MODELLING THE TIME-DEPENDENT RHEOLOGICAL PROPERTIES OF OIL-IN-WATER EMULSIONS STABILIZED WITH GELATINIZED BAMBARA GROUNDNUT FLOUR

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Abstract

The contribution of emulsion main components to the time-dependent rheological properties of oil in water emulsion stabilized by gelatinized bambara groundnut flour (BGNF) was studied. Sunflower oil-in-water emulsions were prepared and stabilized with gelatinized BGNF. Weltman time-dependent rheological model was used to assess the time-dependent properties of oil-in-water emulsion stabilized with gelatinized BGNF and model's suitability was judged by the coefficient of determination, R^2, root mean square error, RMSE and standard error, SE. All emulsions were thixotropic in nature. The time-dependent behaviour of the emulsions was well described by Weltman model (high R^2 and low RMSE and SE values). The mean of parameter A and B values of Weltman model were in the range 11.68 – 315.44 Pa and 0.52 – 25.54 Pa respectively. Both the shear rate of shearing and emulsion main components (BGNF and Sunflower oil (SFO)) greatly influenced the time-dependent model parameters. The results provided the information on the influence of emulsion components and shear rate on rheological properties of BGNF-stabilized emulsions for product and process development.

Keywords: Bambara groundnut flour; Sunflower oil-in-water, rheological model, Weltman model, Hahns model

Introduction

Emulsions are a class of dispersed systems that consist of two immiscible liquids, with one of the liquid dispersed as small droplets in the other called continuous phase (McClements, 2004; Adeyi *et al.*, 2013). Emulsions can be classified as oil-in-water (O/W), water-in-oil (W/O) and oil-in-oil (O/O). Many food systems such as mayonnaise, milk, salad dressing etc. belong to these classes of emulsion. Food emulsions can destabilize via phenomena, such as creaming, droplet flocculation, coalescence and Ostwald ripening or combination of two or more phenomena. Food emulsions can therefore be made kinetically stable by adding an emulsifier which keeps the dispersed phase suspended in a continuous phase. This is made possible because of the intrinsic properties of the emulsifier. However, the unending demand for more natural products by the consumers and increasing legislations for safe and healthy food by governments has made synthetic emulsifiers in food systems increasingly unpopular.

Polymers of plant derivatives and of agricultural origins such as protein isolates and hydrolase (Wang *et al.*, 2010, Papalamprou *et al.*, 2006, Guo and Mu, 2011), native and modified starches (Moghaddam *et al.*, 2013, Ibrahim and Achunda, 2011, Bortnowska *et al.*, 2014, Izidoro *et al.*, 2008) , hydrocolloids (Wang *et al.*, 2011, Vega *et al.*, 2005, Erçelebi and Ibanoflu, 2009, Nor Hayati *et al*, 2009) and whole legume flours (Ma and Boye, 2013) have been used to improve properties of food emulsions. Like other pulses, 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 %, fiber content of 5.2 - 6.4, ash of 3.2 - 4.4 % and 2 % mineral (Murevanhema and Jideani, 2013). 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. Recently, gelatinized bambara groundnut flour dispersion (BGNF) has been reported to stabilize sunflower oil-in-water emulsion (Adeyi et *al*., 2014), however the time dependent rheological behaviour of such emulsion is yet to be reported.

Time-dependent characteristic of materials is related to internal structural destruction during flow and as such it is as a result of structural changes due to shearing. Time-dependent flow properties reflect the nature of a system's structure and can be as a result of viscoelasticity, structural changes, or both (Cheng and Evans, 1965). Time-dependent rheological behavior manifests as a progressive decrease in viscosity (break down of structure) when a material is sheared at a constant shear rate over a period of time and such structure may be recoverable when the system is left unperturbed (Tunan and Tiu, 1991). Such material is said to be thixotropic in nature. Time-dependent rheological behavior is therefore very important for understanding the products changes that occur during processing (Augusto *et. al.,* 2012). 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 (Steffe, 1996). Characterization of time-dependent rheological properties of food systems is also important in order to establish relationships between structure and flow and to correlate physical parameters with sensory attributes (Figoni and Shoemaker, 1983). Time-dependent rheological characteristics of semi-solid food materials have been described in the literature using the Figoni-Shoe maker model (Augusto *et al.*, 2012), Wetman model, and Hahn-Ree-Eyring model. Structural kinetic (SK) model was employed for studying time-dependent rheological behaviour of whey protein isolate-stabilized emulsions (Bellelta *et al.,* 2012).

