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Influence of Preparation Methods on the Properties of Emulsified Films Based on Cassava Starch

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Abstract Composite films in which a lipid compound is incorporated are generally referred to as emulsified films. Since starch, mainly cassava starch, is abundantly available, this study compared two methods for producing emulsified films based on cassava starch, to see which one produces films with interesting barrier and mechanical properties. Both methods involved firstly mixing the base matrix with oil to create the emulsion (Met 2) and secondly separating the two phases by emulsifying the oil in water before mixing with the base matrix (Met 1). The results of this study showed a significant difference between the thickness, water content and solubility of the films obtained according to the preparation method. Thickness values were 81.09 µm and 94.68 µm for Met 1 and Met 2 respectively. For the water content of the films, the values were 18.71% and 24.53% for Met 1 and Met 2 respectively. For water solubility, the values were 14.32% and 53.24% for Met 1 and Met 2 respectively. Statistical analysis showed a significant difference between the water vapor permeability of the films obtained according to the preparation methods (Met 1 and Met 2). The values were $3.93.10^{-11}$ g. $Pa^{-1}s^{-1}m^{-1}$ and $5.44.10^{-11}$ g. $Pa^{-1}s^{-1}m^{-1}$ for Met 1 and Met 2 respectively. The analysis also showed a significant difference between the tensile stress at break and the elongation at break of the films obtained according to the preparation methods (Met 1 and Met 2). Tensile stress at break values were 6.68 MPa and 4.77 MPa respectively for Met 1 and Met 2. Elongation at break values were 65.21% and 56.12% for Met 1 and Met 2 respectively. All the results obtained in this study lead to the conclusion that the preparation method for starch-based emulsified films exerts a significant influence on the properties of the films obtained.

Keywords: *cassava starch, emulsified film, film-forming properties, homogenization techniques, edible films*

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1. Introduction

Edible films are thin layers of polymers that can be consumed [1]. They can be heterogeneous in nature, consisting of a mixture of polysaccharides, proteins and/or lipids. This approach makes it possible to use one of the distinct functional characteristics of each category of film-forming agent [2]. The combination of polymers to form films can be between proteins and carbohydrates, proteins and lipids, carbohydrates and lipids, or synthetic polymers and natural polymers. The main aim of composite film production is to improve permeability or mechanical properties, as required by a specific application. These heterogeneous films are applied either as an emulsion, suspension, or dispersion of immiscible constituents, or in successive coating layers (or multilayer films), or as a solution in a common solvent. As mentioned above, composite films contain both lipids and hydrocolloid components (proteins or polysaccharides) to exploit the advantages of both lipids and hydrocolloid components. When a barrier to water vapor is required, the lipid component fulfils this function, while the hydrocolloid component provides the necessary mechanical strength [3]. Several studies have been carried out into the possible improvement of the properties of films based on starch, which is a hydrocolloid, by incorporating lipid or protein compounds [4,5,6,7,8,9,10,11]. Composite films in which a lipid compound is incorporated are generally referred to as emulsified films. Emulsified films and coatings are in fact films in which lipids are dispersed within a macromolecular network that has a hydrophilic character [12,13]. Emulsified materials are obtained in a single film-forming casting and drying process. Their properties generally depend on preparation techniques, the type and quantity of components (hydrocolloids and lipids) and their compatibility, as well as microstructural heterogeneity [14]. Previous studies have shown that smaller particle sizes and more homogeneous distributions of lipid globules in emulsion-based films lead to lower water vapor permeability values [15,16,17,18,19]. Thus, the homogeneity and integrity of the oil-containing polysaccharide film depend closely on the miscibility of the hydrophobic molecules in the hydrophilic matrix, leading to a laminate structure or emulsified mixture [20]. The process of emulsifying the lipid phase in the aqueous phase is necessary before casting to form a film or apply a coating. Droplet size is a key factor in emulsion stability and affects viscosity and other important properties [13].

