

# Effect of Dietary Fibre and Thermal Condition on Rice Bran Wax Oleogels for Biscuits Preparation

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In this work sunflower oil (SFO) oleogels were obtained using rice bran wax (RBW) at 4.5 and 5.5% wt/wt. Bamboo fibre 40 µm (BF40) at 0.5% was investigated as structuring agent. The effect of fibre addition and cooling temperature (0, 4 and 25°C) on gel properties was evaluated compared to control samples prepared without fibre and at same cooling conditions. Rheological (both in rotational and oscillatory mode), texture and differential scanning calorimetry tests were carried out to assess thermal and structural parameters of gels. Oleogelatinization modified the rheological behaviour of samples, shifting from a typical Newtonian oil trend to a pseudo plastic non-Newtonian behaviour in gels. The gels resulted sensitive to temperature changes since a lower cooling temperature led to a structure weakening. Nevertheless, the fibre addition determined an increase of gel strength especially at 4°C demonstrating the structuring action of bamboo. This latter result was particularly evident on macroscopic scale by texture analysis. In fact, samples prepared at 25°C and without fibre addition showed the highest hardness, gel strength and work of penetration values. All parameters reduced by decreasing in cooling temperature down to 0°C, from which point it was not possible to carry out any more measure. Fibre inclusion determined soaring texture values, where samples prepared at 4°C are comparable to control at 25°C. Gels obtained through cooling at 4°C were used in biscuits preparation, with control recipes with only SFO or with butter. Texture profile analysis on dough revealed that the inclusion of fibre reduces dough firmness affecting positively texture parameters, better resembling the butter control. Biscuits prepared with only SFO showed higher spread values than butter cookies. The use of gelled material reduced the spread ratio even not as much as in butter cookies. Biscuits prepared with fibre-added gels possess a texture closer to butter control, even if all the gel cookies behaved as brittle material with lower fracturability indexes compared to butter ones.

## 1. Introduction

In bakery products lipids have several functions (Demirkesen & Mert, 2019). They act as fillers of gluten structure or interact in protein-starch networks improving the flow properties of dough. Moreover, in leavened products their emulsion activity stabilizes the air bubble formation and distribution that is crucial for volume increase (Kim et al., 2017) and in some cases, as croissant, Danish and puff pastry, they are essential to maintain the layer structure during mechanical lamination (Blake & Marangoni, 2015). The lubricant properties of lipids positively affect also the final texture reducing the chewiness. Fats also play fundamental roles in filling, toppings and creams used to décor or fill to enhance visual appealing but also the technological and in-mouth characteristics of bakery products (Tanti et al., 2016).

Recently, wide attention was paid on oleogelation as advanced and alternative route of structuring (Patel & Dewettinck, 2016). Oleogels are innovative lipid-based system in which liquid oils are transformed into gel form without chemical/enzymatic reactions. The physical transition takes place due to the addition of gelling agents capable of rearranging their-self in 3-D networks entrapping the liquid phase. The great potential of oleogelated system is related to a fully substitution of solid fat with gels containing unsaturated or poly-unsaturated edible oil in their native form (Kim et al., 2017). The gelling agents preserve the nutritional characteristics of the oil and could even preserve them from.

There is a broad variety of gelling agents that operate, alone or in combination, throughout different mechanisms. Ones of the most used are natural waxes (Blake & Marangoni, 2015; Oh et al., 2017). Those materials are widely used for their gelling capacity at low concentration and the capability to form thermo-reversible gels. Moreover, they are cheap and easy to use by direct dispersion in the liquid phase. Among organogelators used in direct dispersion, rice bran wax (RBW) has been proved to be one of the most promising novel ingredients due to its ability to structure oils at very low concentrations. Furthermore, RBW is an innovative and sustainable ingredient produced in large quantities as byproduct of rice bran oil production (Limpimwong et al., 2017). The gel strength is strictly related to gelation conditions and wax concentration, however increasing the percentage of wax strongly affects product taste. To improve the mechanical properties of lipid system reducing gelling, agent concentration dietary fibres were included in formulation. Dietary fibre is well known for its outstanding properties as thickening agents in water-based systems. However, plant fibres have many free hydroxyl groups at the molecular level that easily bond with oil or water (Bazargan et al., 2014). Therefore, they generally exhibit a good affinity for both oil and water. A potential stabilizing and structuring capacity of dietary fibre could be exploited for oleogels using their oil-sorption properties. The aim of this study was to investigate the effect of cooling conditions and bamboo fiber addition on sunflower oil (SFO) oleogels structured using RBW at 4.5 and 5.5% wt/wt. The gel formulations were used as butter replacement in biscuits preparation. Rheological (both in rotational and oscillatory mode), texture and differential scanning calorimetry tests were carried out to assess thermal and structural parameters of gels, doughs and biscuits.

