UNIVERSIDADE ESTADUAL DE MARINGÁ
CENTRO DE CIÊNCIAS AGRÁRIAS
Programa de Pós-Graduação em Ciência de Alimentos

Polpa lavada do suco de laranja como emulsificante em gelado comestível

HELOISA DIAS BARBOSA

Maringá
2021

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Dissertação apresentada ao programa de Pós Graduação em Ciência de Alimentos da Universidade Estadual de Maringá, como parte dos requisitos para obtenção do título de mestre em Ciência de Alimentos.

Maringá

2021

Dados Internacionais de Catalogação-na-Publicação (CIP)
(Biblioteca Central - UEM, Maringá - PR, Brasil)

Barbosa, Heloisa Dias

B238p Polpa lavada do suco de laranja como emulsificante em gelado comestível / Heloisa Dias Barbosa. -- Maringá, PR, 2021.
45 f.: il. color., figs., tabs.

Orientadora: Profa. Dra. Paula Toshimi Matumoto-Pintro.
Dissertação (Mestrado) - Universidade Estadual de Maringá, Centro de Ciências Agrárias, Programa de Pós-Graduação em Ciência de Alimentos, 2021.

1. Fibra alimentar. 2. Emulsificante. 3. Propriedades de derretimento. 4. Reologia. I. Matumoto-Pintro, Paula Toshimi, orient. II. Universidade Estadual de Maringá. Centro de Ciências Agrárias. Programa de Pós-Graduação em Ciência de Alimentos. III. Título.

CDD 23.ed. 664

Marinalva Aparecida Spolon Almeida - 9/1094

HELOISA DIAS BARBOSA

“POLPA LAVADA DO SUCO DE LARANJA COMO EMULSIFICANTE EM GELADO COMESTÍVEL”.

Dissertação apresentada à Universidade Estadual de Maringá, como parte das exigências do Programa de Pós-graduação em Ciência de Alimentos, para obtenção do grau de Mestre em Ciência de Alimentos.

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Maringá – 2021

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BIOGRAFIA

Heloisa Dias Barbosa, nascida em 20 de Setembro de 1993 na cidade de Goioerê, Paraná, Brasil. Filha de Aparecido Donizete Barbosa e Margarete Dias Barbosa. Formada em Engenharia de Alimentos pela Universidade Estadual de Maringá em 2017. Trabalhou por dois anos em um abatedouro de aves, atuando como analista de qualidade. Ingressou no Programa de Pós-graduação em Ciência de Alimentos em Abril de 2019, com defesa da dissertação em Março de 2021. Tem experiência nas áreas de controle de qualidade, ciência e tecnologia de alimentos, atuando na área de ciência e tecnologia de produtos agropecuários.

Dedico

Aos meus pais, meu porto seguro que sempre estarão ao meu lado.

AGRADECIMENTOS

Primeiramente a Deus, pela minha saúde e sabedoria, a Quem eu confio e entrego todos os passos da minha vida.

Aos meus pais, Aparecido Donizete Barbosa e Margarete Dias Barbosa, por todo amor, por sempre me indicarem qual a melhor direção a seguir, sempre priorizando meus estudos e dando todo o suporte em minhas escolhas.

Ao meu irmão Douglas Dias Barbosa e esposa Barbara Almeida Granado Barbosa, que sempre me apoiaram e incentivaram para a realização desta etapa da minha vida.

Ao meu noivo Jean A. Pegorel Gonçalves Dias, por ter me incentivado desde o início e mesmo com a distância, sempre conseguir me tranquilizar, me ouvir, ajudar e apoiar em todos os momentos.

À minha orientadora, Profa. Dra. Paula Toshimi Matumoto Pintro, por todas as conversas que mesmo em meio à nova forma de nos adaptarmos com reuniões online, conseguiu estar presente, dando suporte, compartilhando conhecimento, orientações e conselhos.

Aos professores da Pós-Graduação em Ciência de Alimentos por todo conhecimento repassado durante as disciplinas realizadas.

Aos meus professores da Engenharia de Alimentos, sem os quais não teria chego até aqui.

À Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (Capes), pela concessão da bolsa de estudos.

Aos envolvidos na contribuição de análises para realização deste trabalho, Prof. Dr. Marcos Luciano Bruschi, Msc. Jéssica Bassi Da Silva, Profa. Dra. Francielle Sato.

A todos do grupo de pesquisa em alimentos funcionais da Universidade Estadual de Maringá. Em especial ao Anderson Lazzari, Bianka Rocha Saraiva, Fernando Antônio Anjo, Ana Carolina Viscardi Plefh, Lucas Maldonado da Silva e Elisângela de Césaro por toda a contribuição e companheirismo durante esse período.

A todos meus familiares e amigos que sempre torceram e comemoraram cada conquista minha.

APRESENTAÇÃO

Esta dissertação de mestrado está apresentada na forma de um artigo científico.

1 Heloisa Dias Barbosa; Anderson Lazzari, Fernando Antônio Anjo, Bianka Rocha Saraiva, Jessica Bassi da Silva, Marcos Luciano Bruschi, Francielle Sato, Paula Toshimi Matumoto Pintro. Orange juice pulp wash as emulsifier in ice cream. Food Hydrocolloids.

GENERAL ABSTRACT

INTRODUCTION. Orange juice process generates oil emulsion, orange bagasse and pulp wash as co-products. Bagasse and pulp wash represents around 50% of the production and is usually sent to feed mill. Formed by juice cell vesicles, pulp wash contain about 70% of dietary fiber. Application of dietary fibers in food products brings improvement in texture, increase viscosity, gel-forming ability and reduces moisture of products. Ice cream is an aerated emulsion (oil in water) consisted of air cells, ice crystals and fat globules and is thermodynamically unstable. Emulsifiers are added in order to stabilize the emulsion, providing smooth texture and improving melting properties. New approaches to use food ingredients with a sustainable chain and new colloidal particles with partial ability to be wetted by oil and water, co-products like dietary fiber can have capability to act as an emulsifier in food emulsion systems.

AIMS. The objective of this study was characterize physicochemical and technological properties of orange juice pulp wash (PW) and produce ice cream with PW as an emulsifier, verifying the technological and rheological characteristics of the final product.

MATERIAL AND METHODS. Orange juice pulp wash was donated by Citri Agroindustrial S/A (Paranavaí, Paraná, Brazil), dehydrated in a freeze drier, ground and sieved to 60 mesh. PW was submitted to analysis of chemical composition, pH, color evaluation, technological properties (water retention, oil absorption and emulsion activity capacity) and FTIR (Fourier Transform Infrared). Previous experiments were made to select which concentration of PW was necessary to obtain similar behavior to an ice cream made with 1% of commercial emulsifier (CE) through texture profile analysis (hardness and adhesiveness) and overrun measurements. With 0.5% of PW selected, the ice cream control formulation (C) was made and formulations (E0.5, E0.25 and E0) were made with 0.5% of PW and CE reduction. Treatments were analyzed by overrun measurements, melting and rheological properties.

