGNF). A commercial brand (Ritebrand) of 100% SFO purchased from a local supermarket was used without purification as the hydrophobic dispersed phase in this work. Milli-Q water was used in the preparation of all the emulsions.

2.1. Emulsion Preparation

Emulsions were prepared from a dispersed phase and a continuous phase. The dispersed phase consisted of SFO and continuous phase was gelatinized BGNF dispersion. Response surface methodology (RSM) D-optimal was used for this study. The independent variables (BGNF and SFO) and experimental design in the coded and uncoded forms are as shown in Table 1. Nine formulations of emulsions were made in duplicate. BGNF dispersions of specific concentrations were prepared according to Table 1 by dispersing measured amount of BGNF (5-7% (w/w)) in known quantity of Milli-Q water. The resulting dispersions were gelatinized at a temperature of 84^{o}C for 10 min with constant stirring. The resulting gelatinized BGNF dispersions (GBGNFD) were weighted in order to ascertain the amount of water loss during gelatinization. Water loss during gelatinization was compensated for by adding Milli-Q water to the GBGNFD, stirred and allowed to cool down to 20^{o}C. Measured quantities of SFO (30-40% (w/w)) were added into the gelatinized BGNF to achieve different oil concentrations. Emulsions (100 g) were made by homogenizing SFO and gelatinized BGNF at 20^{o}C using an Ultra Turrax T-25 homogenizer (IKA, Germany) for 10 min at the speed of 11000 rpm.

2.2. Quantification of Droplet Sizes and Distributions of Emulsion by Image Analysis

Microstructure of the emulsions immediately after emulsion preparation was analyzed in terms of droplet size and droplet size distribution by the method of Adeyi *et al.* [3]. Each emulsion was diluted with Milli Q-water at a ratio of 1:5 (w/w) in order to avoid overlapping and agglomeration of oil droplets which can affect further image analysis and processing. Droplet sizes were determined from the images of the oil-in-water emulsion obtained with a light microscope (Ken-A-vision). Emulsion samples were poured onto microscope slides and covered with glass cover slips and visualized using X40 objective lens. The microscope focus and the light intensity were carefully controlled and optimized in order to

Table 1. Response Surface D-Optimal Design for BGNF Stabilized Emulsion[a]

Independent variables				
Coded levels			**Uncoded levels**	
Run	BGNF	Oil-phase	BGNF %(w/w)	Oil-phase %(w/w)
1	+1	+1	5	30
2	+1	0	5	35
3	+1	-1	5	40
4	0	+1	6	30
5	0	0	6	35
6	0	-1	6	40
7	-1	+1	7	30
8	-1	0	7	35
9	-1	-1	7	40

[a] +1 refers to the minimum level; 0 is the center point; -1 is the maximum level; BGNF refers to bambara groundnut flour; SFO is the sunflower oil

obtain the sharpest possible boundaries between the oil-droplets and the surrounding GBGNFD. The images were captured with a digital camera mounted on the microscope. Image processing and further analysis was carried out using public domain software image J v1.36b [13, 14]. The diameters of the oil droplets were measured one by one by an operator according to the method of Tcholakova [15]. A substantial number of droplets (N = 1000) were counted in order to obtain statistical estimate of the oil-droplet diameters and oil droplet size distribution in each sample. Droplet size distributions were generated by grouping the droplets into classes belonging to a common interval. Droplet size frequency distributions were computed using MS-Excel (Microsoft^{TM} Excel 2007) [16]. Oil-droplet sizes were obtained in terms of volume-surface mean diameter ($d_{3,2}$) and equivalent volume-mean diameter ($d_{4,3}$). The volume–surface mean diameter ($d_{3,2}$) and equivalent volume-mean diameter, $d_{4,3}$ were calculated using Equation 1 and 2, respectively.

$$d_{3,2} = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \qquad 1$$

$$d_{4,3} = \frac{\sum n_i d_i^4}{\sum n_i d_i^3} \qquad 2$$

where n_i is the number of droplets with diameter d_i (µm).

2.3. Time-Dependent Rheological Measurement

Time-dependent rheological experiments of the emulsions containing SFO (30-40 % (w/w)) stabilized with BGNF (5-7 % (w/w)) were conducted. Rheological measurements were conducted using a shear rate controlled rheometer (Rheolab MC 1, Physica Inc., Stuttgard Germany). All the experiments were performed at 20^{o}C without previous shearing. Samples were carefully transferred into the rheometer cup and allowed to rest for about 10 min. Viscosity was measured as a function of increasing shear rate from 40 to 750 s^{-1} followed by a decreasing rate from 750 to 40 s^{-1}. The hysteresis loop area was calculated as the area between the upstream data and downstream data using Equation 3 [17, 18].

$$\text{Hysteresis loop area} = \int_{\gamma_1}^{\gamma_2} K\gamma^{n} - \int_{\gamma_1}^{\gamma_2} K'\gamma^{n'} \qquad 3$$

where K, K' are the consistency coefficient, and n, n' are the flow behavior indices for upward and downward measurements, respectively. Each experiment was performed in duplicate.