## **2. Material and Methods**

### **2.1 Materials**

Sunflower oil (Vita D'Or) was purchased from a local grocery store. Bamboo fibre 40  $\mu\text{M}$  (BF40) was kindly offered by Prodotti Gianni Spa (Italy) and RBW was purchased from YumiBio S.r.L. The butter and the other ingredients used for biscuits formulation—wheat flour, sugar, baking powder, eggs—were purchased from local stores.

### **2.2 Oleogel and Biscuits Dough Preparation**

Oil (150 g) was poured in a beaker and heated up to 75°C using a heating plate. Once at 75°C, RBW at 4.5% (wt/wt) or 5.5% (wt/wt) and fibre at 0.5% were added. Then, the temperature was further increased up to 85°C to allow wax melting and maintained for 2 min to assure the complete mixing. The mixture was then poured in Petri dish and cooled at 0°C, 4°C and 25°C for 1 h and stored at 4°C for 24 h before being analysed. Then, SFO oleogels prepared at 4.5% and 5.5% of RBW with and without the addition of BF40 were re-prepared at 4°C cooling temperature and used for complete substitution of butter in biscuits dough. SFO and butter were used for control biscuits. The recipe included eggs, wheat flour, sugar and baking powder. All ingredients were mixed using a spiral mixer Tecknostamp Tk 20 at increasing speed from 33 to 66 rpm for 13 min. The dough was cut in disc shape (0.5 cm height, 5 cm diameter). Biscuits were baked in a semi-industrial oven Wind+ 6040/5 (Polin Italy) for 13 min at 180°C.

### **2.3 Characterization of Oleogel Systems**

#### **Rheology**

The apparent viscosity was measured by using a controlled-stress rheometer (MCR 302, Anton Paar, Austria) provided with 50 mm diameter parallel plates and 1 mm gap. Before measurement, 5 min holding time was selected in order to release loading-induced stress (Kirbas et al., 2019). Measurements were performed at 25 °C  $\pm$  0.1. The shear rate was set between 0.01-100  $\text{s}^{-1}$  and the results were given in Pa·s.

#### **Texture**

Samples were analysed using a texture analyser TVT 6700 (Perten Instruments, Sweden) according to the penetration test by Yilmaz and Ogutcu (2014), with slight modification. A cylindrical probe of 5 mm was used to penetrate the sample placed in a petri dish (7.5 cm diameter and 1 cm height) up to a depth of 5 mm, with 1 mm/s crosshead speed. The test was carried out at room temperature (23 °C  $\pm$  2). The highest measured force value and the magnitude of force detected at 3 mm was defined as gel hardness and gel strength, respectively, and expressed in Newtons (N). The area under the curve is defined as work of penetration and expressed in N·mm. Test were carried out five times.

## Thermal analysis

The assay was performed using a micro differential scanning calorimeter (microDSC) (Setaram, France). The gel (0.8 mg) was weighted and placed in hastelloy capsules. Samples were heated from 5°C to 100°C at 0.7°C/min, maintained at 100°C for 15 min and cooled to 25 °C at 1°C/min (Patel & Dewettinck, 2015).

## 2.4 Characterization of Dough and Biscuits

### Spread ratio and Texture

For each recipe, it was measured diameter (D) and maximum height (H) of six biscuits and the spreadability index (SI) was calculated as  $SI\% = D/H$ .

Texture profile of doughs and biscuits was evaluated with a texture analyser TVT 6700 (Perten Instruments, Sweden). For the dough, the instrument was equipped with a cylindrical mandrel with a diameter of 6 mm (P/6) according to Onacik-Gur and Zbikovska (2020). The dough sample (110 g) was flattened in the cylindrical vessel (A/DP) and penetrated for 20 mm at a speed of 3 mm/s to determine hardness (N) at room temperature ( $23 \pm 2$  °C) in triplicate. Texture of biscuits was determined with both the three-point bend test (TPB) and puncture test, according to San José et al. (2018). TPB was performed at room temperature ( $23 \pm 2$  °C) using a guillotine system with knife speed of 3 mm/s and 10 mm penetration depth (10 replicates). Parameters breaking strength (N) and fracturability index (mm) were obtained. Puncture test was performed (10 replicates) using a cylindrical probe of 2 mm in diameter at 0.5 mm/s test speed. Tests were performed at room temperature ( $23 \pm 2$  °C) with samples punctured in six different points (two in the inner area, four in central area, avoiding the external edges) obtaining breaking strength (N) and fracturability index (mm).