RESULTS AND DISCUSSION. PW is mainly composed by dietary fiber, with 45.91% insoluble fiber and 12.78% soluble fiber. Water retention and oil absorption capacities presented values that can be applied in reduce moisture of products, modify viscosity and texture and also improve stabilization of foods and emulsion. Emulsifying activity of PW can be explained as a function of its water retention capacity, swelling capacity of fibres and combination of Pickering mechanism, allowing to bound oil droplet and get water absorbed. FTIR analysis showed a common structure of fibers, mainly cellulose and lignin. Presence of PW in formulations had more resistance to start melting than control (C), due to water retention effect. All formulations presented pseudoplastic behavior, a good characteristic for ice cream production, with a lower flow viscosity, enabling the mix during production. Higher values for yield stress was displayed for E0.5, E0.25 and E0, than formulation C, explained due the presence of PW, forming greater network structure by swelling of orange fiber. Oscillatory rheology indicates a viscoelastic system for all formulations, meaning elastic modulus (G’) was greater than viscous modulus (G”) and as loss tangent was less than one, formulations represent predominant elastic behavior. Thermo-oscillatory rheology showed steep curves for formulation C and E0, demonstrated by a quicker melting rate analysis.

CONCLUSIONS. Orange juice pulp wash is a source of dietary fiber and had values for water retention, oil absorption and emulsion capacity that allows its application as technological enhancement in food. Replacement of commercial emulsifier by PW improved parameters for ice cream production, being a promising ingredient for food industry.

Key words: dietary fibre; thermo-oscillatory; rheology; melting properties

RESUMO GERAL

INTRODUÇÃO. A produção de suco de laranja gera emulsão de óleo, bagaço de laranja e polpa lavada como coprodutos. O bagaço e a polpa lavada representam 50% da produção e normalmente são enviados para a fábrica de rações. Formado por vesículas de células de suco, a polpa lavada contém cerca de 70% de fibra dietética. A aplicação de fibra dietética em produtos alimentícios traz melhora na textura, aumenta a viscosidade, capacidade de formação de gel e reduz a umidade dos produtos. O sorvete é uma emulsão aerada (óleo em água) composta por células de ar, cristais de gelo e glóbulos de gordura e é termodinamicamente instável. Emulsificantes são adicionados com o objetivo de estabilizar a emulsão, proporcionando textura macia, melhorando as propriedades de fusão. Novas abordagens para utilizar ingredientes com uma cadeia sustentável e novas partículas coloidais com capacidade parcial de serem umedecidos por óleo e água, coprodutos como as fibras dietéticas aparecem como opção por terem a capacidade de agir como um emulsificante em sistemas de emulsão de alimentos.

OBJETIVO. O objetivo deste estudo foi caracterizar as propriedades físico-químicas e tecnológicas da polpa lavada de suco de laranja (PW) e produzir sorvete com PW como emulsificante, avaliando as características tecnológicas e reológicas do produto final.

MATERIAL E MÉTODOS. Polpa lavada do suco de laranja (PW) foi doada pela Citri Agroindustrial S/A (Paranavaí, Paraná, Brasil), desidratada por liofilização, moída e peneirada a 60 mesh. PW foi submetida a análises de composição química, pH, avaliação de cor, propriedades tecnológicas (capacidade de retenção de água, capacidade de absorção de óleo e atividade emulsificante) e FTIR (Infravermelho com Transformada de Fourier). Experimentos prévios foram feitos para selecionar qual a concentração de PW era necessária para obter comportamento similar à um sorvete feito com 1% de emulsificante comercial (CE) através de análises de perfil de textura (dureza e adesividade) e medidas de *overrun*. Com a concentração de 0.5% de PW escolhida, foi feita a formulação controle (C) de sorvete e as formulações E0.5, E0.25 e E0 foram feitas com 0.5% de PW e redução de CE. Os tratamentos foram analisados através do *overrun*, propriedades de fusão e propriedades reológicas.

RESULTADOS E DISCUSSÃO. PW é composta principalmente por fibra dietética, com 45.91% de fibra insolúvel e 12.78% de fibra solúvel. Capacidade de retenção de água e absorção de óleo apresentaram resultados que permitem sua aplicação para reduzir umidade de produtos, modificar viscosidade, textura assim como melhorar a estabilização de alimentos e emulsões. A atividade emulsificante de PW pode ser explicada em função de sua capacidade de retenção de água, capacidade de inchamento de fibras e uma combinação de mecanismo *Pickering*, permitindo a ligação com a gota de óleo e absorção de água. A análise FTIR mostrou uma estrutura comum de fibras, principalmente celulose e lignina. A presença de PW nas formulações fez com que houvesse maior resistência para iniciar o derretimento do que a formulação controle (C), devido ao efeito de retenção de água. Todas as formulações apresentaram comportamento pseudoplástico, que é uma caraterística interessante para a produção de sorvete, com uma viscosidade de fluxo menor, facilitando a mistura durante a produção. Altos valores para a tensão de cedência foi mostrada pelas formulações E0.5, E.25 e E0 devido a presença de PW, formando uma melhor estrutura de ligação através do inchamento da fibra da laranja. Reologia oscilatória indicou um sistema visco elástico para todas as formulações, uma vez que o módulo elástico (G’) foi maior do que o módulo viscoso (G”) e como a tangente de perda foi menor do que um, as formulações representam predominantemente um comportamento elástico. A reologia termo-oscilatória apresentaram curvas mais íngremes para a formulação C e E0, que foi demonstrada pelos valores mais altos na análise de taxa de derretimento.

CONCLUSÃO. Polpa lavada de suco de laranja é fonte de fibra alimentar e obteve valores de retenção de água, absorção de óleo e capacidade emulsificante que permitem sua aplicação para aprimoramento de propriedades tecnológicas em alimentos. A substituição do emulsificante comercial por polpa lavada de suco de laranja melhorou os parâmetros de produção de sorvetes, sendo um ingrediente promissor para a indústria alimentícia.

Palavras chaves: fibra alimentar, termo-oscilatório, reologia, propriedades de derretimento.

ARTICLE

Orange juice pulp wash as emulsifier in ice cream

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Abstract

Orange juice pulp wash (PW) was characterized by physicochemical and technological analysis and incorporated into ice cream to assess its technological behaviour as a replacement of commercial emulsifier (CE). PW is one of the co-products originated from orange juice industries, is a great source of dietary fiber and is usually sent to animal feed. In order to evaluate this material to be used in food production, chemical composition and technological properties like water retention capacity, oil absorption capacity and emulsion activity were analysed. Ice cream was produced with 0.5% of PW and reduction of CE was studied to show the emulsification capacity of the PW in ice cream properties. Physicochemical, melting behaviour and rheological properties were conducted for the ice creams. All formulations had shear-thinning behavior. Formulation E0 (without CE and with 0.5% PW) had the highest yield value, due to linear chain of polysaccharides and its ability to form gel and through swollen of fiber, obtaining a stronger network structure. Formulations that contained PW had higher consistency (k) and longer time to start the melting than control (C) formulation. Control formulation (without PW) and formulation with total replacement of emulsifier (E0) obtained similar overrun ($p > 0.05$). Replacement of commercial emulsifier for orange juice pulp wash improved ice cream rheological parameters.

