Viscoelastic Behavior of Polysaccharide Blends Containing *Pereskia Bleo* (Tujuh Duri) Mucilage

Nurul Farhanah Mohd Aluwi, Nor Hayati Ibrahim, and Yusnita Hamzah

***Abstract*—**The viscoelastic properties of *Pereskia bleo* mucilage (PBM) and its binary blends with xanthan gum (XG), and locust bean gum (LBG) were studied. Blends of PBM, XG and LBG at six binary blends and native PBM dispersions were prepared at 2% (wt/wt) concentration. There was significant ($p < 0.05$) increase in storage modulus of PBM when blended with 75% XG at 10 rad/s and with 75% XG and LBG at 100 rad/s. Besides, there was also evident of improvement PBM blends with XG and LBG yet there were significant ($p < 0.05$) increase in loss modulus of PBM when blended with 75% XG and LBG at 10 rad/s compared native PBM dispersions alone. This study demonstrated improvement of viscoelastic properties of PBM by blending with other commonly known polysaccharides (XG and LBG) which might be attributed with their synergism.

Keywords*—Pereskia bleo* mucilage, viscoelastic properties, polysaccharide blends, modulus.

I. INTRODUCTION

MANY polysaccharides play an important role in the field of food industry due to their unique properties. In recent years there has been a widespread growth of research in the properties of food gum blends, widely used in the food industry, increasing the choice to the user, and improving some of the rheological properties of the individual components [1].

Xanthan gum and locust bean gum are widely used in food formulations, mainly as thickening agents but they are generally considered not to form gels on their own. Synergistic properties of xanthan gum with locust bean gum are well known and widely exploited by the food industry. It is noteworthy that gelation occurs from the mixture of these two non-gelling components. The storage modulus of xanthan/locust bean gum systems could be expected to be increased while the mannose/galactose ratio of the locust bean gum fractions increased. All these observations show that the xanthan/galactomannan interactions are strongly affected by the fine structure of the galactomannan. Recently, synergistic mechanism between yellow mustard mucilage and galactomannans has been proposed [2]. This synergism is due to associative interactions or formation of junction zones between the unsubstituted region of galactomannan backbone and β- [1→ 4]-D-glucosidic backbone of yellow mustard mucilage.

Pereskia bleo is a type of tropical herbs which has long been used for its medicinal benefit among Malays and is also known to contain complex polysaccharide called mucilage. Mucilage and plant gums are widely used in the food industry as thickening agent, gelling agents and stabilizers [3]. However, crude mucilage from *P.bleo* has been found to contain low viscosity and there is no significant change on its viscosity when solution concentration increased [4]. In contrast, in the research done by [5] using *Opuntia ficus indica*, increasing the mucilage concentration in the dispersion, causing the increases in viscosity. Based on a preliminary finding [4], Infrared spectra of PBM suggests that this mucilage mainly composed of β- [1→ 4]-D-glucosidic backbone attributable to IR band at approximately 872 cm^{-1}. In addition, PBM exhibits a high water holding capacity (461.87%) [6] which reflects it is ability to form junction zones by hydrogen bonding between –OH and water molecules. Due to these facts, it is reasonable that PBM will positively interact with other polysaccharide including galactomannans and thus improve its rheological properties.

The aim of the present work was to investigate viscoelastic properties of PBM and its binary blends with XG and LBG. The viscoelastic behavior of these binary blends has been primarily investigated using oscillatory amplitude and frequency measurements, in comparison with native PBM dispersions.

Nurul Farhanah Mohd Aluwi is with the School of Food Science and Technology, Universiti Malaysia Terengganu, 21030 Kuala Terengganu, Terengganu, Malaysia (corresponding author’s phone: 609-6683644 ; fax: 609-6683434; e-mail: nurul.farhanah2008@gmail.com).