Various homogenization techniques are used to prepare film-forming emulsions. Rotor-stator homogenizers are often used and can achieve a particle size of 1 µm [21,22]. Particle size can be further reduced by applying high-pressure homogenizers to the system [23]. Microfluidization can provide emulsions with narrower particle size distributions due to the shear force of ultra-high-pressure homogenization and the severe constraints of head-on collision [24]. Microfluidization and sonication can produce nanoemulsions in the size range 150 to 700 nm. Intensifying the homogenization conditions of the film-forming emulsion affects the size of lipid structures in dried films, promoting mechanical strength, gloss, and film transparency [13]. These different homogenization techniques are essentially applied to improve the barrier and mechanical properties of emulsified films. The findings of the study by [25] on the influence of hydrocolloid nature on the structure and functional properties of emulsified edible films, indicated that the mechanical properties of emulsified films of cassava starch and a hydrogenated vegetable fat were relatively poor. In their methodology for preparing emulsified films of cassava starch and a hydrogenated vegetable fat, the mixture was pre-dispersed under magnetic stirring for 5 min at 600 rpm before being homogenized using an Ultra-Turrax T25 homogenizer for 2 min at 24,000 rpm. However, the characteristics of biopolymer-based films are highly dependent on their composition and preparation method [26]. Such a method applied to starch would undoubtedly lead to a reduction in viscosity and a loss of rigidity in the resulting film. Moreover, cassava starch films prepared by extrusion processes do not have good properties, due to the high shear rates applied [27]. Thus, the use of cassava starch in the production of emulsified films would be limited. Since starch, mainly cassava starch, is abundant and available, this study compares two methods for producing emulsified films from cassava starch and identifies the one that produces films with interesting barrier and mechanical properties.

2. Materials and Methods

2.1. Materials

Native cassava starch was cold extracted from the improved TMS cassava variety harvested at maturity after 12 months of planting in an experimental field at the National Center for Agronomic Research in Côte d'Ivoire (CNRA). After harvesting, the tubers were transported to the chemical laboratory of the Swiss Center for Scientific Research in Adioppodoumé (Yopougon, Abidjan). The following morning, using the method described in [28], the native starch was extracted. Bidistilled glycerol ($C_3H_8O_3$) with a purity of 99.5% was used as plasticizer and soy lecithin (($C_{40}H_{80}ON_8P$) n) was used as emulsifier. To determine the water vapor permeability of the films, anhydrous calcium chloride ($CaCl_2$) (6-14 mm) was used. VWR prolabo Chemicals (Leuven/Belgium) supplied all the chemicals used in this study. The peanut oil used in this study, CORA brand (Gembloux/Belgium), was purchased from a supermarket in Belgium.

2.2. Methods

2.2.1. Preparation of Emulsified Films Based on Improved Cassava Starch

Two methods of preparing emulsified films were used in this study. The first method (Met 1) was based on that described by [29,30]. The emulsified films were prepared in two stages. In the first step, 4 g (w/w, starch) of cassava starch were mixed with glycerol (1.2 g based on starch mass) and 2/3 distilled water in the final mixture. The mixture was heated for 20 minutes from 30 to 75°C with constant stirring at 750 rpm. Next, peanut oil (w/w, based on starch mass) and soy lecithin (w/w, based on the amount of oil added) with 1/3 distilled water were added to the total mixture, which was also heated for 20 minutes from 30 to 75°C with constant stirring at 750 rpm. The solution of peanut oil, soy lecithin and distilled water was homogenized at 24,000 rpm for 2 minutes using the Ultra Turrax T 25 (IKA-WERKE/ from Germany). In the second stage, the homogenized solution was mixed with starch and glycerol, then heated from 75 to 95°C for 25 minutes at 750 rpm. The second method (Met 2) of the study was based on that described by [25], considering pre-dispersion (600 rpm for 5 min) and homogenization (24,000 rpm for 2 min) with Ultra-Turrax. On the surface of a Petri dish, 20 g of the final solution obtained from both methods is poured and dried in a ventilated oven model Memmert UF-110 (from Germany) at 35°C for 24 hours. Oven-dried films are removed and stored at 62% humidity (ammonium nitrate, NH_4NO_3) for 48 hours in a desiccator at 25°C prior to testing.