Keywords: dietary fiber; thermo-oscillatory; rheology; melting properties

1. Introduction

Orange juice process generates large quantity of co-products: oil emulsion, obtained by peel oil recovery, orange bagasse, formed by peel, orange tissues and seed which are usually sent to feed mill and pulp wash. Orange juice pulp wash is formed by vesicles or juice cell sacs, and undergoes a washing process to recover soluble sugars, which can increase the juice yield of soluble solids by 4 to 7%. Most of this orange juice pulp wash is sent with the bagasse directly for drying in pellets for animal feed, and a small portion can be inserted again in the juice itself or sold frozen for juice reconstituted. As it can contain about 70% of dietary fiber, an opportunity to value this co-product is its use as an ingredient in food production (Hui, 2004; Passarelli, Matthiesen, Vanhemelrijck, Gusek & Reeder, 2009; Yamanaka, 2005).

Dietary fibers are obtained through the cell walls of fruits, vegetables and other foods. They consist mainly of cellulose, hemicellulose, pectin, β-glucans, gums and lignin not hydrolyzed by human digestive enzymes (Figuerola, Hurtado, Estévez, Chiffelle & Asenjo, 2005; Raghavendra et al.., 2006). Incorporation of dietary fiber in food product brings textural improvement, due to high water holding capacity, gel-forming ability, increase of viscosity; being this behavior correlated with the ratio of insoluble to soluble fiber and their interaction in food system (Campidelli et al., 2020; Soukolis, Lebesi & Tzia, 2009). Growing interest of food products named as clean label has demanded searching for new ingredients as replacements for food formulations with a sustainable chain (Kurt & Atalar, 2018).

Ice cream is a frozen dairy dessert, mainly composed by milk, fat, nondairy fats, water, sweeteners, stabilizers and emulsifiers (Goff & Hartel, 2013). It is a complex colloidal frozen system, characterized as an aerated emulsion (oil in water) consisted of air cells, ice crystals and fat globules dispersed in a continuous phase of unfrozen solution (serum) (Muse & Hartel, 2004; Soukolis et al., 2009; Akbari, Eskandari & Davoudi., 2019; Loffredi, Moriano, Masseroni & Alamprese, 2021). Each ingredient of the mix composition influence technological, physical and chemical structure and sensory characteristics of ice cream (Campidelli et al., 2020).

Ice cream is a food emulsion system thermodynamically unstable, formed by oil droplets dispersed into a continuous aqueous phase. Due to immiscible properties, phase separation can occur in this product (Dickinson, 1992; Berton-Carabin & Schroën, 2015; Gould, Vieira & Wolf, 2013). Emulsifiers are added in order to stabilize the emulsion and make an interfacial layer between immiscible fluids, providing smooth texture, improving melting properties, reducing ice crystal growth and promoting fat destabilization (Baer, Wolkow & Kasperson, 1997; Goff & Hartel, 2013). Surfactants are the most used emulsifier in food industry (Zafeiri & Wolf, 2019). There are new approaches about use of colloidal particles that have as a characteristic their partial ability to be wetted by oil and water and act as emulsifiers (Berton-Carabin & Schroën, 2015). Regarding these particles, co-products can have valuable sources of nutrients, and substitutes of emulsifier can be obtained through these materials, aggregating sustainable chain of the food process (Loffredi et al., 2021). Dietary fiber like cyclodextrins has capability to be an emulsifier in Pickering emulsion due the three-dimensional structure and its hydrophobic and hydrophilic characteristics (Loffredi et al., 2021).

The aim of this work was characterize physicochemical and technological properties of orange juice pulp wash and analyze their inclusion as an emulsifier in ice cream.

2. Materials and methods

2.1 Materials

Orange juice pulp wash was kindly donated by Citri Agroindustrial S/A (Paranavaí, Paraná, Brazil). The ingredients used in the ice cream manufacture, pasteurized whole milk, whole milk powder, pasteurized milk cream 37% (w/w), sugar, glucose syrup, invert sugar, emulsifier (Duas Rodas, Jaragua do Sul, Brazil) were purchased in local market and carrageen gum was acquired from IBRAC (Rio Claro, Brazil). Enzymatic assay kit was from Megazyme (Megazyme International Ireland Ltd., Bray, Ireland).

2.2 Orange Juice Pulp Wash

2.2.1 Raw material preparation

Orange juice pulp wash (PW) was dehydrated in a freeze drier (Christ Alpha 1-4 LD plus, Marin Christ, Germany), ground, standardized at 60 mesh and stored at room temperature protected from light.

2.2.2 Physicochemical properties

Chemical composition was analyzed according to AOAC (2005) by moisture, ash content, crude protein by Kjeldahl method (nitrogen-to-protein conversion factor of 6.38) and total fat (Bligh & Dyer, 1959). Total dietary fiber (TDF), soluble (SDF) and insoluble dietary fiber (IDF) were determined by following the enzymatic and gravimetric method according to AOAC (1995). Carbohydrate was calculated by difference of other components from total. The results were expressed on a dry basis. The pH was determined using a pHmeter (Tecnopon, mPA-210). Color parameters L* (luminosity), coordinate a* (−a green /+a red) and coordinate b* (−b blue /+b yellow) of PW was determined using a CR-400 digital colorimeter (Konica Minolta, light source D65) using the CIElab system.

2.2.3 Water retention capacity

Water retention capacity (WRC) was determined according to Du, Zhu & Xu (2014). Dried PW (1.0 g) was placed into a graduated centrifugal tube with 30 mL of distilled water and stirred for 18 h. Following centrifugation at 3000 g for 20 min, the supernatant was removed and the hydrate weight recorded, and the sample was dried at 105 °C for 2 h to obtain the dry weight. Water retention capacity was expressed according to Equation 1:

$$WRC\ (g/g) = \frac{hydrated\ weight - dry\ weight}{dry\ weight} \quad (1)$$

2.2.4 Oil absorption capacity

Oil absorption capacity (OAC) was determined according to Kaur & Singh (2005) with modifications. Dried PW (0.5 g) and soybean (3 mL) were stirred for 1 min and allowed to stand at ambient temperature for 30 min. Following centrifugation at 1200 g for 30 min, the supernatant was removed and the residual weight recorded. Oil absorption capacity was expressed as gramme of oil bound per gramme of the sample on a dry basis.

2.2.5 Emulsion activity

Emulsion activity was determined according to Yasumatsu et al. (1972) with modifications. 2 g of PW, 20 mL distilled water, 20 mL soybean oil were mixed for 5 min (3600 rpm). The emulsion was centrifuged at 2000 g for 5 min. The ratio of the height of the emulsion layer to the height of the liquid layer was calculated as the emulsion activity expressed in percentage.

2.2.6 Fourier transform infrared spectroscopy – FTIR

The PW spectrum was obtained by using FTIR spectrometer (Bruker, Vertex 70v, Germany) equipped with attenuated total reflectance (ATR) accessory (Bruker, Platinum, Germany). The ATR crystal used was a diamond. The final spectrum is an average of 128 scans with 4cm^{-1} of spectral resolution from 400 to 4000 cm^{-1} (Ubaldini et al., 2012).

2.3 Ice cream Manufacture

In order to evaluate the effect of PW as a replacement of commercial emulsifier (CE), preliminary experiments were made to select which concentration of PW was necessary to obtain similar behavior compared to an ice cream made with 1.0% of CE. Five ice cream formulations were made, the control (PW0, without PW and 1% CE), PW0.25 (with 0.25% PW and 0.75% CE), PW0.5 (with 0.5% PW and 0.5% CE), PW0.75 (with 0.75%PW and 0.25% CE) and PW1.0 (with 1% PW and without CE).Concentration of PW was chosen by evaluation of texture profile (hardness and adhesiveness) and overrun parameters of ice cream formulation with similar behavior of ice cream control (PW0). Texture profile analysis was done using a Brookfield Texture Analyzer CT-III with acrylic circular probe TA4/1000 (38.1 mm in diameter and 20 mm in height) 50 g trigger and speed of 2 mm/s.