The objectives of this study were to (1) characterize the time-dependent rheological properties of oil-in-water emulsion stabilized with gelatinized BGNF (2) determine the effect of emulsion ingredients (BGNF and SFO) on the time-dependent rheological behaviour (3) describe the thixotropy of the emulsions using popular Weltman models.

Experimental

Materials

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 hrs 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 % sunflower oil (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.

Emulsion preparation

Emulsions were prepared from a dispersed phase and a continuous phase (Adeyi et *al.*, 2014). The dispersed phase consisted of SFO and continuous phase was gelatinized bambara groundnut flour (BGNF) dispersion. BGNF dispersions of specific concentrations were prepared 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 minutes 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 minutes at the speed of 11000 rpm.

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 minutes. 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}. Each experiment was performed in duplicate. The time-dependent rheological properties were investigated by shearing oil-in-water emulsions stabilized with gelatinized BGNF samples at 20 minutes at constant shear rates of 50, 100 and 150 s^{-1}. Then the emulsion viscosity was measured as a function of shearing time. All measurements were done at a constant temperature of 20 ^{o}C. The data were modelled in order to describe the time-dependent flow properties of the BGNF-stabilized emulsions using popular Weltman model as represented in Eq. (1)

$$\tau = A + B\ \mathrm{In}(t) \qquad (1)$$

Where, τ is the shear stress, t is the shearing time, and A and B are constants that characterize a material's time dependent behavior. Parameter A represents the initial shear stress and parameter B; the time coefficient of thixotropic breakdown is the product of rate in breakdown of thixotropic structure and time of agitation at constant rate of shear (Koocheki and Razavi, 2009).

Data Analysis

Mean and standard deviation were calculated using IBM SPSS version 21 software. Curve fittings for time dependent rheological model was performed using the solver function in Microsoft Excel adopting the generalized reduced gradient (GRG2) nonlinear optimization code to determine the rheological parameters. The best fit line with minimum sum of square errors (SSE) was used as the sole criterion during curve fitting. The goodness of fit, R^2 was calculated as:

$$R^2 = 1 - \frac{SSE}{SST} \quad (2)$$

where SST is the total corrected sum of square (Chin *et al.*, 2009).

The best fit model was selected on the basis of high coefficient of determination R^2, low root mean square error (RMSE, equation 3) and standard error (SE, Eq, (4)).

$$RMSE = \left[\frac{\Sigma(Y_m - Y_c)^2}{n}\right]^{1/2} \quad (3)$$

$$SE = \left[\frac{\Sigma(Y_m - Y_c)^2}{n-1}\right]^{1/2} \quad (4)$$

Where Y_m is the measured value, Y_c is the calculated value for each data point, and n is the number of observations (Nindo *et. al.* 2007; Keshani *et. al.* 2012).

Results and discussion

Effect of constant shear decay on apparent viscosity of oil-in-water emulsion stabilized with gelatinized BGNF emulsion