2.4. Empirical Modeling and Response Surface Analysis

Stability and rheological data obtained from the D-Optimal experimental design in Table 1 were treated using the Response Surface Methodology (RSM) in order to establish the relationship between the measured parameters and main emulsion component (BGNF and SFO). All experiments as designed were performed in duplicates for this study. The variance for each parameter assessed was divided into linear, quadratic, and interactive components and was represented using a second-order polynomial model (Equation 4):

$$Y = b + \sum b_i X_i + \sum b_{ii} X_i^2 + \sum b_{ij} X_i X_j \qquad 4$$

where Y is the estimated parameters response; equation coefficients were represented by b (constant term), b_i (linear effect), b_{ii} (quadratic effect) and b_{ij} (interaction effect). X_i and X_j are the emulsion components (BGNF and SFO). The statistical significance of each parameter was determined by analysis of variance (ANOVA) at 5% [19]. The surface graphical presentations of the response surface models were performed using Design Expert 9 software.

The quality of the fit of the polynomial model was judged by coefficient of determination R^2 and R_{adj}^2 as expressed below in Equations 5 and 6, respectively.

$$R^2 = 1 - \frac{SS_{residual}}{SS_{model} + SS_{residual}} \qquad 5$$

$$R_{adj}^2 = 1 - \frac{SS_{residual} / DF_{residual}}{SS_{model} + SS_{residual} / DF_{model} + DF_{residual}} \qquad 6$$

SS is the sum of squares and *DF* is the degrees of freedom.

3. Results and Discussions

3.1. Effect of BGNF and SFO on Droplet Size Distribution

Figure 1 shows the oil droplet size distribution of all the studied emulsions. Figure 1 A, B and C are the oil droplet size distribution of freshly prepared emulsions containing 30, 35 and 40% SFO, stabilized with 5, 6 and 7% (w/w) GBGNF dispersions respectively. None of the distribution showed a perfect Gaussian shape and all tended to have a shoulder which is an indication of second population and therefore are poly dispersed in nature. The mean droplet size of the emulsion is as detailed in Table 2.

Table 2. De-Sauter ($d_{3,2}$) and De Brouker ($d_{4,3}$) of the BGNF Stabilized Emulsion[a,b]

Emulsion BGNF (%w/w)	**SFO (%w/w)**	**$d_{3,2}$ (μm)**	**$d_{4,3}$ (μm)**
5	30	4.05 ± 0.09	4.36 ± 0.11
	35	4.53 ± 0.03	4.77 ± 0.21
	40	5.35 ± 0.12	5.92 ± 0.04
6	30	3.07 ± 0.24	3.19 ± 0.12
	35	3.45 ± 0.07	3.61± 0.31
	40	3.90 ± 0.61	4.16 ± 0.15
7	30	2.80 ± 0.23	2.89 ± 0.41
	35	2.91± 0.01	3.00 ± 0.22
	40	3.45 ± 0.31	3.66 ± 0.45

[a] Values ± standard deviation; [b] $d_{3,2}$ refers to the volume surface mean diameter of the emulsions; $d_{4,3}$ is the equivalent volume-mean diameter of the emulsions; BGNF equals bambara groundnut flour; SFO is the sunflower oil

The $d_{3,2}$ is the volume-surface mean diameter or the Sauter-diameter of the emulsions and it provides information regarding the mean diameter where most of the particles fall [20]. The $d_{3,2}$ is as well important when the study of a catalyst or the amount of surfactants are of interest [21]. $d_{4,3}$ on the other hand is the equivalent volume-mean diameter or De Broucker diameter. It is important when total amount of oil in the dispersed phase is significant. $d_{4,3}$ is related to changes in particle size involving destabilization process and hence is more sensitive to existence of large particles and fat droplet aggregation [20]. Thus, $d_{4,3}$ is more sensitive to the phenomenon of droplet flocculation [21]. The oil-droplet size distribution shows that both the BGNF and SFO concentrations clearly affected the oil-droplet size. The mean value of the volume-surface mean diameter, $d_{3,2}$ of BGNF emulsions was in the range of 2.80-5.35 μm while the $d_{4,3}$ in the range of 2.89-5.92 μm. The emulsions stabilized with 5% (w/w) BGNF recorded the highest oil-droplet size diameters ($d_{3,2}$ and $d_{4,3}$) while emulsions stabilized with 7% (w/w) recorded the least. This indicated that emulsion stabilized with 7% (w/w) had a strong tendency to remain stable over a long period of time.