Nor Hayati Ibrahim is with School of Food Science and Technology, University Malaysia Terengganu, 21030 Kuala Terengganu, Terengganu, Malaysia (e-mail: yati@umt.edu.my).

Yusnita Hamzah is with School of Food Science and Technology, University Malaysia Terengganu, 21030 Kuala Terengganu, Terengganu, Malaysia (e-mail: yusnita@umt.edu.my).

II. MATERIALS AND METHODS

A. Materials

The leaves of *P.bleo* were collected from control sites at Jabatan Pertanian, Kelantan, Malaysia. Sodium hydroxide, saturated barium hydroxide, acetone, XG and LBG were purchased from Sigma (Sigma-Aldrich, St. Louis, MO).

B. Extraction and Purification of Mucilage

Crude mucilage from leaves of *P.bleo* was extracted by using 0.14 M NaOH solution at temperature, 70°C. Saturated barium hydroxide was used as a purification medium. Crude mucilage was dissolved in distilled water and stirred for 30 minutes at 70°C, 250 rpm with magnetic stirring. The solution was precipitated with chemical medium, saturated barium hydroxide with ratio 1:3 (weight of crude mucilage: weight of chemical used). The sample solution was stirred for 5 minutes at 250 rpm and allowing the sample-solvent slurry to stand for 30 minutes. The precipitate was separated by a centrifuge at 10,000 rpm for 15 minutes. The precipitate were collected by filtration was washed twice with saturated barium hydroxide and acetone. Purification was done after it was dried in the oven overnight at 40°C [6,7].

C. Preparation of Polysaccharide Blends

Blends of PBM, XG and LBG were prepared at six binary blends and native PBM dispersions as a control. The polysaccharide blends (2% w/w) concentration were first individually prepared by dispersing polysaccharide powder in deionized distilled water with vigorous stirring at room temperature (25±1°C) by a magnetic stirrer for 5 minutes, 450 rpm with binary blends (six binary blends). The polysaccharide solution were heated at 80°C for 2 hours, 450 rpm on a thermo stated hot-plate, while continuously stirred and the solutions were cooled to room temperature and left overnight to ensure a complete hydration. Suspensions were homogenized by using ultrasonic homogenizer at amplitude 20 for 2 minutes and continue with amplitude 30 for 2 minutes and amplitude 40 for 2 minutes [8,9].

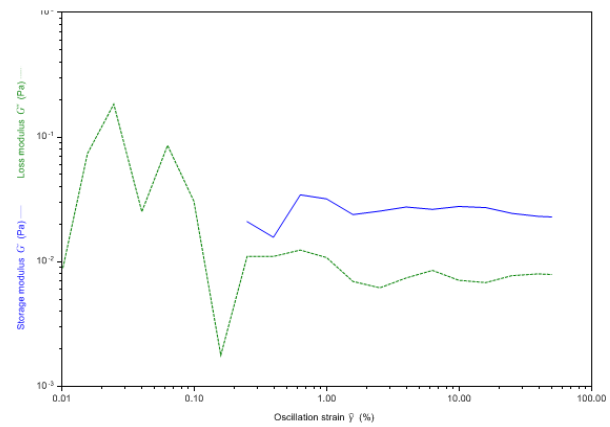
D. Rheological Measurements

Rheological measurements were performed on a Discovery HR-2 hybrid rheometer equipped with a thermo cube solid state cooling system (TA instrument-water LLC, Lukens Drive, Nest Castle) at room temperature (25 ± 1°C). The geometry used was 40 mm cone plate. Approximately 0.75 ml of polysaccharide blends was loaded onto the plate and allowed to conditioning for 600 s. For oscillation amplitude sweep, the test parameters were programmed to frequency 1 Hz, gap 46 micrometer, temperature 25 °C, and strain 0.01 - 50%. Oscillation strain for emulsion was identified in order to ensure that the frequency sweep measurements were performed within the linear viscoelastic region. The test parameters in oscillation frequency sweep test were programmed to gap 46 micrometer, temperature 25 °C, angular frequency 1.0 to 100 rad/s and strain was followed the value of oscillation strain that identified in oscillation amplitude sweep test [10]. Significant differences among samples were further tested using a One- way ANOVA with Tukey's Multiple Comparison at significant level of $p < 0.05$.