2.2.2. Thickness of Emulsified Films

The thickness of all films was determined using an NSK (Japan) manual micrometer. Measurements were taken at 10 random points on the films, in triplicate for each film.

2.2.3. Moisture Content of Emulsified films

The water content of the films was determined gravimetrically by oven-drying at 105°C to a constant weight (dry sample weight) [31]. The results were expressed as a percentage of the initial film weight according to the following expression ((equation (1)):

$$\mathbf{H}(\%) = (\mathbf{P0} - \mathbf{Pf}) \times \mathbf{100} \qquad (1)$$

With H, humidity; P0, initial weight; Pf, final weight. At least triplicate analyses were performed per variety and formulation.

2.2.4. Water Solubility of Emulsified films

Determination of the water solubility of the films was carried out according to the method described by [32]. 2 x 3 cm pieces of each film were cut and stored for seven days in a desiccator containing silicate gel. After the seven days, samples were weighed (to the nearest 0.0001 g) g) (initial dry weight) using a precision balance (Mettler-Toledo AE-200, Precisa Instruments Ltd, Greifensee/Switzerland) and placed in beakers containing 80 mL deionized water. Samples were kept under constant stirring at 200 rpm in a stirring chamber (Heidolph promax 1020 mechanical stirrer, Schwabach/Germany) for one hour at laboratory temperature (~25°C). The film pieces were then collected by filtration and re-dried in an oven at 60°C to constant weight. The percentage of total solid matter (or percentage solubility) was calculated as follows ((Equation (2)):

$$\mathbf{S}(\%)=\left(\left(\mathbf{P0}-\mathbf{Pf}\right)/\mathbf{P0}\right)\times\mathbf{100} \tag{2}$$

With S, solubility; P0, initial dry weight; Pf, final dry weight. The measurements were made in triplicate for each film.

2.2.5. Water Vapor Permeability (WVP) of Emulsified films

The water vapour permeability of the films was carried out using the method [33] with a few modifications. Samples of 9 cm-diameter films were mounted and sealed on the opening of a cylindrical jar containing 50 g of anhydrous calcium chloride to maintain a relative humidity of 0%. The assembly was placed in a desiccator at 25°C containing a saturated solution of sodium chloride (75% RH). Once equilibrium conditions had been reached (2 h), eight weight measurements were taken over 8 h. Changes in cup weight were recorded to the nearest 0.0001 g and plotted against time. The slope of each line was calculated using linear regression ($r^2 > 0.99$) and water vapour transmission (WVTR) was calculated as the ratio of the slope of the straight line (g/s) to the transfer surface (m^2). The exposed surface (the cylindrical pot surface of 5.6 cm in diameter) was 0.00246 m^2 and WVP ($g.\ Pa^{-1}\ s^{-1}\ m^{-1}$) calculated according to the following expression (Equation (3)):

$$WVP=\left[WVTR/S\ \left(R_1-R_2\right)d\right] \tag{3}$$

With S, vapor pressure at saturation of the water (Pa) at the test temperature (25°C) = 3170 Pa; R_1, relative humidity in the desiccator; R_2, relative humidity in the pot; d, thickness of the film (m). All tests were conducted in triplicate.

2.2.6. Mechanical Properties of Emulsified films

Tensile tests were performed using a TA.XT2 texture analysis (Stable Micro Systems, Godalming/England) according to [34] using an A/TG traction handle system. The parameters determined were tensile strength and deformation at break (elongation at break) for emulsified films. The tested films trips (8 x 2.5 cm) were cut with a scalpel for each preconditioned sample (62 % relative humidity, 25°C) and placed between the machine jaws. The thickness of each sample was measured at four points along its length with a Mitutoyo micrometer (NSK/Tokyo, Japan Micrometer). The initial separation between the handles was set at 50 mm. The upper part of the probe gradually rises by stretching the film at a constant speed of 10 mm/min until it ruptures. The force-elongation curves were recorded. Force and elongation were measured at the point of failure. For each formulation, eight samples were tested. The measurements were made in triplicate for each film.