The emperical model developed for $d_{3,2}$ and $d_{4,3}$ as a function of BGNF and SFO, as well as the analysis of variance (ANOVA) and coefficients of regression are as presented in Table 3. The response surface graphs is also presented in Figure 2. The quadratic polynomial was significant ($p < 0.0001$) and with high values of R^2, making it suitable for predicting the droplet size as a function of of emulsion components. The linear terms of SFO and BGNF and quadratic term of BGNF were the only significant model terms ($p < 0.005$) for the $d_{3,2}$. The interaction between BGNF and SFO and the quadratic effect of SFO did not have significant effect on $d_{3,2}$. The linear terms of BGNF and SFO were negative while the quadratic effect of BGNF was positive on droplet size ($d_{3,2}$). Regarding $d_{4,3}$, all model terms were significant ($p < 0.05$) except the interaction term of SFO and BGNF. The effect of the linear terms of BGNF and SFO and their quadratic terms were negative and positive, respectively. The interaction between BGNF and SFO did not have a significant effect on both $d_{3,2}$ and $d_{4,3}$, (Figure 2). As expected, there was an increase in oil droplet size with an increase in dispersed phase (SFO) concentration for all BGNF concentration studied (Figure 2).

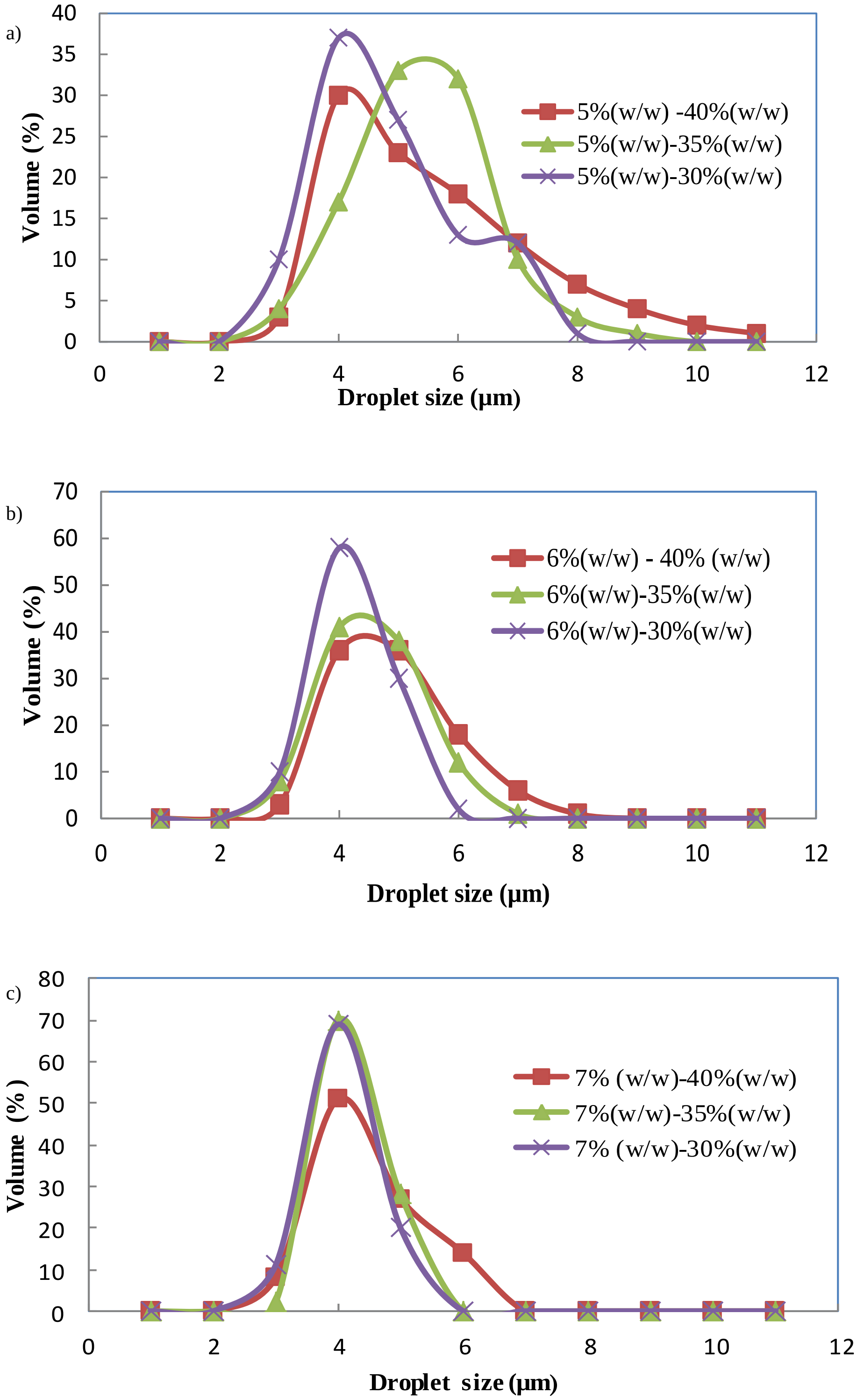


Figure 1. Droplet Size Distribution of Dispersed Phase Particles in Emulsion Stabilized with Bambara Groundnut Flour (BGNF) (a) 5% (w/w) (b) 6% (w/w) (c) 7% (w/w)

Similar trend were reported by Wang *et al.* [22] for soybean oil-in water emulsions. However, the increase in $d_{3,2}$ was moderate, for example the mean value of $d_{3,2}$ for emulsions containing 30% (w/w), 35% (w/w) and 40% (w/w) stabilized with 5% (w/w) BGNF were 4.05 µm, 4.53 µm and 5.35 µm, respectively. The effect of BGNF concentration on oil-droplet size was also significant ($p < 0.05$). With an increase in the BGNF concentration, the $d_{3,2}$ decreased. For example the $d_{3,2}$ of the emulsions containing 40% (w/w) SFO, stabilized with 5% (w/w), 6% (w/w) and 7% (w/w) are 5.35 µm, 3.91 µm and 3.45 µm, respectively.