Figures 1, 2 and 3 show the effect of shearing time on the apparent viscosity of concentrated oil-in-water emulsion containing 30, 35 and 40 % w/w SFO, stabilized with 5, 6 and 7 % w/w BGNF concentrations. The thixograms of the BGNF stabilized emulsions were different and dependent on the BGNF and SFO concentration as well as the rate of shearing. All the emulsion presented a time-dependent behavior when constant shear rates were applied. The apparent viscosity of the emulsion decreased with shearing time, which is a characteristic behaviour for thixotropic fluids. There was a greater dependency of apparent viscosity on time at the initial stage of the shearing process. The dependency of viscosity decreased as a function of shearing time, and it reached a steady state at about 800 seconds. Similar observation was reported for egg and eggless mayonnaise (Singla *et al.*, 2013), coffee flavored yogurt (Mathias *et al.*, 2011) and semi dairy desserts (Tarraga *et al.*, 2004). In addition, the viscosity tended to decay at a faster rate at higher shear rates towards a steady state viscosity and was lower than the steady state viscosity at low shear rates. This observation implied that the breakdown rate of samples under a shear field accelerates at high shear rates (Razavi and Karazhiyan, 2009). It is clear from Figures 1, 2 and 3 that BGNF concentration, SFO concentration and rate of shearing all affected the changes in the apparent viscosity from the initial value to final value. The higher changes were found between the initial and final values of apparent viscosity when the BGNF and SFO concentrations in the sample emulsions were increased. Emulsions that exhibit thixotropic properties do often contain oil droplets that are aggregated by weak forces and shearing causes the aggregated droplets to be progressively deformed and disrupted, thereby decreasing the resistance to flow and therefore causing a reduction in viscosity over time (Bellalta *et al.*, 2012). Furthermore, it is expected that the degree of oil droplet coalescence increase with increased oil content which led to an increment in the rate of structural breakdown. This result is in agreement with the report that changes in apparent viscosity when a constant shear rate is applied are higher when oil content in the emulsion increases (Bellalta *et al.*, 2012). Emulsion containing 40 % w/w SFO, stabilized with 7 % w/w BGNF showed the highest changes in apparent viscosity when sheared over a period of 1200 seconds. 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) as evident from initial backscattering result (data not reported) 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.