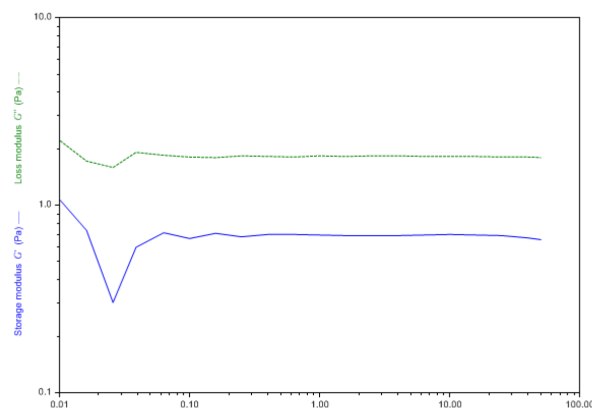
III. Results And Discussions

Fig. 1 demonstrates a representative rheogram of oscillation amplitude sweep test for polysaccharide blends at six binary blends and native PBM dispersions as a control. According to Fig. 1 : representative rheogram of oscillation amplitude sweep test for (b) 50% PBM and 50% XG, (c) 25% PBM and 75% XG and (f) 25% PBM and 75% LBG, the critical oscillation of binary polysaccharide blends was 10%. Below 10% oscillation strain, the structure is intact, the polysaccharide blends behaves solid-like (soft gel), and G' is higher than G", indicating that the polysaccharide blends was highly structured. Increasing the oscillation strain above the critical strain, the network structure of the polysaccharide blends was disrupted.

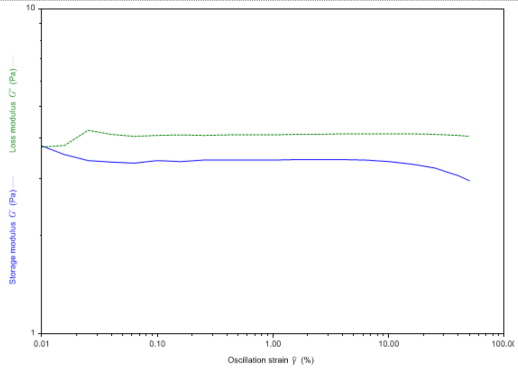
When the value of G' is higher than G", the material is exhibits gel character, viscoelastic solid and a certain rigidity as shown in Fig. 1: representative rheogram of oscillation amplitude sweep test for (a) 100% of PBM. In contrast, the material exhibits the character of a liquid or viscoelastic liquid in the LVE region when the value of G" is higher than G' as shown in Fig. 1: representative rheogram of oscillation amplitude sweep test for (b) 50% PBM and 50% XG, (c) 25% PBM and 75% XG, (d) 75% PBM and 25% XG, (e) 50% PBM and 50% LBG, (f) 25% PBM and 75% LBG and (g) 75% PBM and 25% LBG.



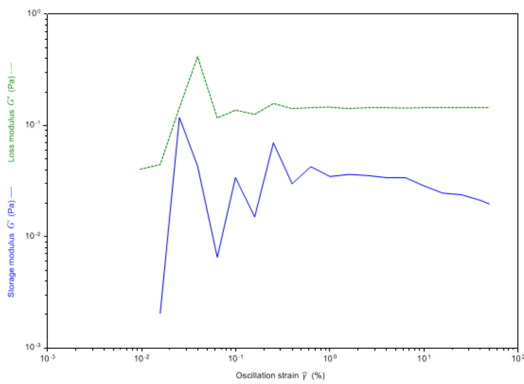
(a)