The decrease in the particle size with the addition of polymer has been explained to be due to an increase in viscosity of the continuous phase of the emulsion. For a given speed of the homogenizer, the shear stresses are higher when the viscosity of the continuous phase of the emulsion is high and hence finer droplets are produce in the mixer [23, 24]. The De Broucker diameter, $d_{4,3}$ which is related to destabilization processes were found to increase with an increase in sunflower oil concentration and decrease with an increase in BGNF concentration. The behavior of colloidal systems in terms of creaming and coalescence has been explained to be dependent on the population of higher droplet sizes in the system. The higher the droplet sizes in an emulsion system, the greater the tendency to coalesce because the impact forces and their magnitudes are higher during collision [20].

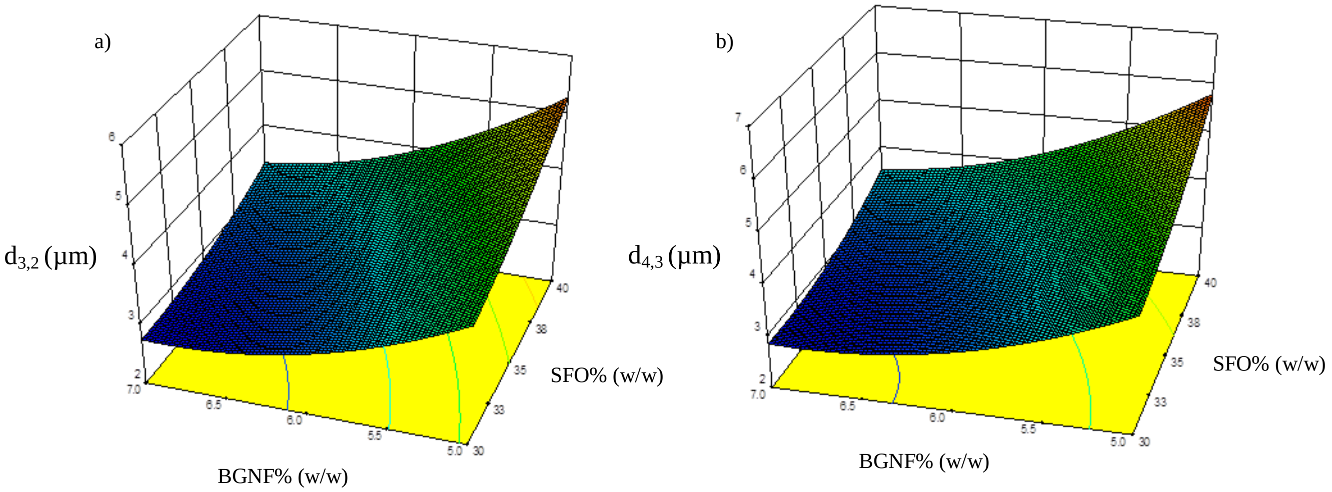


Figure 2. Response Surface for the Effect of Bambara Groundnut Flour (BGNF) and Sunflower Oil (SFO) Concentrations on (a) De-Sauter ($d_{3,2}$) (b) De Brouker ($d_{4,3}$)

Table 3. Analysis of Variance (ANOVA) for the Quadratic Model of Oil-Droplet Size[a]

		$d_{3,2}$ (µm)				**$d_{4,3}$ (µm)**		
Source	**DF**	**Coefficient**	**Sum of square**	**F-ratio p-value**	**DF**	**Coefficient**	**Sum of square**	**F-ratio p-value**
Model	5	+18.55	11.04	<0.0001	5	+24.527	15.05	<0.0001
Linear								
b_1	1	-0.1137	2.58	<0.0001	1	-0.3157	3.63	<0.0001
b_2	1	-4.1392	7.62	<0.0001	1	-4.8942	10.08	<0.0001
Quadratic								
b_{11}	1	+5.73E3	0.082	0.0634	1	+9.46E3	0.22	0.0146
b_{22}	1	+0.3733	0.56	0.0002	1	+0.4467	0.80	0.0002
Interaction								
b_{12}	1	-0.0325	0.21	0.0066	1	-0.0395	0.31	0.0056
Residual	12		0.24		12		0.33	
Pure error	9		0.20		9		0.24	
Lack of fit	3		0.040	0.6287	3		0.0090	0.3888
Total	17		11.28		17		15.38	
R^2		0.9791				0.9785		
Adj-R^2		0.9704				0.9695		
CV		0.9496				4.20		
Adequate precision		31.143				30.605		