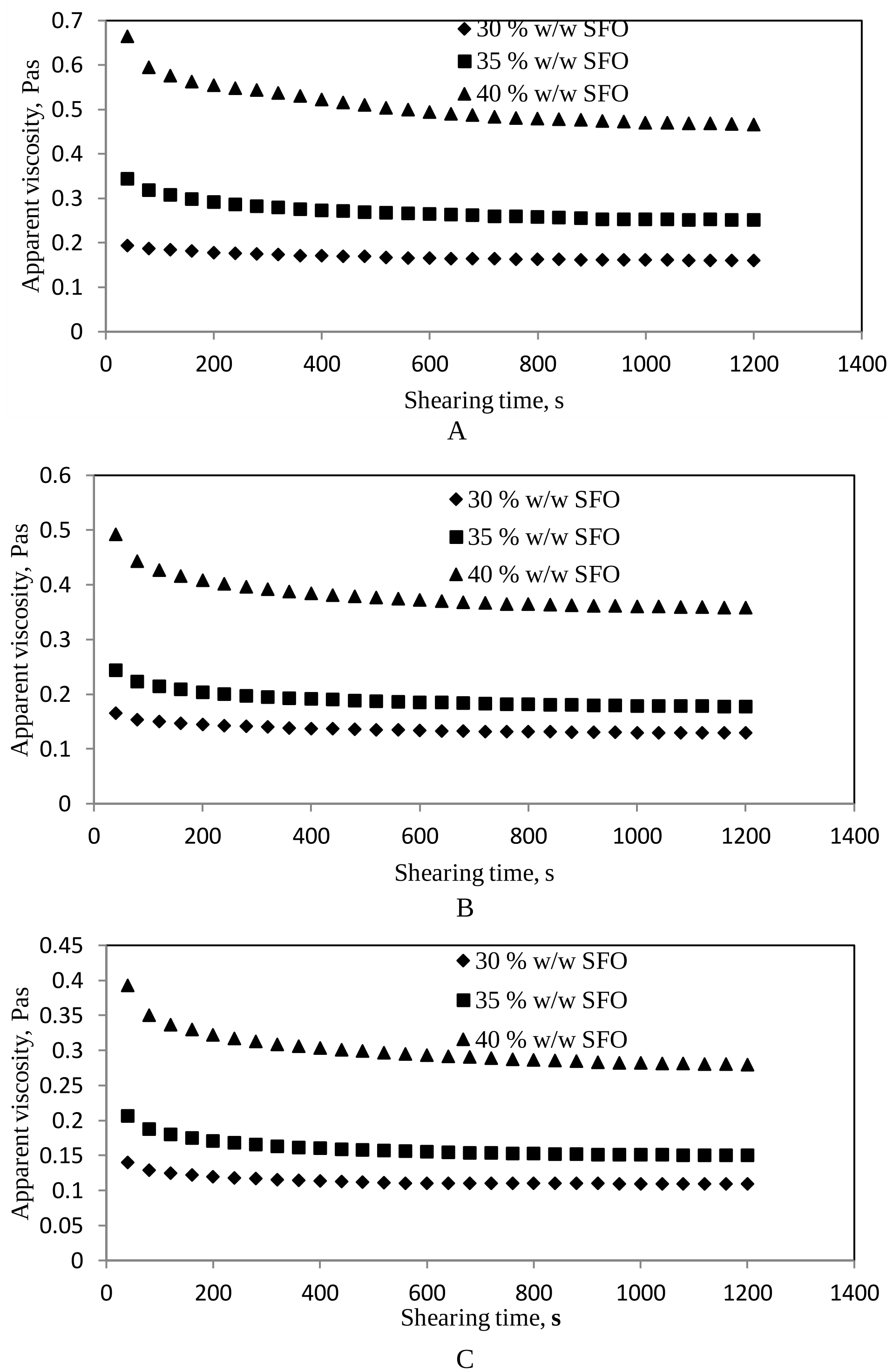

Figure.1 **Relationship between apparent viscosity and shearing time of emulsions stabilized with 5 % w/w Bambara groundnut flour (BGNF) at (A) 50 s^{-1} (B) 100 s^{-1} (c) 150 s^{-1}**