The formulation selected was the PW0.5, and with this formulation, reduction of the commercial was studied in order to analyze the PW emulsifier properties in ice cream. Four ice cream formulations were prepared (Table 2). Control formulation (0.5% CE and 0.0% PW) was also prepared.

Ice cream formulations were done by mixing pasteurized whole milk and milk cream under heating (40 °C, 10 min), dry ingredients (milk powder, sucrose, stabilizer, PW) and liquid ingredients (glucose syrup and invert sugar) were added and heated at 90 °C (10 min). Ice cream mix was refrigerated at 8 °C for 16 h (maturation) and commercial emulsifier was added and stirred thoroughly 10 min and frozen in a homestyle ice cream maker (Cuisinart, East Windsor, USA) during 25 min. Ice cream (30 g) were placed in polypropylene cups and stored at -17 °C for 48 h before analysis.

2.3.1 Overrun measurements

A fixed volume ice cream mix and frozen ice cream samples was weighed. Overrun values were calculated according to Equation 2 (Goff & Hartel, 2013).

$$\% \, Overrun = \frac{Ice\ cream\ mix\ (g) - Ice\ cream\ (g)}{Ice\ cream\ (g)} \qquad (2)$$

2.3.2 Melting properties

Samples (30 g) were placed on a wire screen (5 mm mesh) at 21 ± 1 °C. Melting mass and time were recorded every 1 min. Melting rate was obtained from the linear part of the melting curves (Tekin, Sahin & Sumnu, 2017).

2.3.3 Rheological properties

2.3.3.1 Continuous shear rheology

Ice cream samples were analyzed at -2 °C in rheometer (Mars II, Hacke Thermo Fisher Scientific Inc., Newington, Germany), with a parallel steel cone-plate geometry (35 mm, separated by 0.105 mm fixed gap). Flow curves for each sample were obtained in flow mode over shear rate ranging from 0 to 2000 s^{-1}, increased over 150 s, kept at the highest limit for 10 s, and then decrease over 150 s. The up curve was fitted by Herschel-Bulkley model as shown in Eq.(3):

$$\tau = \tau_0 + k\,\dot{\gamma}^{\,n} \qquad (3)$$

Where τ is the shear stress (Pa), τ_0 is the yield stress (Pa), k represents the consistency index $(Pa.s)^n$, $\dot{\gamma}$ is the rate of shear (s^{-1}), and n is the flow behavior index (dimensionless).

2.3.3.2 Oscillatory rheometry

Linear viscoelastic region (LVR), where storage modulus (G') and loss modulus (G") remains constant was determined. Deformation within the LVR region rate was selected for subsequent frequency sweep analyses from 0.1 to 10 Hz. G', G" and loss tangent (δ) were calculated using RheoWin 4.20.003 Haake® software.

2.3.3.3 Oscillatory thermo-rheometry

For the oscillatory thermo-rheometry, temperature was continuously increased from -10 °C to 5 °C. The heating rate in the test was 1 °C/min. Frozen ice creams were analyzed at a frequency of 1 Hz and 0,1% strain. Modulus G' and G" were calculated using RheoWin 4.20.003 Haake ® software.

2.4 Statistical analysis

Experiment was performed three times with three replicates per treatment. Analysis of variance (ANOVA) was performed using the general linear model with SPSS (v.19.0) (IBM SPSS Statistics, SPSS Inc., Chicago, USA) for Windows. Means and standard deviations were calculated for each variable. Differences were considered significant at $p < 0.05$ using the Tukey test.

3. Results and discussion

3.1 Orange juice pulp wash characterization

Composition of PW is shown in Table 1. PW is mainly composed of dietary fiber, about 57%. The ratio of insoluble dietary fiber (IDF) to soluble dietary fiber (SDF) can play an important role when applied in food products (La Peña, Odriozola-Serrano, Oms-Oliu & Martín-Belloso, 2020). PW has a ratio of 3.6:1 IDF to SDF. This can vary according to ripening, part of the fruit evaluated, growing area and extractor type in the juice processing (Hui, 2004). Dietary fiber is defined as plant cell wall polysaccharides, mainly represented by soluble fiber, pectins, β-glucans and insoluble fiber, cellulose, hemicellulose (Guardiola-Márquez, Santana-Gálvez & Jacobo-Velázquez, 2020). The soluble fraction in this co-product presents higher concentration allowing to improve more physiological effects when consumed (Grigelmo-Miguel & Martín-Belloso, 1999a). PW present important technological properties related to water retention, oil absorption and emulsion activity, enabling a new approach for application in emulsions systems. Functionality of PW for applications in bakery, dairy products and dressings is indicated due to high value for WRC, allowing better quality parameters related to reducing moisture of products, modify viscosity and texture and avoid syneresis (Grigelmo-Miguel & Martín-Belloso, 1999a, b, 1999; Lundberg, Pan, White, Chau & Hotchkiss, 2014). It can be associated with the content of soluble fiber (Table 1) and the parenchyma cells present in orange pulp, that are capable to retain the juice in the fruit tissue (Schalow, Baloufaud, Cottancin, Fischer & Drusch, 2018).

The value obtained for OAC is interesting for food applications, for improvement in stabilization of high-fat foods and emulsion (López-Marcos, Bailina, Viuda-Martos, Pérez-Alvarez & Fernández-López, 2015). Lignin, present in PW (Figure 1) can contribute for this behavior (Navarro-González, García-Valverde, García-Alonso & Periago, 2011). Cellulose, part of the insoluble fiber present in cell wall of fruits, have hydrogen bonds present intra and inter molecular in its structure and has hydrophobic characteristic (Takahashi, 2009).

Emulsifying functionality of citrus fiber can be explained due its water holding and swelling capacity, allowing them to expand and form a three-dimensional structure, avoiding flocculation and coalescence of droplets (Qi, Song, Zeng & Liao, 2021). Combination of Pickering mechanism effect may be related, once its solid particles can be partially wetted by oil and water, making an interface that allow to bound oil droplet and get water absorbed (Berton-Carabin & Schroën, 2015; Qi et al., 2021).

Color parameters were presented in Table 1. The ranges of color obtained allow the utilization of PW in various food products, with minor impacts in visual appearance (Lundberg et al., 2014). As the co-product is derived from citrus juice, the value of b* can be related to the carotene of orange fruit (López-Marcos et al., 2015).

3.1.2 FTIR-ATR spectroscopy

FTIR-ATR spectroscopy (Figure 1) indicates common structure of fibers, mainly cellulose and lignin (Yang, Yan, Chen, Lee & Zheng, 2007; Zapata, Balmaseda, Fregoso-Israel & Torres-García, 2009*)*. High energy range (3000-3600 cm^{-1}) is related to absorptions due to O-H stretching vibration, free and intermolecular bonded hydroxyl groups from carbohydrates. The peaks at 2920 and 2854 cm^{-1} was assigned to C-H stretching, which can be attributed to alkyl, aliphatic and aromatic compounds (Yang et al., 2007). The presence of the band at 1733 cm^{-1} can be assigned to C=O stretch vibration of carbonyls groups, such as ester. At 1620 cm^{-1} the observed band can be attributed to C=C stretching from aromatic rings (aliphatic/unsaturated) containing compounds (Yang et al., 2007*;* Zapata et al., 2009). The bands observed at 1098, 1050 and 1014 cm^{-1} are due to C-O stretching of cellulose and hemicellulose (Puccini et al., 2016) while at 1436 cm^{-1} is a CH_2 and CH_3 bending vibration of aliphatic chains, typical for lignin structure.