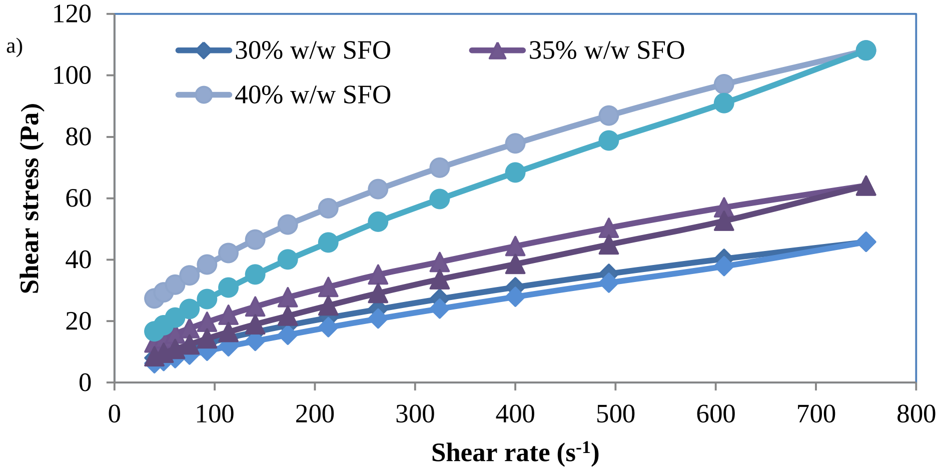
[a] b_1 and b_2 equal the coefficient of the linear term of bambara groundnut flour and sunflower oil; b_{11} and b_{22} are the coefficients of the quadratic terms of the bambara groundnut flour and sunflower oil; b_{12} equals the coefficients of interaction between the bambara groundnut flour and sunflower oil; DF equals degree of freedom; R^2 is the coefficient of determination; Adj R^2 equals the adjusted coefficient of determination; CV is the coefficient of variation

The variation of oil-droplet size as a function of BGNF and SFO is therefore in line with earlier hypothesis that emulsion components will have significant effect on the properties of BGNF stabilized oil-in-water emulsion. The significance dependence of oil-droplet sizes on the emulsion components (BGNF and SFO) may therefore have notable differences on their respective microstructure and eventual emulsion stability.

3.2. Effect of BGNF and SFO on Time-Dependent Rheology with Respect to Hysteresis Loop Area

Among the principal reasons for time-dependent rheological characterization of food systems (including emulsions) are to establish connections between structure and flow, to relate physical parameters with sensory evaluation and for the purpose of optimization of processing stages such as mixing, handling, storage, and final quality [25]. The shear rate sweep of BGNF stabilized emulsions at different BGNF and SFO concentrations are as presented in Figure 3. Hysteresis loop areas were observed when the BGNF emulsions were subjected to increasing and decreasing shear rate. The hysteresis area has been explained to represent an index of energy per unit of time and per unit of volume needed to eliminate the influence of time in the flow behavior [17]. The existence of the hysteresis area between the increasing and decreasing shear rate curves indicated that all the BGNF stabilized emulsions were time dependent and thixotropic in nature [26]. The thixotropy of a material has been interpreted as the continuous breakdown or rearrangement of the network links and this could be quantified by the evaluation of the magnitudes of the area of hysteresis loop between the upward and downward curves [18, 27]. The larger the magnitude of the area, between the flow curves, the higher the thixotropic effect [28]. Table 4 showed the magnitude of the hysteresis loop area for the emulsions at different BGNF and SFO concentrations. The mean values of hysteresis loop areas ranged from 36.198 to 694.45 Pas^{-1}. Time dependency was also found in vegetable-based infant purees [29]; ayran [27], and semi-solid dairy desserts [18].

The ANOVA for the quadratic models developed for the hysteresis loop area is presented in Table 5. The model for the hysteresis loop area as a function of emulsion components was significant ($p < 0.0001$) with high R^2, coefficient of variation, adequate precision values and no significant lack of fit. All model terms were significant ($p < 0.05$) for the hysteresis loop areas. The effect of the linear term of SFO and BGNF were both negative. The quadratic effect of BGNF and SFO and interaction effect of BGNF and SFO were positive on the hysteresis loop area. The response surface for the individual and simultaneous increase of SFO and BGNF on the hysteresis loop area is shown in Figure 4. Hysteresis loop area was dependent on both the BGNF and SFO contents in the emulsions. Simultaneous increase of SFO and BGNF however caused a significant increase in the hysteresis loop area. This indicated that the thixotropic behavior of the entire emulsion samples were built up with an increase in the concentration of BGNF and SFO and this might be due to the increase in the viscosity as the concentration of BGNF and SFO increased. Emulsion formulated with 7% (w/w) BGNF and 40% (w/w) SFO showed the highest hysteresis loop area, while emulsion formulated with 5% (w/w) BGNF and 30% (w/w) SFO had the least. Therefore, if it is assumed that hysteresis loop area is an index of the energy needed to destroy the structure responsible for flow time dependence, the emulsion with 7% (w/w) BGNF and 40% (w/w) SFO was the sample that needed the highest energy to breakdown such structure. This could be as a result of the observed high viscosity of the emulsion.