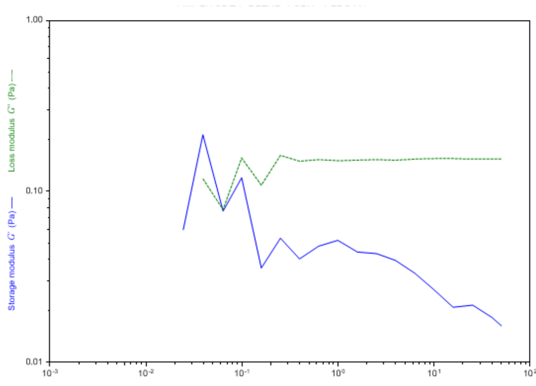
(b)



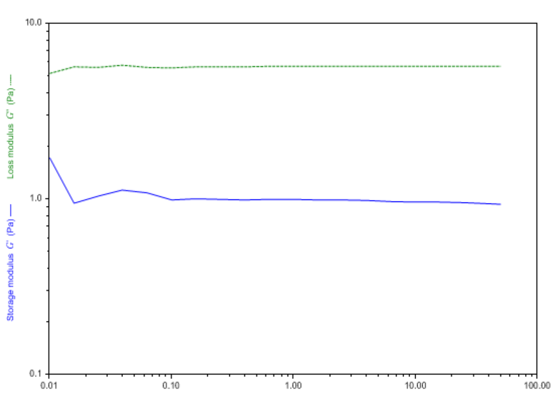
(c)



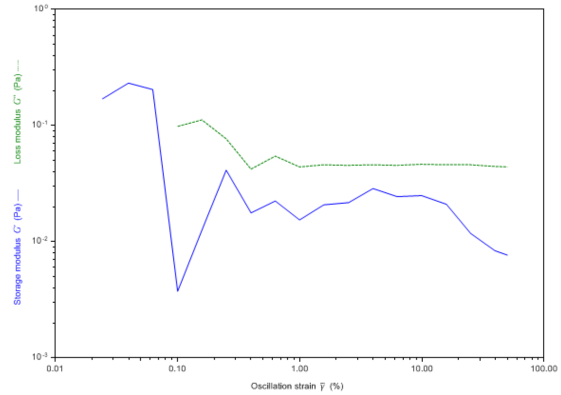
(d)



(e)



(f)



(g)

Fig. 1 : Representative rheogram of oscillation amplitude sweep test for (a) 100% PBM, (b) 50% PBM and 50% XG, (c) 25% PBM and 75% XG, (d) 75% PBM and 25% XG, (e) 50% PBM and 50% LBG, (f) 25% PBM and 75% LBG and (g) 75% PBM and 25% LBG

High-viscosity materials with entangled molecule chains but without any chemical network or physical network of forces are showing this behavior. In addition, the behavior is sometimes called at the 'gel point' or 'crossover point' when the value of G' and G" in the LVE region are balanced. It shows a material behaving at the border line between liquid and gel-like but this state is seldom found for samples in industry [11].

Oscillation amplitude sweep test were carried out to ensure that the oscillation frequency sweeps were performed within the LVE region. Based on the range of oscillation strain within the LVE region, the constant oscillation strain value that used in the oscillation frequency sweep was 0.25 % for (100% PBM), (50% PBM and 50% LBG), (75% PBM and 25% LBG) and (75% PBM and 25% XG). While, 0.5% for (50% PBM and 50% XG), (25% PBM and 75% XG) and (25% PBM and 75% LBG).

Table I demonstrates the oscillation strain within the LVE region for polysaccharide blends containing PBM, XG and LBG. In general, the LVE region for polysaccharide blends was between 0.03% - 10%. The length of the LVE region is a measure of stability. If the tested sample has shorter LVE region, it will be breaks down more easily.