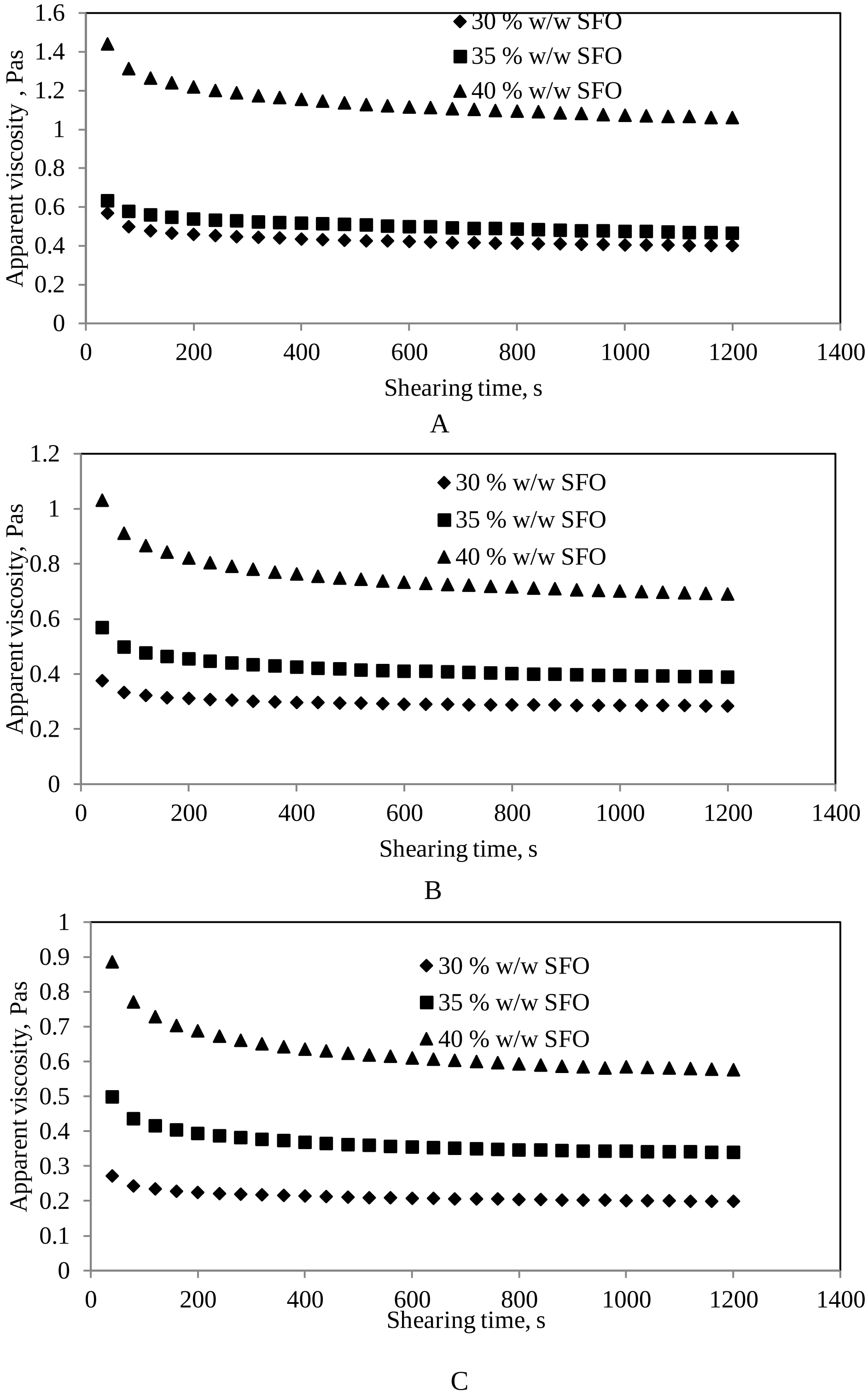

Figure 2. **Relationship between apparent viscosity and shearing time of emulsions stabilized with 6 % w/w Bambara groundnut flour (BGNF) at (A) 50 s^{-1} (B) 100 s^{-1} (c) 150 s^{-1}**