### 2.5 Statistical analysis

Results are reported as mean values of the replicates with their corresponding standard deviations. The influence of lipid fraction on characteristics of doughs and breads was evaluated through the one-way analysis of variance (ANOVA) followed by Tukey's post-hoc test for means discrimination, at p d 0.05 level, using statistical software SPSS®(version 21.0, SPSS Inc., Chicago, IL, USA).

## 3. Results and Discussion

### 3.1 Oleogel Characterization

Gels exhibited a pseudo plastic non-Newtonian behaviour in which viscosity decreases at higher shear rate typical of semi-solid product, Figure 1a. Shear thinning behaviour in wax oleogel was confirmed by Doan et al. (2015) using different wax sources (including RBW) in rice bran oil system. Also Onacik-Gur et al. (2017) and Ögütcü et al. (2015) found same results for SFO/RBW and olive oil/bees wax gels. Moreover, it is known that this rheological model is applicable for other structuring agents such as ethyl-cellulose (Ceballos, et al., 2014) and <sup>2</sup>-sitosterol (Wan et al., 2014). In general, increasing wax concentration determined an increase in consistency shifting up the viscosity curve. In SFO oleogels the hardening fibre effect was visible at 25°C and 4°C at both wax concentrations. Temperature did not seem to affect the flowing properties of gels.

At test conditions, values of hardness, gel strength and work of penetration were determined only for SFO gels at 5.5% RBW, Figure 1b. The magnitude of hardness was in agreement with Onacik-Gur & Zbikowska (2020). A possible explanation for failing test in other samples could be due to a weaker structure or possibly to a not appropriate storage time (24 h) before trials since, according to Doan et al. (2017), polymorphic transitions reinforcing the network could occur over storage time.

Macroscale textural properties better highlighted the strong dependency of network strength on cooling temperature. Lowering the cooling temperature had a significant detrimental effect on gel tri-dimensional structure that caused the reduction in texture attributes. For control reference without any fibre addition, sample structured at 25°C showed the greatest hardness, gel strength and work of penetration values. Decreasing the temperature, all parameters were reduced and samples cooled at 0°C could not be measured. However, when fibre is included a visible soaring in texture values at lower temperature is detected. Fibre added sample prepared at 4°C resembles similar texture parameters compared to reference at 25°C. Nevertheless, the hardening effect was clear in samples prepared at 0°C for which the texture parameters can be determined.

Thermal analysis revealed an endothermic peak for all gels with a maximum around 63-64°C, probably related to wax melting phenomena (Limpimwong et al., 2017). Moreover, samples prepared at 5.5% of RBW exhibited higher enthalpy values, confirming a greater network resistance. Lower cooling temperature showed a negative effect compensated by fibre addition. This latter result is in agreement with texture analysis showing similar values of enthalpy for SFO+RBW(5.5%)-25°C and SFO+RBW(5.5%)+BF40 - 4°C (data not shown).

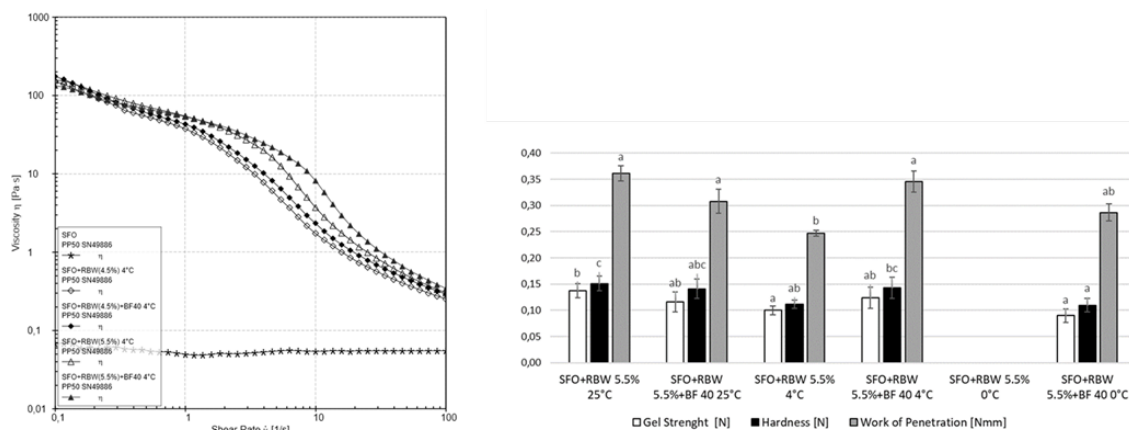


Figure 1: a. Viscosity (Pa\*s) in function of shear rate (s<sup>-1</sup>) of SFO at 4°C at 4.5% (squared) and 5.5% (triangle) RBW without (void) and with (solid) fiber addition. b. mean value (±DS) of gel strength (N), Hardness (N) and work of penetration (N\*mm) for SFO gels prepared at 5.5% RBW with and without bamboo fiber cooled at 25°C, 4°C and 0°C.