3.2 Choice of PW concentration

Texture parameters of hardness, adhesiveness and overrun presented similar behavior for PW0.25, PW0.5 and the control ice cream (PW0) (Figure 2). Hardness parameter brings measurements about deformation when external force is applied and adhesiveness can give understanding about the scoopability of ice cream. PW0.75 and PW1.0 present significant difference ($p < 0.05$) of hardness, adhesiveness and overrun compared with PW0. In view of the results presented in Figure 2, formulation PW0.5 obtained similar behavior than the formulation made with 1% of commercial emulsifier (PW0). PW0.5 formulation was selected to evaluate PW emulsifier proprieties (E0.5) in ice cream reducing commercial emulsifier from 0.5% until zero, maintaining 0.5% of PW (Table 2).

3.3 Ice cream physicochemical characteristics

Melting profile is represented in Figure 3 and values of melting rate and first drop times in Table 3. Ice creams with PW (E0.5, E0.25 and E0), showed more resistance to starting the melting, observed by first drop times, which can be due to the effect caused by water retention of PW. Ice cream E0.5 and E0 exhibited longest time to start melting. Furthermore, between samples with PW, melting rate had no difference between E0.5 and E0.25 ($p > 0.05$) and between E0.25 and E0. Higher melting rate means that heat transfers occurs quicker from the exterior of ice cream to the center of ice crystals, indicating smaller shape retention once it starts to melt (Loffredi et al., 2021; Muse & Hartel, 2004). Regarding to the control (C) and E0 formulations, they were similar related to melting rate. However, E0 showed significant difference ($p < 0.05$) for first drop times, which is a good quality parameter for ice cream, since it takes a long time to start melting.

Ice cream with PW showed a pH lower than C. The overrun measurements did not change ($p > 0.05$) for E0 and C formulations, demonstrating similar effect in this parameter for ice cream between commercial emulsifier and orange juice pulp wash.

3.3.1 Continuous shear rheology

Dimensionless parameter (n) can describe about flow behavior, and how close ($n = 1$) it can be to Newtonian fluid. As it can be seen in Table 4, all values were $n < 1$, they present pseudoplastic behavior. This characteristic is expected for ice cream production, with a lower flow viscosity than viscosity at rest, facilitating the mix flow during production. Formulation E0.5 had the lowest value for n, meaning a more notable shear-thinning behavior (Javidi, Razavi, Behrouzian & Alghooneh, 2016; Pereira, Resende, De Abreu, Giarola & Perrone, 2011).

Yield stress is a parameter related to the minimum stress required to start a flow (Da Silva, Cook & Bruschi, 2020). Formulations E0.5, E0.25 and E0 presented higher values ($p < 0.05$) than control (C). Higher values for the yield stress display a more structured sample and greater resistance to melt (Goff & Hartel, 2013). This can be explained due to the fiber present in PW (Table 1), they absorb water and expand, forming a network structure by swelling of orange fiber (Lundberg et al., 2014; Qi et al., 2021). As the mainly composition of PW is formed by polysaccharides, they are used in food matrix due to the capability to form gels and an increasing in its concentration can cause a hardness characteristic (Schalow et al., 2018). Formulation E0 was significant different than others ($p < 0.05$) and presented the highest value, indicating the firmest structure of them. Stiffer product displayed by the E0 formulation is probably due to presence of carrageenan gum and PW. Both are polysaccharides which can enhance viscosity and stabilize solutions. Stabilizer carrageenan gum, cellulose, pectin probably present in PW, is formed by linear chain. When in movement, these linear chains increase the occupied space and produce viscosity. Moreover, the ability to form gel and through swollen of fiber give a firmer structure, can be correlated with the increase of the yield stress (BeMiller & Huber, 2010). As this formulation did not have commercial emulsifier present, it is an interesting result because higher yield stress values and viscosity can be related to a good capability to stabilize emulsions and avoid phase separation (Kirtil & Oztop, 2016; Kurt & Atalar, 2018).

Consistency index parameter (k) brings information about the flow properties of samples as it is related with the viscosity of the material. Higher values mean that samples are more viscous. Formulations E0.5, E0.25 and E0 presented significant difference ($p < 0.05$) related to the control (C) sample meaning that inclusion of PW increase viscosity of them. Ice cream with addition of fiber was related to increase the solid content of the mix and have an extended network formed by hydrated fibers, which leads to increased consistency of the formulation (Kurt & Atalar, 2018; Soukolis et al., 2009).

Flow curves of shear stress as a function of shear rate is showed in Figure 4. Flows curve showed positive hysteresis area, profile of thixotropic fluids. The addition of PW increased hysteresis area, however no statistical difference was observed between formulations C, E0.5 and E0.25. Lower values for hysteresis indicate less difficulty to, after applied stress, restructure initial configuration. Formulation E0 had the highest value and statistical difference ($p < 0.05$) from others, suggesting that absence of commercial emulsifier led to a more pronounced characteristic of PW and stabilizer, implying that this formulation has the longest period of time for the restoration of the initial molecular configuration system (Dos Santos et al., 2020).

3.4.1 Oscillatory rheology

In the oscillation test, the storage and loss moduli G' and G" were measured. Formulations showed elastic modulus (G') greater than viscous modulus (G") (Figure 5A), which characterizes a viscoelastic system (Dos Santos et al., 2020).

Higher values for storage moduli G' and loss moduli G" were obtained for formulations with PW. An emulsion stabilized by citrus fiber in various conditions presented similar behavior as viscoelastic emulsion. Thus, the tight values of G' and G" for PW ice creams can be related to a stronger network structure formed by citrus fiber in continuous water phase (Qi et al., 2021).Values of the dynamic moduli showed a better network between milk and gum compared to a water and gum constituents of ice cream (Kurt & Kahyaoglu, 2016). Both moduli presented higher value for E0.5, E0.25 and E0, compared to the control, demonstrating an enhancement with PW inclusion.

Loss tangent is the division between G"/G', and values less than one means a structure with a predominantly elastic behavior, since G' is greater than G". For all formulations, this value was less than one and no crossover point was presented throughout the frequency range, which means a gel-like behavior (Figure 5B) (Qi et al., 2021).

3.4.2 Thermo-Oscyllatory rheology

Evaluation of storage (G') and loss (G") moduli were measured in the oscillation test for ice cream samples in an increasing temperature range of -10 °C to 5 °C (Figure 6). At lowest temperature, all water content of ice cream is frozen, values for G' are expected to be highest because of the maximum solids fraction (Wildmoser et al., 2004). During all temperature ramp test (Figure 6), G' was higher than G", presenting a viscoelastic behavior verified in the oscillatory rheology (Figure 5), which could be explained due to PW in the formulation. At the lowest temperature (-10 °C), values for storage moduli were higher than loss modulli, which indicates a solid-like ice cream.