2.2.7. Color of Emulsified films

The colour of the films was determined by the method described by [35] and by [36]. This determination was made using an international Datacolor spectrophotometer D65/10 (Miniscan XE Virginia, USA). L* represents the luminance (from white to black), (a*) the chromatic index from green to red and (b*) that from yellow to blue. From this data, equation (4) was used to calculate the colour difference (ΔE* ab) of emulsified starch-based films compared to the colour of a white reference surface:

$$\Delta E=\sqrt{\left(L^*-L_0^*\right)^2+\left(a^*-a_0^*\right)^2+\left(b^*-b_0^*\right)^2} \tag{4}$$

2.2.8. Statistical Analysis

Single-factor ANOVA variance analysis was performed on the mechanical, optical and barrier properties of starch-based emulsified films, prepared by two different methods, using STATISTICA 7.1 software. The DUNCAN test at a 5 % threshold was applied to detect differences between sample sets.

3. Results

3.1. Film Thickness, Water Content and Water Solubility

Table 1 shows the values for film thickness, water content and water solubility, depending on the method used to prepare the emulsified films. Thickness values were 81.09 µm and 94.68 µm respectively for the different methods of preparing Met 1 and Met 2 emulsified films. Film water content values were 18.71% and 24.53% for Met 1 and Met 2 respectively. For water solubility, the values were 14.32% and 53.24% for Met 1 and Met 2 respectively. Statistical analysis of variance ANOVA showed a significant difference (p< 0.05) between the thickness, water content and solubility of films obtained according to the preparation method (Table 1).

Table 1. Film thickness, water content, solubility and water vapor permeability by preparation method

Methods	Thickness (µm)	Water content (%)	Solubility (%)	WVP ($g.\ Pa^{-1}s^{-1}m^{-1}$)
Met 1	81,09 ± $3{,}45^a$	18,71 ± $0{,}71^a$	14,32 ± $2{,}47^a$	$3{,}93.10^{-11}$ ± $1{,}09^a$
Met 2	94,68 ± $4{,}88^b$	24,53 ± $0{,}92^b$	53,24 ± $1{,}11^b$	$5{,}44.10^{-11}$ ± $1{,}21^b$

Values assigned different lowercase letters in the same column are significantly different at $p < 0.05$. **Met 1:** first method for preparing emulsified films; **Met 2:** second method for preparing emulsified films.

3.2. Water Vapor Permeability (WVP)

Water vapor permeability values are shown in Table 1.

Statistical analysis of variance ANOVA showed a significant difference (p< 0.05) between the water vapor permeability of the films obtained according to the preparation methods (Met 1 and Met 2). The values were 3.93.10-11 g. Pa-1s-1m-1 and 5.44.10-11 g. Pa-1s-1m-1 for Met 1 and Met 2 respectively.

3.3. Film Mechanical Properties

The values for stress at break (TS) and elongation at break (EB) are shown in Table 2. Statistical analysis of variance (ANOVA) showed a significant difference ($p < 0.05$) between the tensile stress at break and the elongation at break of films obtained according to the preparation methods (Met 1 and Met 2). The values for tensile strength at break were 6.68 MPa and 4.77 MPa for Met 1 and Met 2 respectively. Elongation at break values were 65.21% and 56.12% for Met 1 and Met 2 respectively (Table 2).

Table 2. Mechanical properties of films by preparation method

Méthodes	TS (MPa)	EB (%)
Met 1	6,68 ± 0,28^a	65,21 ± 14,86^a
Met 2	4,77 ± 0,55^b	56,12 ± 9,56^b

Values assigned different lowercase letters in the same column are significantly different at $p < 0.05$. **Met 1:** first method for preparing emulsified films; **Met 2:** second method for preparing emulsified films.