### 3.2 Dough and Biscuit Characterization

Saturated fats are crucial in the formation of the well-aerated structure of the cookies. The composition and properties of fat affect the leavening level and determine the dimensional changes and the rise of dough product during baking (Renzyaeva, 2013). Diameter of butter biscuits did not significantly change but a visible increment in height occurred with H values at least 40% higher than the raw biscuits with a reduction of the spreadability index (SI), Figure 2a. Furthermore, unlike butter, dough prepared with vegetable oils was not able to retain enough air resulting in higher SI. The use of gelled material improved the H rise and a certain decrease in spread ratio even if the biscuits were still not similar to butter ones (Onacik-Gur et al., 2017).

Another important parameter, that is strongly affected by incorrect aeration, is the texture of final product. Dough with an incorrect air bubble entrapment can be unable to keep the gas during baking resulting in structure collapse and harder texture (Onacik-Gur & Zbikovska, 2020). For those reasons according to Jacob & Leevalathi (2007) hardness of the dough could not necessarily control the texture of final biscuits. Dough and cookie texture parameters are then crucial properties and hardness and stickiness of dough and hardness and fracturability of biscuits are respectively reported in Figures 2b, 3a and 3b. A similar trend can be seen for both dough and biscuits. Oil samples were much harder than butter ones. According to Onacik-Gur et al. (2015) and Jacob and Leevalathi (2007) the strength required to break products with high amount of saturated fat is lower than with oil. Solid lipid fraction interacts in gluten-protein network formation as air bubble stabilizer and steric agent retarding gluten polymerization and shrinkage (Biguzzi et al., 2014). The less its content, the faster gluten can polymerize and pack resulting in low porosity and harder texture (Sciarini et al., 2013). SFO samples (before and after gelling process) possess greater hardness values than butter control. However, it is visible how the inclusion of fibre reduces dough firmness affecting positively texture parameters that better resemble the butter biscuits. Fibres may interact with other ingredients, especially water, during mixing resulting in a different morphology with volume increase and dough consistency reduction. Finally, higher discrepancies were detected in fracturability index parameter, Figure 3b. The fracturability index is defined as the distance necessary to the probe to break biscuits and it is inversely proportional to fracturability: higher travel distances are linked to higher force required to break the sample, indicating stiff and brittle materials. No differences among samples were detected by puncture test. It is reasonable that a penetration test over the entire volume of each biscuits is not able to detect exactly the material behaviour under breaking if sample heights are similar. TPB is instead a bending test in which the influence of fat type can be more visible. Butter dough presented the highest fracturability index compared to all other samples which showed similar results. Greater magnitude of fracturability index is related to ductile breaking mechanism. On the contrary, lower values encompass rigidity and brittleness: the material tends to deform poorly before fracture (Torres-Gonzales et al., 2018). The different fracture mechanism of butter cookies might have been induced by plastic fat characteristics that could not be reproduced with neither oil nor oleogels.