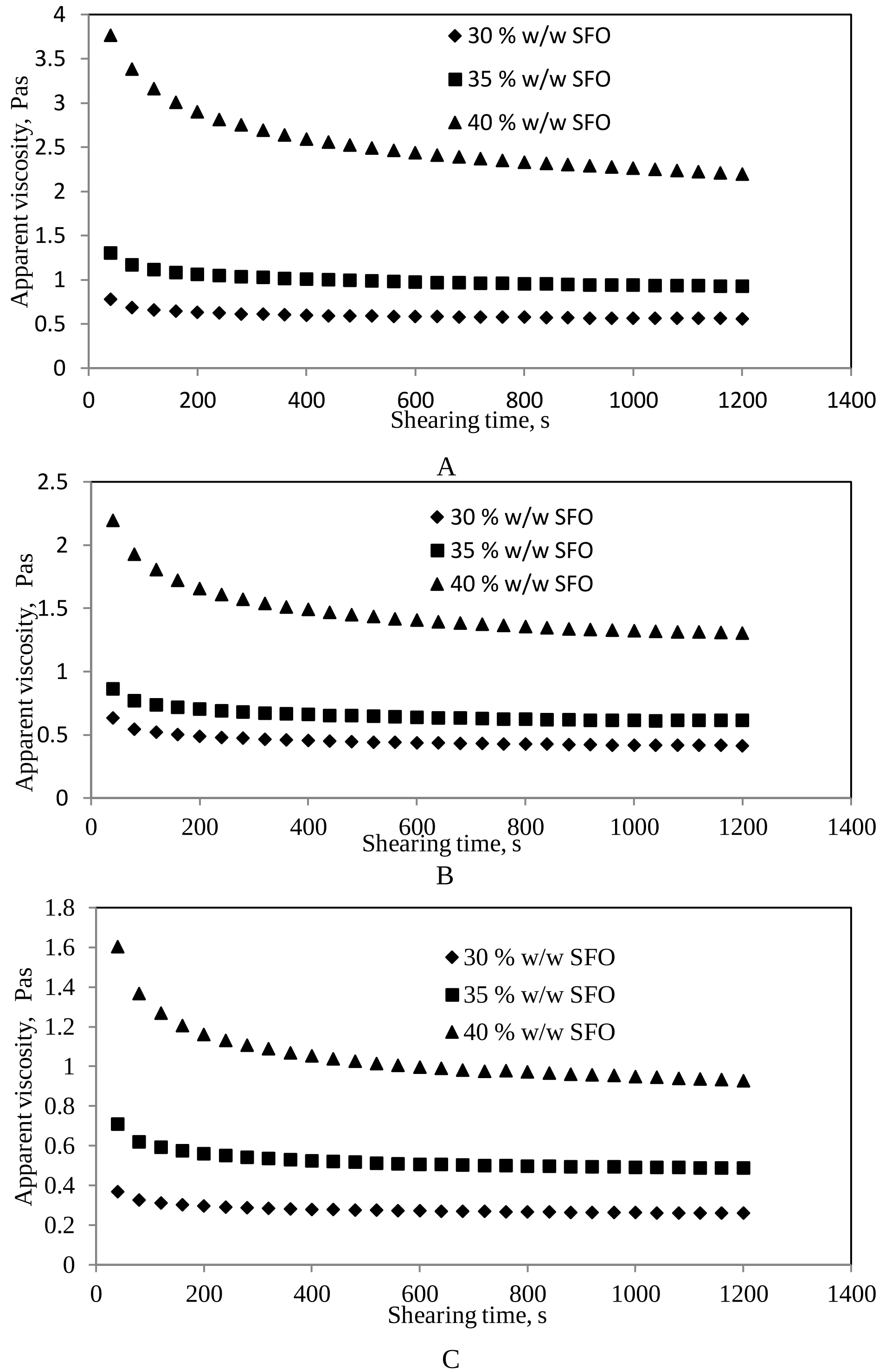

Figure 3. **Relationship between apparent viscosity and shearing time of emulsions stabilized with 7%w/w bambara groundnut flour (BGNF) at (A) 50 s^{-1} (B) 100 s^{-1} (C) 150 s^{-1}**

Thixotropic modeling of BGNF stabilized emulsion

Thixotropic modeling of BGNF stabilized emulsion using Weltman model

The observed time dependent behaviors of the samples were modeled using Weltman equation (Eq. (1)). Table 1 shows the Weltman model parameters at shear rates of 50, 100 and 150 s^{-1} as a function of BGNF and SFO concentration. Parameter *A* in the Weltman model is the quantification of initial stress of the emulsions while the parameter *B* value is an indication of the extent of thixotropy. A negative value of *B* measures how fast the shear stress drops from the initial value to final equilibrium value (Koocheki and Razavi, 2009). The mean of initial stress of the emulsions ranged from 11.68 to 315.44 Pa, while the extent of thixotropy ranged from 0.52 to 25.54 Pa. The high coefficient of determination (> 0.92) showed the appropriateness of Weltman model to describe the time dependent properties of the emulsions. Weltman model has been reported suitable to characterize the time dependent behaviour of food products like semi-solid dairy desserts (Tarrega *et al.*, 2004) and salad dressing (Paredes *et al.*, 1988). The coefficients of Weltman model obtained for BGNF stabilized emulsions were comparable to other food products reported in the literature. For example, pistachio butter with varying amount of lecithin and monoglyceride contents at 25 and 40 ºC was characterized and time dependent properties were modeled (Razavi *et al.*, (2010). The authors reported initial stress value (*A)* and extent of thixotropy (B) at 150 s^{-1} to be in the range of 376.43 - 2213.49 Pa and 21.62 - 235.19 Pa, respectively. In a related study conducted to characterize the time-dependent rheological properties of sesame paste/date syrup blend at 25 ºC (Razavi and Habibi-Najafi, 2006). Weltman parameter *A* and *B* were reported in the range of 189.2 - 730.6 Pa and 0.278 - 1.571 Pa, respectively. Parameter *A* and *B* for mayonnaise at 20 ºC and 10 s^{-1} was reported to fall in the range of 5.051 - 5.106 Pa and 0.0005 - 0.0007 Pa, respectively (Singla *et al.* (2013). The three-way analysis of variance showed that the coefficients of Weltman model were also significantly affected by concentration of BGNF, SFO and shear rate. In addition, other interaction effects including the interaction of BGNF, SFO and shear rate had significant effect on Weltman model parameters (Table 2). In all the emulsions, parameters *A* and *B* increased significantly as the shear rate increased. The increase in *B* with increase in shear rate is in agreement with works done on the rheological characterization of low fat sesame paste/date syrup blend and Salep respectively (Razavi *et al.*, 2008; Razavi and Karazhian, 2009).