Temperature between -10 °C and 0 °C is related to the melting of the ice phase as the temperature increases, also by fat destabilization, serum and air phase. Besides, steep curves are correlated to a quicker melting. This behavior is in accordance with the melting rate evaluated (Table 3), where C and E0 presented higher values and also show steep curves in the ramp test (VanWess, Rankin & Hartel, 2019; Wildmoser et al., 2004). Region between 0 °C and 5 °C, both moduli present a plateau level. At this temperature, all ice phase was melted and the rheological performance corresponds mainly to the disperse air and fat phase. Additionally, characteristic of melted ice cream remains as viscoelastic, maybe due to network formed by air cells, partial coalesced fat and also for fiber network structure, in the case of the samples which contains it (VanWess et al., 2019; Wildmoser et al., 2004).

4. Conclusion

Orange juice pulp wash utilized in this work is a source of dietary fiber, with capacity of water retention, oil absorption and emulsion activity. Ice creams presented a pseudoplastic behavior with increase of consistency with orange juice pulp wash inclusion. Formulation with orange juice pulp wash and without commercial emulsifier displayed the high yield stress and oscillatory modulus, indicating a strong structure formed. Rheology parameters displayed a viscoelastic property. Orange juice pulp wash has emulsifier propriety in ice cream, being a promising ingredient for food industry.

Acknowledgments

We thank the Coordination for the Improvement of High Educational Personnel (CAPES) by scholarship.

Declaration of interest

No conflict of interest exists.

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Figure caption

Figure 1. FTIR-ATR spectrum of orange juice pulp wash powder. ν and δ symbols means stretch and bend vibration modes, respectively. CH_n represents CH_2 and CH_3.

Figure 2. Textural properties (hardness, adhesiveness) and overrun of ice creams with orange juice pulp wash (PW) and commercial emulsifier (CE). PW0: without PW and 1% CE, PW0.25: 0.25% PW and 0.75% CE, PW0.5: 0.5% PW and 0.5% CE, PW0.75: 0.75% PW and 0.25% CE, PW1.0: 1% PW and without CE. Bars with same letters above did not differ $p < 0.05$.

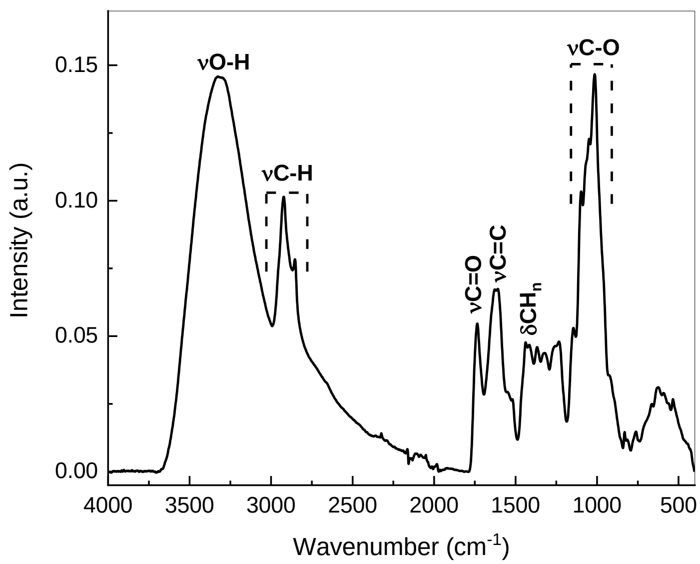
Figure 3. Melting behavior of ice cream with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction. C: control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW.

Figure 4. Continuous flow rheology of ice cream formulations with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction. Upward curve (solid line); Downward curve (dotted line). C: Control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW.

Figure 5. Oscillatory rheology curves of ice cream formulations with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction: (A) G' – storage module (solid symbols); G" – loss module (open symbols); (B) tan δ. C: Control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW.

Figure 6. Thermo-Oscillatory Rheology curves of ice cream formulations with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction. G' – storage module (solid symbols); G" – loss module (open symbols). C: Control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW.

Figure 1.



νO-H
νC-H
νC=O
νC=C
δCHn
νC-O
0.15
0.10
0.05
0.00
Intensity (a.u.)
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

Figure 2.

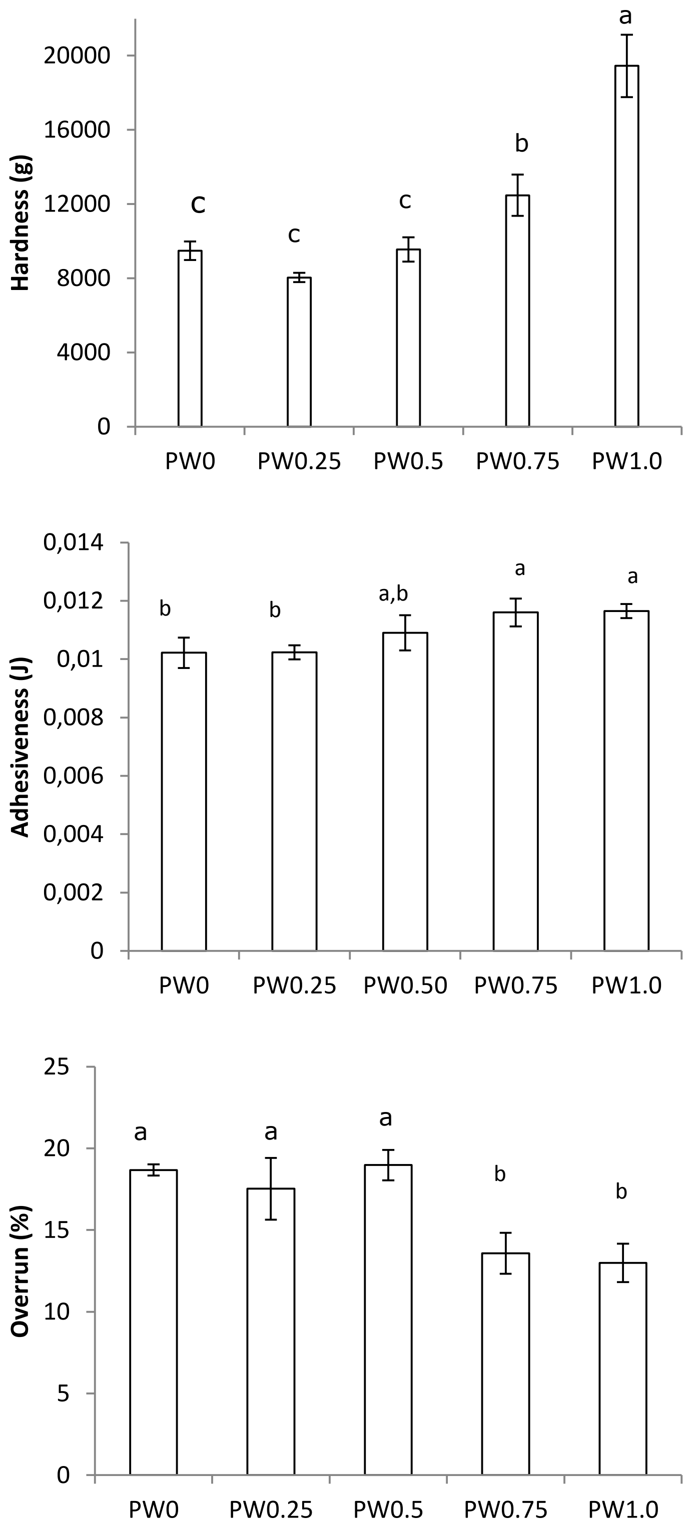

Hardness (g)
0
4000
8000
12000
16000
20000
c
c
c
b
a
PW0
PW0.25
PW0.5
PW0.75
PW1.0
Adhesiveness (J)
0
0,002
0,004
0,006
0,008
0,01
0,012
0,014
b
b
a,b
a
a
PW0
PW0.25
PW0.50
PW0.75
PW1.0
Overrun (%)
0
5
10
15
20
25
a
a
a
b
b
PW0
PW0.25
PW0.5
PW0.75
PW1.0

Figure 3.