3.4. Film Color Parameters

The values of the colour parameters, except for a* (Table 3), varied significantly (p< 0.05) according to the statistical analysis of variance ANOVA. L* parameter values were 91.17 and 91.42 for Met 1 and Met 2 respectively. Values for a* were -0.38 and -0.43 for Met 1 and Met 2 respectively. For b*, the values were 2.02 and 1.93 respectively for Met 1 and Met 2. For ΔE*, the values were 3.01 ± 0.001 and 2.76 ± 0.014 for Met 1 and Met 2 respectively (Table 3).

Table 3. Film color parameter values by preparation method

Méthodes	L^*	a^*	b^*	ΔE^*
Met 1	91,17 ± 0,06a	-0,38 ± 0,01a	2,02 ± 0,08a	3,01 ± 0,001a
Met 2	91,42 ± 0,01b	-0,43 ± 0,01b	1,93 ± 0,03a	2,76 ± 0,014b

Values assigned different lowercase letters in the same column are significantly different at $p < 0.05$. **Met 1:** first method for preparing emulsified films; **Met 2:** second method for preparing emulsified films.

4. Discussion

Statistical analysis of variance ANOVA showed a significant difference ($p < 0.05$) between the thickness, water content and solubility of films obtained according to the preparation method. Film thickness is an important characteristic that needs to be evaluated. It can affect the film's mechanical, optical and water vapor barrier properties [37,38,39]. The difference observed between film thicknesses depending on the preparation method could be due to the total solids content of the films. Indeed, according to [40], film thickness is influenced by the solids content of the film-forming solution and the processing parameter. In addition, [41] reported that films with essentially the same total solids content did not differ significantly in thickness, and that increasing total solids content increased film thickness. Also, film thickness depends on the reorganization of molecular chains to form a more compact matrix in composite starch films [11]. In this study, the films obtained with the Met 1 preparation method were less thick than those obtained with the Met 2 preparation method, despite the same film composition (starch, glycerol, oil, lecithin in the same proportions). The technique employed in Methodology 1 (Met 1) certainly enabled a reduction in the size of oil droplets in the starch matrix [42,43,44].

About the water content of the films, a significant difference was observed between those obtained using the Met 1 preparation method and those obtained using the Met 2 method. This difference could be linked to the difference in film preparation technique.

Water solubility is an important property for food packaging materials and water-insoluble or water-resistant packaging materials for food are generally desired, and films with high water solubility may also be desired for encapsulation of drugs, foods or additives [44]. The water solubility of the emulsified films prepared in this study was significantly different. This difference could be explained by the fact that high-pressure treatment in Met 2 would have degraded the intermolecular bonds of the starch molecules and formed new inter- and intramolecular bonds with polar glycerol groups. On the other hand, soluble starch increased after high-pressure treatment, thus increasing the likelihood of amylose leaching from starch granules, contributing to the high solubility observed [45]. Also, the high-pressure process could convert the starch's crystalline regions into semi-crystalline ones.

In this situation, the number of remaining (non-crosslinked) starch functional groups would have increased [45], which would certainly have facilitated the solubility of the films obtained with Met 2.

The Met 1 emulsified film preparation method produced films with significantly lower water vapor permeability values than the Met 2 method. This observed difference is linked to the good distribution and reduction of lipid particle sizes due to the homogenization method and conditions adopted in this study. It has been shown that the water barrier properties of emulsion-based films are closely related to the type, concentration, and size of the lipid particles and the emulsifying agent [46]. The particle size distribution of film-forming emulsions, which is directly related to the homogenization method and conditions, has a strong effect on water vapor transfer through the films [44]. The reduction and homogeneous size distribution of lipid particles or globules significantly reduce the water vapor permeability of edible emulsion-based films [15,16,17,18,19] [47].

Also, several studies have shown that there is a negative correlation between the droplet size of emulsions and the water barrier characteristics of emulsified biopolymers [48]. On the other hand, the formation of an interconnected lipid network in the film matrix creates a tortuous path through the film for water molecules. The Met 1 method enabled good dispersion of oil droplet sizes. It has been shown that the tortuosity parameter is higher as the dispersed phase ratio increases or as the dispersed droplet size decreases [49].