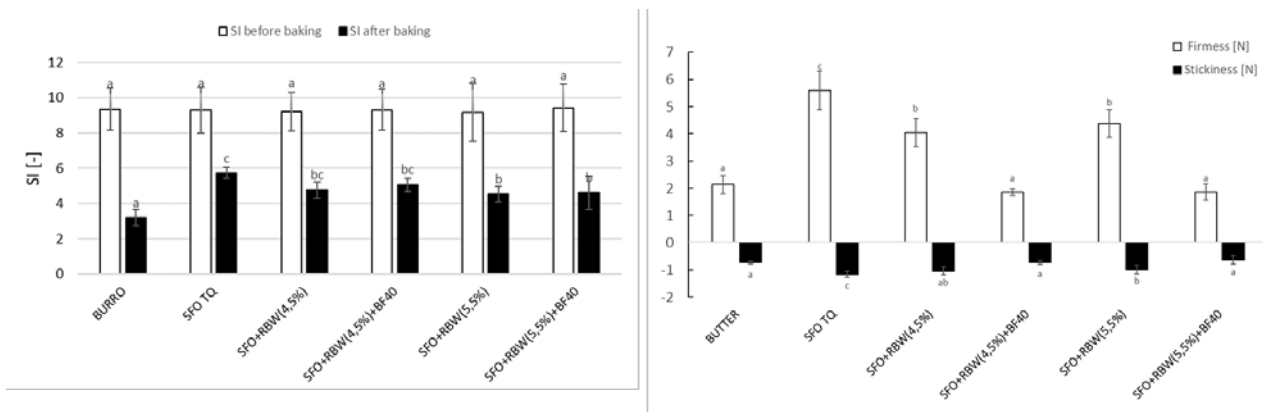


Figure 2: a. mean value ( $\pm$ DS) of spreadability index SI (-) of biscuits before and after baking. b. mean value ( $\pm$ DS) of firmness (N) and stickiness (N) and work of penetration (N\*mm) of dough prepared by SFO oils and gel and EVO oils and gels compared to butter cookies.

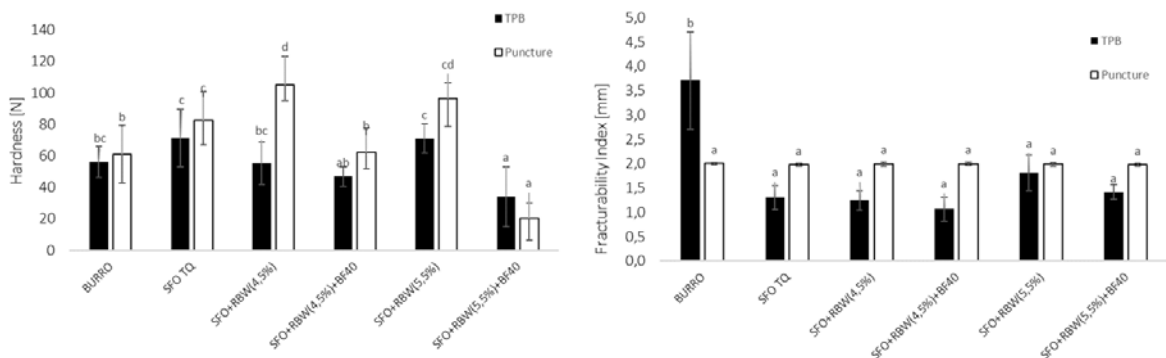


Figure 3: a. mean value ( $\pm$ DS) of hardness (N) and fracturability (mm) determined by TPB (solid bars) and Puncture (void bars) and stickiness (N) of biscuits prepared by SFO oils and gel and EVO oils and gels compared to butter cookies.

#### 4. Conclusions

The aim of this study was to investigate the effect of composition (use of rice bran wax at 4.5 and 5.5% wt/wt and bamboo fibre addition) and of cooling temperature (25°C, 4°C, 0°C) on the rheological behaviour of SFO oleogel systems. The second purpose was to investigate the use of the gels in biscuits preparation.

Oleogelatinization substantially modified the rheological behaviour of samples shifting from a typical Newtonian oil trend to a pseudo plastic non-Newtonian behaviour in gels. SFO gels were sensitive to cooling temperature with a structure weakening at lower temperature. Fibre addition showed a structuring action leading to an increased gel strength. This latter result was particularly evident on macroscopic scale by texture analysis since samples prepared at 25°C and without fibre addition showed the greatest hardness, gel strength and work of penetration values. Samples prepared at 0°C could not be measured with the exceptions of those with fibre inclusion showing comparable results for samples prepared at 4°C. The greatest properties of those samples were also confirmed by DSC analysis. SFO oleogels at both wax concentrations and with or without fibre addition, prepared at 4°C were tested as butter substitutes in biscuits. During mixing, dough with SFO got too stiff to be easily handled. On contrary, the use of fibre-added oleogel gave a softening effect of the structure possibly due to the interaction of the gel with other ingredients. Texture analysis confirmed the inclusion of fibre reduced dough stiffness with a behaviour similar to butter dough.

Butter biscuits showed a 40% increase in height, while biscuits prepared with SFO showed higher spread values, and the use of oleogel led to intermediate values. Two different methods (TPB and Puncture tests) were selected to assess the hardness and fracturability index of cookies, obtaining similar trends and order of magnitude. Samples with SFO and oleogels showed hardness values higher than butter control in agreement with

rheological and texture analysis of the doughs. The inclusion of fibre reduced biscuits stiffness making them more similar to butter ones. Higher discrepancies were detected in fracturability parameters, but, contrary to the TPB bending test, no differences among samples were detected with the puncture test.

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