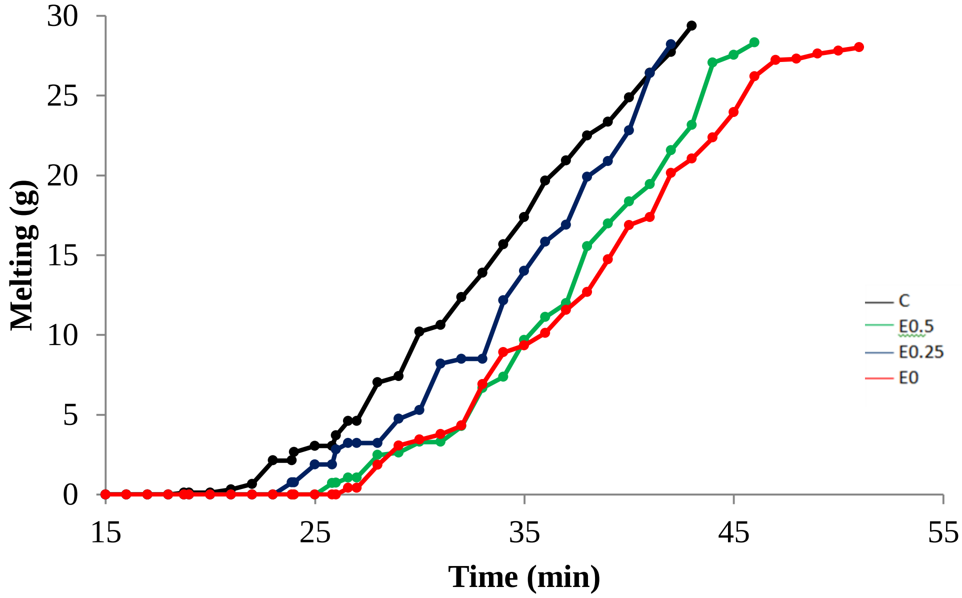


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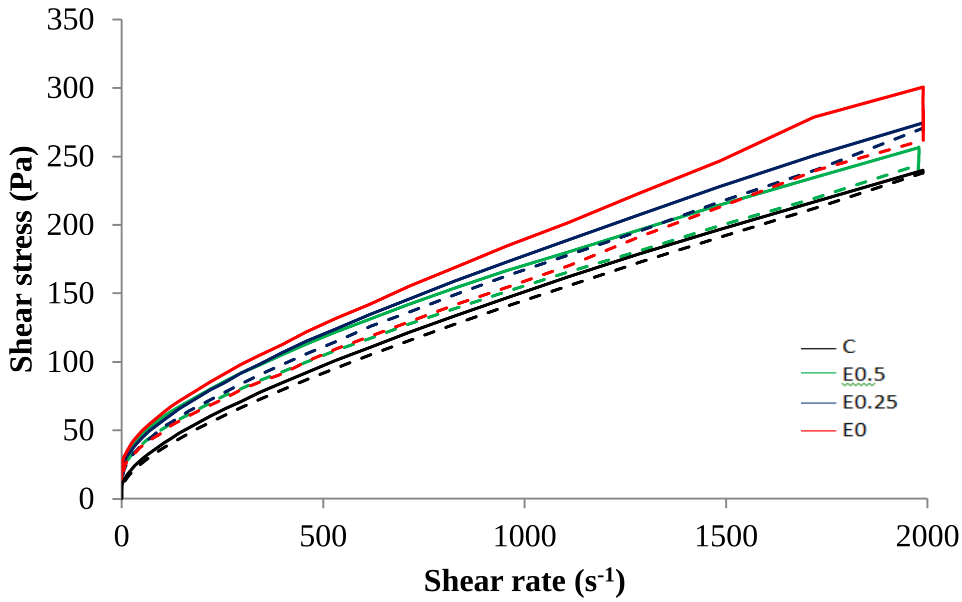
Figure 4.



350
300
250
200
150
100
50
0
Shear stress (Pa)
0
500
1000
1500
2000
Shear rate (s-1)
C
E0.5
E0.25
E0

Figure 5.