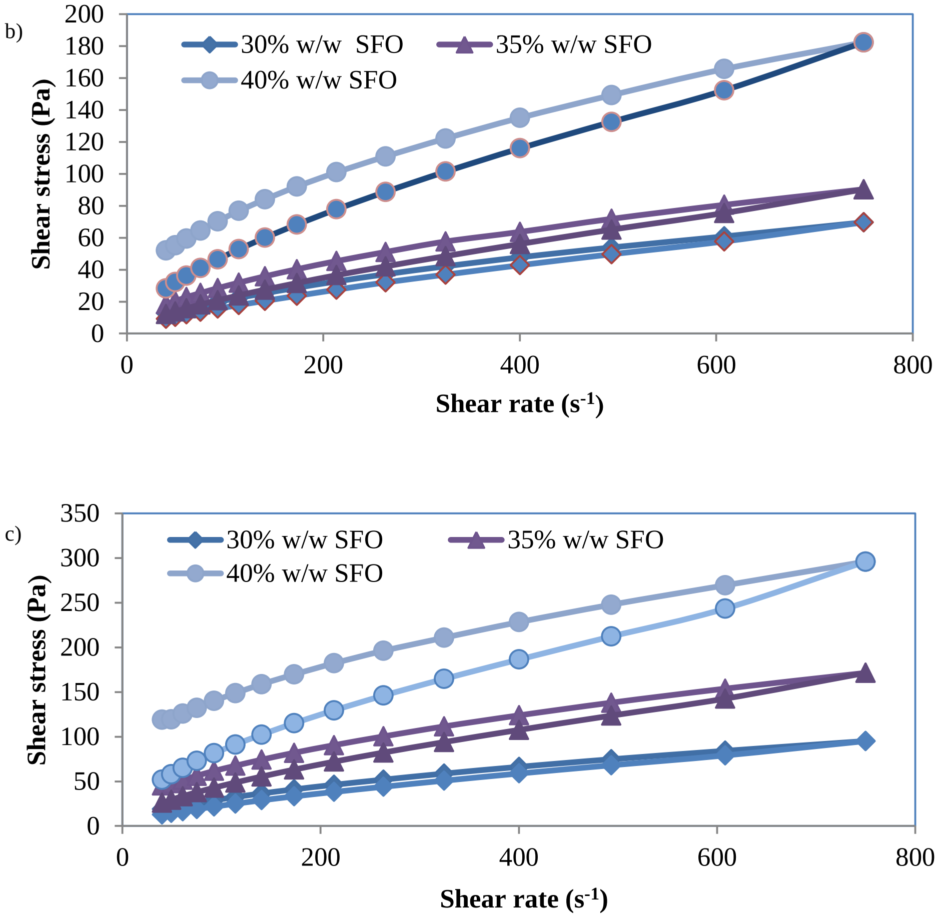


Figure 3. Hysteresis Loop Obtained for Emulsions Containing 30% (w/w), 35% (w/w) and 40% (w/w) Sunflower Oil (SFO), Stabilized with (a) 5% (w/w) (b) 6% (w/w) (c) 7% (w/w) Bambara Groundnut Flour (BGNF)

Table 4. Hysteresis Loop Area Obtained for BGNF Stabilized[a,b]

Emulsion **BGNF (%w/w)**	**SFO (%w/w)**	**Integrating area for upward curve**	**Integrating area for downward curve**	**Hysteresis loop area (Pas^{-1})**
5	30	321.55 ± 5.61	285.35 ± 1.88	36.20 ± 3.73
	35	472.30 ± 5.93	398.09 ± 1.58	74.21 ± 4.35
	40	854.65 ± 9.33	704.09 ± 15.30	150.56 ± 5.98
6	30	494.70 ± 7.94	433.56 ± 5.06	61.14 ± 2.88
	35	677.81 ± 8.99	572.36 ± 5.44	105.45 ± 3.56
	40	1453.86 ± 95.39	1175.00 ± 51.43	278.86 ± 43.95
7	30	696.79 ± 10.30	596.67 ± 9.07	100.12 ± 1.23
	35	1349.00 ± 38.18	1110.04 ± 20.99	238.96 ± 17.19
	40	2683.65 ± 85.35	1989.20 ± 20.73	694.45 ± 64.62

[a] Values ± standard deviation; [b] BGNF is bambara groundnut flour; SFO is sunflower oil

The high stability possessed by emulsion formulated with 7% (w/w) BGNF and 40% SFO was therefore connected to high structural formation (high droplet-droplet interaction) which subsequently led to high viscosity. This has led to the high time-dependence (rigidity) and correspondingly high index of energy necessary to destroy the droplet-droplet structures.