TABLE I
OSCILLATION STRAIN WITHIN THE LINEAR VISCOELASTIC REGION FOR DIFFERENT POLYSACCHARIDE BINARY BLENDS

Formulation	Range oscillation strain within the linear viscoelastic region (%)
100% PBM	0.04 - 1.00
50% PBM + 50% XG	0.20 - 10.00
25% PBM + 75% XG	0.03 - 10.00
75% PBM + 25% XG	1.00 - 7.00
50% PBM + 50% LBG	0.20 - 1.00
25% PBM + 75% LBG	0.10 - 10.00
75% PBM + 25% LBG	4.00 - 9.00

All value given are range of triplicate results

In a frequency sweep, measurements are made over a range of oscillation frequencies at constant oscillation amplitude. Below the critical strain, the storage modulus, G' is often nearly independent of frequency, as would be expected from a structured or solid-like material. The more frequency dependent is the elastic modulus; the material is more fluid-like. Fig. 2 and 3 show the dependency of storage modulus and loss modulus values on frequency of polysaccharide blends at binary blends of PBM, XG and LBG respectively. The difference between storage modulus (G') and loss modulus (G") value was small at lower frequency ranges while the differences was high at high frequency.

From Fig. 2 and 3, the most parallel between G' and G" values with angular frequency was the polysaccharide blends with 25% of PBM and 75% of LBG. This shows that the polysaccharide blends with 25% of PBM and 75% of LBG was the most stable polysaccharide blends compare to native PBM dispersions alone. This can be well supported by [12], who reported that a mechanical spectrum of a stable emulsion including polysaccharide blends with greater internal strength shows a storage modulus, G' higher than its loss modulus, G" and both modulus should be almost parallel throughout the observed frequencies. However, 25% of PBM and 75% of LBG have a loss modulus, G" higher than storage modulus, G' for the present case but still both modulus of 25% PBM and 75% LBG almost parallel throughout the observed frequencies.

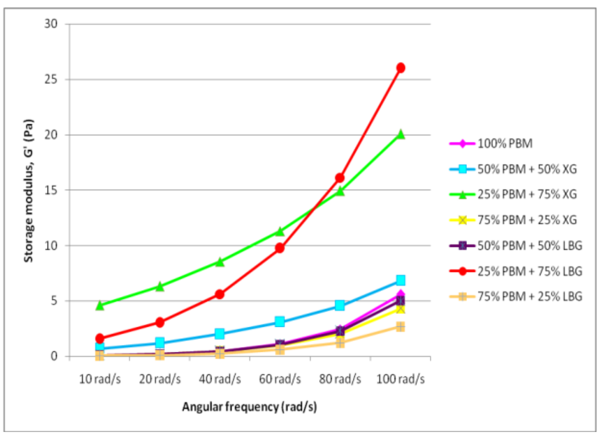


Fig. 2 Storage modulus (G') of polysaccharide blends at different binary blends. All values given are the means of triplicate results. Standard deviation range was 0.02 – 6.78 Pa

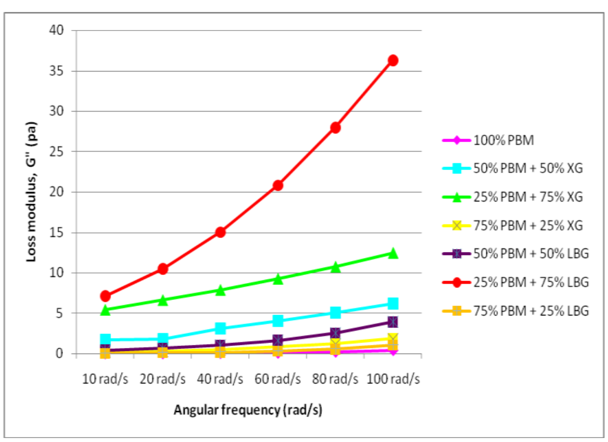


Fig. 3 Loss modulus (G") of polysaccharide blends at different binary blends. All values given are the means of triplicate results. Standard deviation range was 0.01 – 7.35 Pa

According to Fig. 2, it can be seen that the polysaccharide blend with 25% of PBM and 75% LBG showed the highest G' while the polysaccharide blend with 75% of PBM and 25% of LBG showed the lowest G'. Based on [13], a higher G' indicates a more elastic nature and higher recovery. Therefore, the polysaccharide blends with 25% of PBM and 75% of LBG is said to be more elastic and have a higher recovery compare to others polysaccharide blends that have lower G'.