Table 1. Weltman model parameter for emulsion stabilized with BGNF[1]

BGNF % w/w	SFO % w/w	$\gamma\ /s^{-1}$	A, Pa	$-$B, Pa	R^2	RMSE	SE
5	30	50	11.68 ± 1.37	0.52 ± 0.18	0.99	0.03	0.04
		100	19.78 ± 5.92	0.99 ± 0.44	0.99	0.10	0.10
		150	24.44 ± 6.52	1.20 ± 0.34	0.99	0.06	0.07
	35	50	21.65 ± 2.70	1.30 ± 0.28	0.99	0.12	0.12
		100	30.07 ± 1.95	1.79 ± 0.18	0.98	0.24	0.24
		150	37.73 ± 1.32	2.23 ± 0.11	0.96	0.39	0.39
	40	50	42.34 ± 1.62	2.72 ± 0.08	0.98	0.28	0.36
		100	59.77 ± 0.88	3.49 ± 0.29	0.97	0.52	0.53
		150	72.00 ± 1.47	4.36 ± 0.15	0.97	0.69	0.70
6	30	50	34.08 ± 1.73	2.02 ± 0.25	0.95	0.41	0.41
		100	42.73 ± 2.67	2.10 ± 0.34	0.95	0.43	0.43
		150	48.35 ± 2.20	2.69 ± 0.27	0.95	0.81	0.52
	35	50	38.98 ± 0.23	2.39 ± 0.22	0.99	0.16	0.16
		100	69.69 ± 4.23	4.45 ± 0.42	0.96	0.80	0.81
		150	92.00 ± 3.45	6.01 ± 0.62	0.95	1.18	1.20
	40	50	85.29 ± 8.43	4.99 ± 0.80	0.98	0.58	0.59
		100	129.58 ± 15.08	8.72 ± 1.40	0.97	1.35	1.36

		150	167.39 ± 6.40	11.77 ± 1.15	0.95	2.17	2.20
7	30	50	46.04 ± 2.03	2.61 ± 0.19	0.93	0.62	0.63
		100	57.35 ± 2.97	3.45 ± 0.34	0.94	0.70	0.72
		150	66.48 ± 6.25	3.97 ± 0.52	0.94	0.87	0.88
	35	50	79.22 ± 6.64	4.74 ± 0.35	0.96	0.80	0.82
		100	104.86 ± 8.89	6.50 ± 0.64	0.96	1.16	1.18
		150	129.08 ± 10.74	8.15 ± 0.90	0.93	1.82	1.85
	40	50	263.50 ± 27.10	21.99 ± 2.66	0.99	1.87	1.90
		100	296.53 ± 11.93	24.13 ± 0.60	0.97	3.39	3.42
		150	315.44 ± 4.29	25.54 ± 0.09	0.95	5.04	5.13

[1]γ / s^{-1} is the shear rate; *A* / Pa is the initial shear stress; *B* / Pa is the extent of thixotropy ;
R^2 is the coefficient of determination between the experimental data and predicted Weltman model data;RMSE refers to the root mean square error between the experimental and predicted model data; SE refers to the standard error value between the experimental and predicted model data.