Table 5. Quadratic Model Parameters for the Hysteresis Loop Area[a]

		Hysteresis loop area (Pas^{-1})		
Source	**DF**	**Coefficient**	**Sum of Square**	**p-value**
Model	5	+4053.93	103700	<0.0001
Linear				
b_1	1	-155.53	82070.26	<0.0001
b_2	1	-621.78	48976.85	<0.0001
Quadratic				
b_{11}	1	+1.55	4726.52	0.0057
b_{22}	1	+25.56	2033.00	0.0443
Interaction				
b_{12}	1	+11.49	11548.40	0.0003
Residual	10		83437.39	
Pure error	8		2318.45	
Total	15		107500	
Lack of fit	2		1527.43	0.3913
R^2	0.9642			
Adj-R2	0.9463			
CV	15.01			
Adequate precision	19.107			

[a] b_1 and b_2 equal the coefficient of the linear term of bambara groundnut flour and sunflower oil; b_{11} and b_{22} are the coefficients of the quadratic terms of the bambara groundnut flour and sunflower oil; b_{12} equals the coefficients of interaction between the bambara groundnut flour and sunflower oil; DF equals degree of freedom; R^2 is the coefficient of determination; Adj R^2 equals the adjusted coefficient of determination; CV is the coefficient of variation

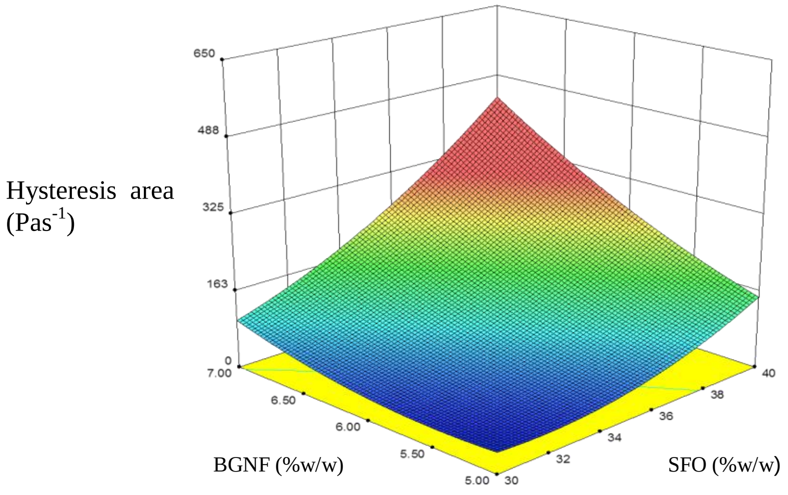


Figure 4. Response Surface for the Effect of Bambara Groundnut Flour (BGNF) and Sunflower Oil (SFO) Concentrations on Hysteresis Loop Area

4. Conclusions

BGNF was able to stabilized oil-in-water emulsion. Both BGNF and SFO affected the oil-droplet size and time-dependent properties of BGNF-stabilized emulsions. Mutual increase of BGNF and SFO in the emulsion did not have effect on the size of the oil droplets, however, independent increase of the ingredient seems to have a tremendous effect. The magnitude of hysteresis loop area quantification indicated that BGNF stabilized emulsions were time dependent and thixotropic in nature. The time dependent property was a function of both SFO and BGNF concentrations and their interactions. Emulsion containing 7% (w/w) BGNF and 40% (w/w) SFO had aggregated droplets network suspended in continuous BGNF phase (matrix). The very high droplet population (highest in this study) made it the most structured emulsion system. The high structure of the emulsion is as a result of the contribution of the SFO and BGNF concentrations and this manifested as very high viscosity and stability. Therefore, it is not unlikely that the high thixotropy of the emulsion is unrelated to high structural formation and emulsion stability. This study showed that BGNF stabilized emulsion possessed desirable characteristics which are a function of ingredients of formulation. Formulators should however know that prolong processing of BGNF stabilized emulsions could lead to breakdown/destabilization of emulsions.

REFERENCES

[1] Sarker D.K., 2013. What is an emulsion? Pharmaceutical Emulsions: A Drug Developer's Toolbag, 15-48.

[2] Samavati V., Emam-djomeh Z.A.H.R.A., Mohammadifar M.A., Omid M., Mehdinia A.L.I., 2012. Stability and rheology of dispersions containing polysaccharide, oleic acid and whey protein isolate. J. Texture Stud., 43(1), 63-76.

[3] Adeyi O., Ikhu-Omoregbe D., Jideani V., 2014. Emulsion Stability and Steady Shear Characteristics of Oil-In-Water Emulsion Stabilized by Gelatinized Bambara Groundnut Flour. Asian J. Chem., 26, 4995-5002.

[4] Dapčević Hadnađev T., Dokić P., Krstonošić V., Hadnađev M., 2013. Influence of oil phase concentration on droplet size distribution and stability of oil-in-water emulsions. Eur. J. Lipid Sci. Tech., 115(3), 313-321.

[5] Chanamai R., McClements D.J. 2000. Dependence of creaming and rheology of monodisperse oil-in-water emulsions on droplet size and concentration. Colloids Surf. A Physicochem. Eng. Asp., 172 (1), 79-86.