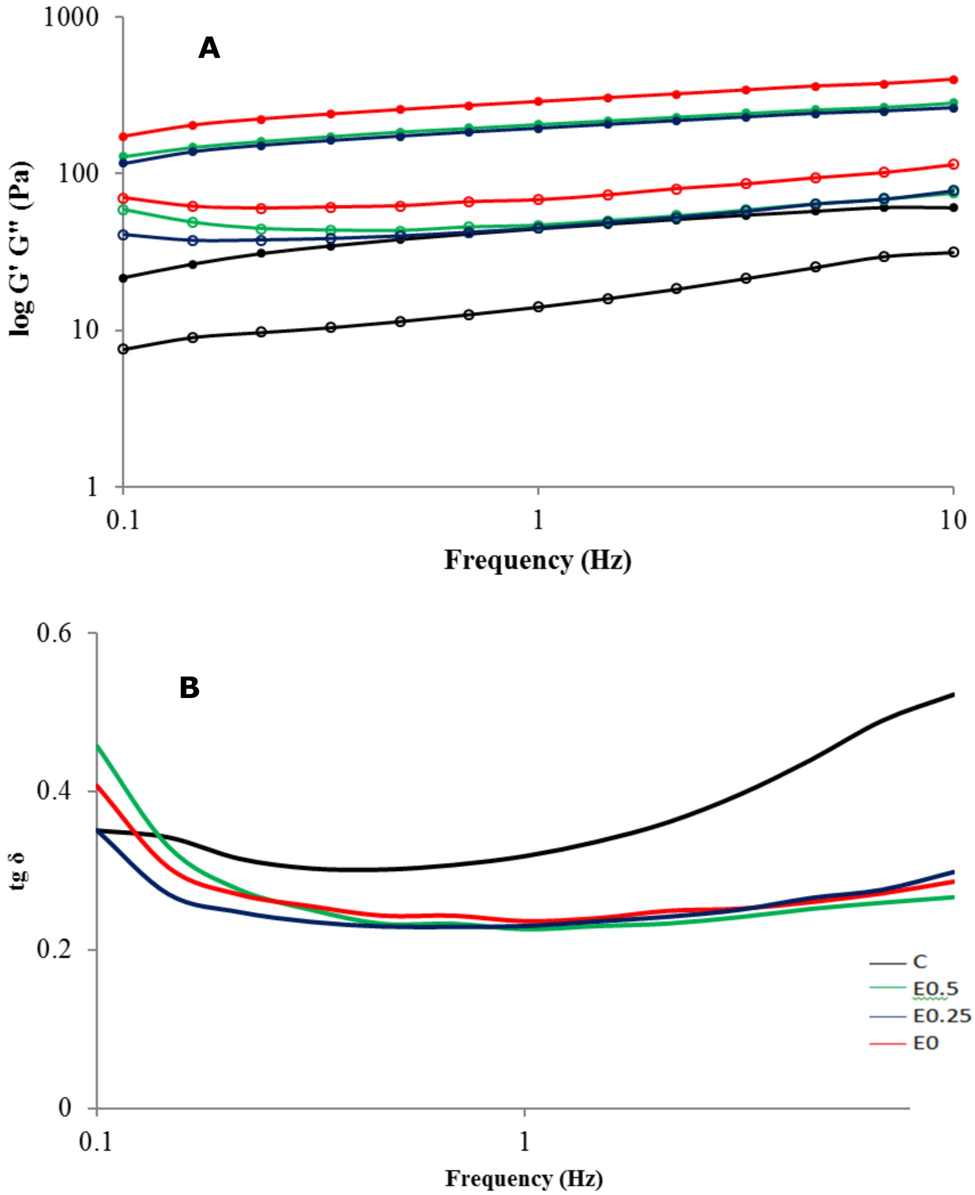

A
1000
100
10
1
log G' G" (Pa)
0.1
1
10
Frequency (Hz)
B
0.6
0.4
0.2
0
tg δ
0.1
1
Frequency (Hz)
C
E0.5
E0.25
E0

Figure 6.

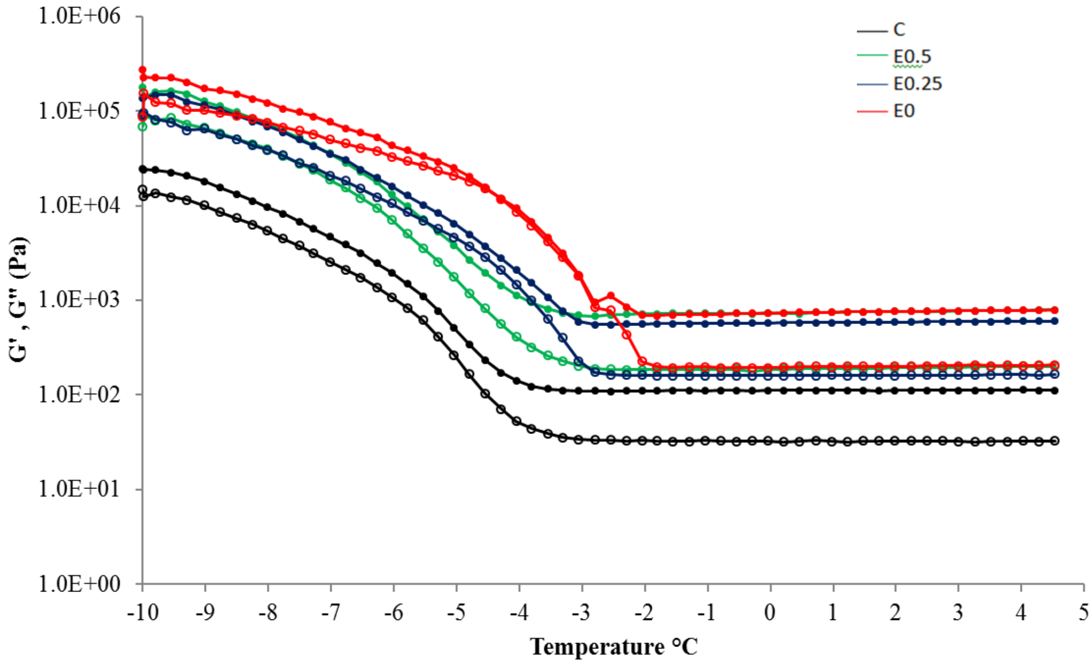

1.0E+06
1.0E+05
1.0E+04
1.0E+03
1.0E+02
1.0E+01
1.0E+00
G' , G'' (Pa)
-10
-9
-8
-7
-6
-5
-4
-3
-2
-1
0
1
2
3
4
5
Temperature °C
C
E0.5
E0.25
E0

Table 1. Physical-chemical and technological properties of orange juice pulp wash

	Results
Protein (%)	9.57±0.18
Ash (%)	3.28±0.09
Moisture (%)	8.11±0.19
Insolube Fiber (%)	45.91±0.85
Soluble Fiber (%)	12.78±0.09
Fat (%)	3.53±0.03
Carbohydrates (%)	16.82±0.79
Lightness (L*)	90.61±0.13
Redness (a*)	-4.73±0.02
Yellowness (b*)	57.42±0.05
pH	4.33±0.05
Emulsion Activity (%)	49.00±0.54
WRC (g/g)	14.18±0.47
OAC (g/g)	5.08±0.03

Results were expressed in dry matter as mean ± standard deviation.WRC: water retention capacity, OAC: oil absorption capacity.

Table 2. Ice cream formulations with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction

Ingredients (%)	C	E0.5	E0.25	E0
Whole milk	65	65	65	65
Sugar	10	10	10	10
Pasteurized fat milk	7.5	7.5	7.5	7.5
Whole milk powder	6.5	6	6.25	6.5
Invert sugar	5	5	5	5
Glucose syrup	5	5	5	5
Carrageenan gum	0.5	0.5	0.5	0.5
Commercial Emulsifier	0.5	0.5	0.25	0
Orange Pulp Wash	0	0.5	0.5	0.5

C: Control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW.

Table 3. Physicochemical characteristics of ice cream with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction

	Overrun (%)	pH	Melting rate (g/min)	First drop times (min)
C	13.46[c]	6.53[a]	0.61[a]	18.97[c]
E0.5	18.96[a]	6.32[c]	0.43[b]	25.78[ab]
E0.25	17.58[ab]	6.35[b]	0.51[ab]	23.51[b]
E0	15.42[bc]	6.33[bc]	0.57[a]	26.57[a]
SEM	0.539	0.014	0.026	1.127
P value	<0.0001	<0.0001	0.010	0.001

Means with different letters in the same column are significantly different ($p < 0.05$). SEM: standard error mean C: control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW

Table 4. Rheological parameters of ice cream with orange juice pulp wash (0.5%) (PW) and commercial emulsifier (CE) reduction

	r^2	τo(Pa)	*k*([Pa.s]^n)	*n*(dimensionless)	Hysteresis x 10^3 (Pa.s)
C	0.998	4.64^c	1.89^c	0.631^a	10.2^b
E0.5	0.995	9.22^b	4.88^a	0.509^c	26.9^b
E0.25	0.996	9.83^b	3.58^b	0.560^b	18.5^b
E0	0.993	13.64^a	3.28^b	0.581^b	55.3^a
SEM	-	1.214	0.405	0.017	6.59
P value	-	<0.0001	0.001	0.001	0.005

Means with different letters in the same column are significantly different ($p < 0.05$). SEM: standard error mean C: control without PW; E0.5: ice cream with 0.5% CE and 0.5% PW; E0.25: ice cream with 0.25% CE and 0.5% PW; E0: ice cream without CE and with 0.5% PW. τo: yield value; *k:* consistency index; *n:* flow behaviour index.