It is however in contrast with the report that *B* decreased with increased shear rate for maize and waxy starch pastes (Nguyen *et al.*, 1998). Also parameters *A* and *B* increased significantly with increase in SFO and BGNF concentrations. For example, at a constant shear rate of 50 s^{-1} and SFO concentration of 30% (w/w), *A* and *B* values, increased from 11.68 ± 1.37 to 46.04 ± 2.03 Pa and 0.52 ± 0.18 to 2.61 ± 0.19 Pa for 5 to 7% w/w BGNF, respectively.

Table 2. Adjusted mean square for coefficient of Weltman model at different concentration of BGNF and SFO and shear rate[1]

Source of variation	df	A / Pa	p	B / Pa	p
BGNF	2	91866.50	<0.0001	590.88	<0.0001
SFO	2	106590.63	<0.0001	723.31	<0.0001
SRT	2	9139.41	<0.0001	42.71	<0.0001
BGNF*SFO	4	26843.58	<0.0001	243.39	<0.0001
SRT*BGNF	4	546.37	<0.0001	3.94	<0.0001
SRT*SFO	4	866.48	<0.0001	5.42	<0.0001
SRT*BGNF*SFO	8	155.25	0.0212	1.51	0.0114
Error	54	61.79	<0.0001	0.54	<0.0001

[1]A is the initial shear stress of the Weltman model; B is the extent of thixotropy.; BGNF equals bambara groundnut flour; SFO is sunflower oil; BGNF*SFO is the interaction between the bambara groundnut flour and sunflower oil; SRT*BGNF refers to the interaction between the shear rate of shearing and bambara groundnut flour; SRT*SFO refers to the interaction between shear rate and sunflower oil; SRT*BGNF*SFO is the interaction between the shear rate, bambara groundnut and sunflower oil.

Similar increase was observed at constant shear rates of 100 and 150 s^{-1} and at all the three levels of BGNF. The same trend was also observed when SFO concentration was increased from 30 to 40% w/w at constant BGNF concentration for all shear rates studied. In other word, increased BGNF and SFO concentration in the emulsion led to increased initial stress value which can be interpreted as an increased viscosity (Razavi and Habibi-Najafi, 2006, Razavi *et al.*, 2010). The larger the *B* value the greater the departure from thixotropy behavior (Razavi and Habibi-Najafi, 2006). Increased degree of thixotropy as a function of both emulsion components indicated increased structural breakdown. Emulsions formulated with lower concentrations of SFO and BGNF were less thixotropic at all shear rates. Emulsion formulated with 5 % w/w BGNF and 30 % w/w SFO possessed the lowest values of parameters *A* and *B*, while the highest values of parameters *A* and *B* belonged to sample formulated with 7 % w/w and 40% w/w SFO. High values of parameters *A* and *B* could be linked to high emulsion stability as a result of high structural network formed by the BGNF-SFO interactions of this emulsion (Razavi and Habibi-Najafi, 2006).

Conclusion

The time-dependent rheological properties of oil-in-water emulsion stabilized with gelatinized BGNF were characterized and modeled using the popular Weltman model. The effects of main emulsion ingredients (BGNF and SFO) and shear rate were determined and modeled. 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. Constant shearing of the BGNF stabilized emulsions at all the tested shear rates for 1200 s was enough to destroy the structures responsible for flow time dependency (rigidity) in all of the emulsions. The time-dependency of the BGNF stabilized emulsions was due to the increased interactions between the BGNF and SFO as their concentrations increased. Increased interactions between SFO and BGNF subsequently led to increased emulsion viscosity. It therefore seemed that the time dependent thixotropic characteristic of BGNF-stabilized emulsions is highly dependent on structural formation. Therefore, it is not unlikely that the high thixotropy of the emulsion is unrelated to high structural formation and emulsion stability.

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