[6] Steffe J.F., 2001. Rheological methods in food process engineering, Freeman Press, East Lansing, MI.USA. pp. 86-91.

[7] Fischer P., Windhab E.J., 2011. Rheology of food materials. Curr. Opin. Colloid Interface Sci., 16(1), 36-40.

[8] Gao T, Hu H.H., Castañeda P.P., 2011. Rheology of a suspension of elastic particles in a viscous shear flow. J. Fluid Mech., 687-209.

[9] Gallegos C., Franco J.M., Partal P., 2004. Rheology of food dispersions. Rheology reviews. 19-65.

[10] Augusto P.E., Falguera V., Cristianini M., Ibarz A., 2012. Rheological behavior of tomato juice: steady-state shear and time-dependent modelling. Food Bioprocess Technol., 5, 1715-1723.

[11] Yang Y, Leser M.E., Sher A.A., McClements D.J., 2013. Formation and stability of emulsions using a natural small molecule surfactant: Quillaja saponin (Q-Naturale®). Food Hydrocoll., 30(2), 589-596.

[12] Murevanhema Y.Y., Jideani V.A., 2013. Potential of Bambara Groundnut (Vignasubterranea (L.) Verdc) Milk as a Probiotic Beverage—A Review. Crit. Rev. Food Sci., 53(9), 954-967.

[13] Caubet S., Guer Y.L., Grassil B., Omari K.E., Normandin E., 2011. A low-enegy emulsification batch mixer for concentrated oil-in-water emulsion. AlChe J., 57, 27-39.

[14] Perrechil F.A., Cunha R.L., 2010. Oil-in-water emulsions stabilized by sodium caseinate: Influence of pH, high-pressure homogenization and locust bean gum addition. J. Food Eng., 97(4), 441-448.

[15] Tcholakova S., Denkov N., Danner T., 2004. Role of Surfactant Type and Concentration for the Mean Drop Size during Emulsification in Turbulent Flow. Langmuir, 20, 7444-7458.

[16] Bellalta P., Troncoso E., Zuniga R.N., Aguilera J.M., 2012. Rheological and microstructural characterization of WPI-stabilized emulsions exhibiting time-dependent flow behaviour. Lwt-Food Sci. Technol., 46, 375-38.

[17] Koocheki A., Razavi S.M.A., 2009. Effect of Concentration and Temperature on Flow properties of Alyssum homolocarpum Seed Gum Solution: Assessment of Time Dependency and Thixotropy. Food Biophys., 4, 353-364

[18] Tarrega A, Duran L., Costell E., 2004. Flow behaviour of semi-solid dairy desserts. Effects of temperature. Int. Dairy J., 14, 345-353

[19] Izidoro D.R., Scheer A.D.P., Sierakowsk M.R., 2009. Rheological properties of emulsions stabilized by green banana (Musa cavendishii) pulp fitted by power law model. Braz. Arch. Biol. Technol., 52(6), 1541-1553.

[20] Camino N., Pilosof A.M.R., 2011. Hydroxypropymethylcellulose at the oil-waterinterface. Part II. Submicron-emulsions as affected by pH. Food Hydrocoll., 25(5), 1051-1062.

[21] Ibrahim N.H., Achudan S.N., 2011. Physical Properties and Stability of Emulsions as Affected by Native and Modified Yam Starches. WASET., 81, 470-474

[22] Wang B., Li D., Wang L. J., Adhikari B., Shi J., 2010. Ability of flaxseed and soybean protein concentrates to stabilize oil-in-water emulsions. J. Food Eng., 100(3), 417-426.

[23] Pal R., 1996. Viscoelastic properties of polymer-thickened oil-in-water emulsions. Chem. Eng. Technol., 51(12), 3299-3305.

[24] Payet L., Terentjev E.M., 2008, Emulsification and stabilization mechanisms of O/W emulsions in the presence of chitosan. Langmuir, 24(21), 12247-12252.

[25] Razavi S.M., Taghizadeh M., Ardekani A.S., 2010. Modelling the time-dependent rheological properties of pistachio butter. IJNRS., 1(1), 38-45 .

[26] Emadzadeh B., Razavi S.M., Mahallati M.N., 2012. Effects of fat replacers and sweeteners on the time-dependent rheological characteristics and emulsion stability of low-calorie pistachio butter: A response surface methodology. Food Bioprocess Tech., 5(5), 1581-1591.

[27] Köksoy A., Kılıç M., 2003. Effects of water and salt level on rheological properties of ayran, a Turkish yoghurt drink. Int. Dairy J., 13(10), 835-839.

[28] Mathias T.R.D.S., Carvalho-Junior I.C., Carvalho C.W.P.D., Servulo E.F.C., 2011. Rheological characterization of coffee-flavored yogurt with different types of thickener. Alimentos e nutricao, 22(4), 521-530.

[29] Alvarez M.D., Canet W., 2013. Time-independent and time-dependent rheological characterization of vegetable-based infant purees. J. Food Eng., 114(4), 449-464.