The tensile stress at break of films prepared using the Met 1 method was higher than that of films prepared using the Met 2 method. Similarly, the elongation at break of Met 1 films was higher than that of Met 2 films. This difference in tensile strength at break could be linked to the procedure adopted to prepare the emulsified films. In the Met 2 preparation method, cassava starch, peanut oil, soy lecithin and glycerol were homogenized at 24,000 rpm for 2 minutes. The high shear rate in the starch matrix leads to a decrease in viscosity and a loss of rigidity in the resulting film. [27] have shown that cassava cassava starch films prepared by extrusion processes do not have good properties, due to the high shear rates applied.

Our observations are in line with the findings of the study by [25] on the influence of hydrocolloid nature on the structure and functional properties of emulsified edible films. In this study, the authors reported that the mechanical properties of emulsified films of cassava starch and a hydrogenated vegetable fat were relatively poor. Also, the characteristics of biopolymer-based films are highly dependent on their composition and method of preparation [26]. Furthermore, droplet size is a determining factor for emulsion stability and affects viscosity and other important properties [13]. Certainly, the Met 1 preparation methodology had a greater effect on the size of the lipid structures, thus promoting good mechanical strength. In fact, intensifying the homogenization conditions of the film-forming emulsion affects the size of the lipid structures in the dried films, favoring mechanical strength, gloss, and transparency of the films [13]. These various homogenization techniques are mainly applied to improve the barrier and mechanical properties of emulsified films [25]. Other studies have shown that the emulsification conditions of emulsified films significantly influence the final microstructure of the biopolymer and its mechanical behavior [43,49,50]. The difference observed is also due to the high solid lipid content in films obtained using the Met 2 preparation method. Indeed, the higher the solid lipid content, the poorer the mechanical properties of emulsified films [25].

The L* parameter value of the films obtained with the Met 2 method was significantly higher than the L* value of the films obtained with the Met 1 method. The a* parameters of the films were negative. Color parameters are the most important factors affecting consumer acceptance of a food product. The films in this study were made from the same materials in the same proportions (starch, peanut oil, soy lecithin and glycerol). The observed difference in parameters would be linked to changes in the average size of the oil droplets due to the different homogenization methods. [44] made similar observations in their study of the effect of ultrasonic treatment on the properties of nano-emulsion films obtained from hazelnut flour proteins and clove essential oil.

5. Conclusion

Composite films in which a lipid compound is incorporated are generally referred to as emulsified films. In this study, two methods of producing emulsified films based on cassava starch were investigated. The results showed a difference between the thickness, water content and solubility, water vapor permeability and mechanical properties of the films obtained according to the preparation method. All the results obtained in this study lead to the conclusion that the preparation method for starch-based emulsified films exerts a significant influence on the properties of the films obtained. The high shear rate in the starch matrix leads to a reduction in viscosity, a loss of stiffness and thus poor film properties. Emulsified starch-based films with interesting film-forming properties can only be obtained if less dense homogenization techniques are employed.

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Author Contribution Statement

Adjouman Yao Désiré: conceived and designed the experiments; performed the experiments; analyzed and interpreted the data; analyzed and interpreted the data; wrote the paper. Gnagne Eliane Hadiowe: contributed reagents, materials, analysis tools or data; wrote the paper. Benal Kouassi Kouadio: conceived and designed the experiments; analyzed and interpreted the data; wrote the paper. Nindjin Charlemagne: contributed reagents, materials, analysis tools or data. Tetchi Fabrice Achille: analyzed and interpreted the data; wrote the paper. Amani N'Guessan Georges: contributed reagents, materials, analysis tools or data.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

List of Abbreviations and Symbols

Met 1: Method 1
Met 2: Method 2
µm: micrometer
g : gram
Pa: pascal
s: second
m: meter
MPa : megapascal
mm : millimeter
rpm : turns per minute
min : minute
mL : milliliter
m^2 : square metre
cm : centimeter

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