The storage and loss modulus are the subtle descriptions of the viscoelastic properties and microstructure of the system. Storage modulus is a measure of the energy which is stored during the vibration procedure. As observed for the storage modulus, there was significant ($p < 0.05$) increase in storage modulus of PBM when blended with 75% XG at selected angular frequency i.e. 10 rad/s. Besides, there was also improvement PBM blends with XG and LBG where was significant ($p < 0.05$) increase in storage modulus of PBM when blended with 75% XG and LBG compare to native PBM dispersions at 100rad/s as depicted in Table II.

TABLE II
STORAGE MODULUS (G') OF DIFFERENT POLYSACCHARIDE BINARY BLENDS AT 10 RAD/S AND 100 RAD/S

Sample (2%)	G' (Pa) at 10 rad/s	G' (Pa) at 100 rad/s
100% PBM	0.09 ± 0.05^{c}	5.58 ± 1.42^{b}
50% PBM + 50% XG	0.71 ± 0.46^{bc}	6.82 ± 4.44^{b}
25% PBM + 75% XG	4.62 ± 0.70^{a}	20.07 ± 5.44^{a}
75% PBM + 25% XG	0.09 ± 0.03^{c}	4.31 ± 3.10^{b}
50% PBM + 50% LBG	0.09 ± 0.02^{c}	5.02 ± 1.61^{b}
25% PBM + 75% LBG	1.57 ± 0.59^{b}	25.98 ± 6.78^{a}
75% PBM + 25% LBG	0.05 ± 0.04^{c}	2.68 ± 3.24^{b}

Data represent mean and standard deviation from three independent replications (n = 3) for each sample. Value with same superscripts letter within same column are not significantly different (p>0.05)

Loss modulus represents the energy dissipated during the vibration procedure. A one way ANOVA statistical analysis demonstrated that there was significant ($p < 0.05$) increases in loss modulus of PBM when blended with 75% XG and LBG at selected angular frequency i.e. 10 rad/s as shown in Table III. However, at selected angular frequencies, 100 rad/s, only 25% of PBM with 75% of LBG gave a significant ($p < 0.05$) increases in loss modulus compared to other polysaccharide blends. For the main effect, different blends of polysaccharide blends containing PBM was significantly affected the loss modulus at 10 and 100 rad/s. It was shown to be much stronger than 100% native PBM dispersions alone.

TABLE III
LOSS MODULUS (G") OF DIFFERENT POLYSACCHARIDE BINARY BLENDS AT 10 RAD/S AND 100 RAD/S

Sample (2%)	G" (Pa) at 10 rad/s	G" (Pa) at 100 rad/s
100% PBM	0.02 ± 0.01^{b}	0.42 ± 0.02^{c}
50% PBM + 50% XG	1.73 ± 0.66^{b}	6.18 ± 1.22^{bc}
25% PBM + 75% XG	5.42 ± 0.27^{a}	12.45 ± 0.12^{b}
75% PBM + 25% XG	0.23 ± 0.01^{b}	1.85 ± 0.05^{c}
50% PBM + 50% LBG	0.41 ± 0.19^{b}	3.93 ± 1.50^{c}
25% PBM + 75% LBG	7.14 ± 1.95^{a}	36.34 ± 7.35^{a}
75% PBM + 25% LBG	0.06 ± 0.02^{b}	1.05 ± 0.03^{c}

Data represent mean and standard deviation from three independent replications (n = 3) for each sample. Value with same superscripts letter within same column are not significantly different (p>0.05).

IV. Conclusion

This study has shown that the viscoelastic properties of PBM were improved by blending with other polysaccharides, 75% XG and LBG. Thus, better functionality of this mucilage can be achieved when applied in related food system.

Acknowledgment

This work was fully supported by research grant of FRGS, Grant no. 59284.

References

[1] J. A. Lopes da Silva, M. P. Gonçalves, and M. A. Rao, "Viscoelastic behaviour of mixtures of locust bean gum and pectin dispersions," *Journal of Food Engineering 18*, pp. 211-228, Feb. 1992.

[2] Y. Wu, W. Cui, N. A. M. Eskin, and H. D. Goff, "Stress relaxation in synergistically associated polysaccharides: Galactomannans and a non-pectic polysaccharide fraction from yellow mustard mucilage," *Carbohydrate Polymers 84*, pp. 984-989, Dec. 2010.

[3] M. A. Nurul Farhanah, I. Nor Hayati, and T. H. Ng, "Chemical and functional properties of purified mucilage from *Pereskia bleo* (Tujuh Duri)," Proceedings 13th Symposium Malaysian Society of Applied Biology, 8-10 June 2014, The Legend Resort Cherating Pahang, Malaysia 2014., 08/06/2014, 10/06/2014.

[4] T. H. Ng, "Extraction and characterization of mucilage from *Pereskia bleo* (Tujuh Duri) (Unpublished work style)," unpublished.

[5] N. Gebresamuel, and T. Gebre-mariam, "Comparative physico-chemical characterization of the mucilages of two cactus pears (*Opuntia spp*.) obtained from mekelle, northern Ethiopia," *Journal of Biomaterials and Nanobiotechnology 3*, pp.79-86, Dec. 2011.

[6] T. H. Ng, and I. Nor Hayati, "Extraction and characterization of mucilage from leaves of *Pereskia bleo* (Rose Cactus)," *Journal of Food Technology and Industry*, ISSN 1979-7788, Vol. XXX111 (2), pp. 210-216, Dec. 2012.

[7] B. T. Amid, and H. Mirhosseini, "Effect of different purification techniques on the characteristics of heteropolysaccharide-protein biopolymer from Durian (*Durio zibethinus*) seed," *Molecules 17*, pp. 10875-10892, Sep. 2012.

[8] I. Nor Hayati, Y. B. Che Man, C. P. Tan, and I. Nor Aini, "Droplet characterization and stability of soybean oil/ palm kernel olein O/W emulsions with the presence of selected polysaccharides," *Food Hydrocolloids 23*, pp. 233-243, Jan. 2008.

[9] I. Nor Hayati, Y. B. Che Man, C. P. Tan, and I. Nor Aini, "Mixture design experiment on flow behavior of O/W emulsions as affected by polysaccharide interactions," *World Academy of Science, Engineering and Technology 43*, pp. 1000-1006, 2010.

[10] R. P. Vianna-Filho, C. L. O. Petkowicz, and J. L. M. Silveira, "Rheological characterization of O/W emulsions incorporated with neutral and charged polysaccharides," *Carbohydrate Polymers 93*, pp. 266-272, May. 2012.

[11] T. G. Mezger, "The rheology handbook: for users of rotational and oscillatory rheometers,"2nd ed., Germany Vincentz Network GmbH & Co., 2006, pp. 124-247.

[12] R. Brummer, Rheology Essentials of Cosmetic and Food Emulsions. Berlin: Springer, 2006, pp. 17-23.

[13] P. Tarancon, M. J. Hernandez, A. Salvador and T. Sanz, "Relevance of creep and oscillatory test for understanding how cellulose emulsions function as fat replacers in biscuits," *LWT- Food Science and Technology 62*, pp. 640-646, June. 2014.