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GERLANDIA DA SILVA PEREIRA

CARACTERIZAÇÃO SENSORIAL E REOLÓGICA DE SOLUÇÕES DE ESPESSANTES E ACIDULANTES COM SACAROSE

SENSORY AND RHEOLOGICAL CHARACTERIZATION OF THICKENERS AND ACIDULANTS SOLUTIONS WITH SUCROSE

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SENSORY AND RHEOLOGICAL CHARACTERIZATION OF THICKENERS AND ACIDULANTS SOLUTIONS WITH SUCROSE

Tese apresentada à Faculdade de Engenharia de Alimentos da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Doutora em Alimentos e Nutrição, na Área de Consumo e Qualidade de Alimentos.

Thesis presented to the Faculty of Food Engineering of the University of Campinas in partial fulfillment of the requirements for the degree of Doctor, in the area of Food and Nutrition.

Orientadora: Profa. Dra. Helena Maria André Bolini

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RESUMO

Na indústria alimentícia, os aditivos alimentares têm um alto potencial de aplicação. Os espessantes são utilizados principalmente para modificar textura e conferir viscosidade aos alimentos. No caso dos ácidos orgânicos, eles atuam principalmente como acidulantes e reguladores de acidez. O uso de tecnologias emergentes, como o homogeneizador de alta pressão, tem sido estudado para melhorar os processos tecnológicos existentes ou para aplicação no desenvolvimento de produtos, enquanto mantém atributos de qualidade nutricional e sensorial dos alimentos. Nesse tipo de tecnologia, o fluido é bombeado passando através de uma válvula de orifício estreito, induzindo o aumento da velocidade, seguida de despressurização, causando cisalhamento e cavitação. Esses efeitos promovem mudanças físicas no produto, como a alteração da viscosidade. Assim sendo, este estudo objetivou avaliar a influência do processo de homogeneização nas propriedades reológicas e sensoriais em sistemas modelos para aplicação em néctar de fruta. Diferentes soluções de espessantes acidificadas com ácidos orgânicos e adicionadas de sacarose foram avaliadas quanto ao perfil temporal e comportamento reológico. Para a elaboração dos sistemas modelos, foram utilizados os espessantes goma guar (0,1 %), goma xantana (0,2 %) e goma gelana (0,05 %); os acidulantes (ácido cítrico, málico e tartárico) (0,3 %) foram utilizados separadamente em cada solução de espessante. Os sistemas modelos foram adoçados com sacarose (10 %). Após o preparo das amostras, estas foram homogeneizadas (0 - controle, 25 e 50 MPa) à 25 ºC, e logo após armazenadas sob refrigeração (7 ºC) por 21 dias. Ao final do processo foram obtidas 9 amostras para cada espessante utilizado. Os sistemas modelos foram submetidos à análise reológica e avaliados sensorialmente através da análise tempo-intensidade, determinando assim as características dos atributos gosto ácido, gosto doce e viscosidade em função do tempo. O processo de homogeneização alterou a percepção temporal da viscosidade. Verificou-se que os sistemas modelos submetidos ao processo de homogeneização apresentaram menor intensidade para o atributo viscosidade, caracterizando-se como menos viscosos. Por outro lado, as amostras controle e homogeneizadas apresentaram perfis temporais similares para o atributo gosto doce. A percepção sensorial do gosto ácido demonstrou maior intensidade nos sistemas modelos homogeneizados, destacando-se pela acidez mais intensa e prolongada. As amostras apresentaram comportamento pseudoplástico e foram descritas pelo modelo de Ostwald-de-Waele. Os diferentes ácidos orgânicos não interferiram no comportamento reológico dos sistemas modelos. Além disso, a viscosidade aparente reduziu com o aumento da pressão de

homogeneização. Sendo assim, o estudo do perfil sensorial e reológico torna-se importante, não só para qualidade do produto, mas também para otimização de processos e aplicações industriais. Portanto, pesquisas com diferentes soluções de espessantes, acidificadas com ácidos orgânicos, consistem numa ferramenta importante para o desenvolvimento de néctares de frutas com características sensoriais específicas de gosto doce, gosto ácido e viscosidade.
Palavras-chave: espessantes, ácidos orgânicos, homogeneização, análise tempo-intensidade, reologia.

ABSTRACT

In the food industry, food additives have a high potential for application. Thickeners are mainly used to modify texture and impart viscosity to the foods. In the case of organic acids, they act mainly as acidulants and acidity regulators. The emerging technologies, such as the high pressure homogenizer, has been studied to improve existing technology processes or for application in the development of products, while maintaining attributes of nutritional and sensory quality of food. In this type of technology, the fluid is pumped through a narrow orifice valve, inducing the speed increase, followed by depressurization, causing shear stress and cavitation. These effects promote physical changes in the product, such as change in viscosity. Therefore, this study aimed to evaluate the influence of the homogenization process on the rheological and sensorial properties in model systems for application in fruit nectar. Different solutions of thickeners acidified with organic acids and added of sucrose were evaluated for the temporal profile and rheological behavior. The thickeners guar gum (0.1 %), xanthan gum (0.2 %) and gellan gum (0.05 %) were used for the elaboration of the model systems; the acidulants (citric, malic and tartaric acid) (0.3 %) were used separately in each thickener solution. The model systems were sweetened with sucrose (10 %). After the samples were prepared, they were processed in a high pressure homogenizer (0 - control, 25 and 50 MPa) at 25 ºC, and then stored under refrigeration (7 ºC) for 21 days. At the end of the process, 9 samples were obtained for each thickener used. The model systems were submitted to rheological analysis and evaluated sensorially through the time-intensity analysis, thus determining the characteristics of the attributes acid taste, sweet taste and viscosity as a function of the time. The homogenization process altered the temporal perception of viscosity. It was verified that the model systems submitted to the homogenization process presented lower intensity for the viscosity attribute, characterizing them as less viscous. On the other hand, the control and homogenized samples presented similar temporal profiles for the sweetness attribute. The sensorial perception of the sourness demonstrated greater intensity in the homogenized models systems, detaching the more intense and prolonged acidity. The samples showed pseudoplastic behavior and were described by the Ostwald-de-Waele model. The different organic acids did not interfere in the rheological behavior of the model systems. In addition, the apparent viscosity decreased with the increase of the homogenization pressure. Therefore, the study of the sensorial and rheological profile becomes important, not only for the quality of the product, but also for the optimization of processes and industrial applications. Therefore, researches with different solutions of thickeners, acidified with

organic acids, are an important tool for the development of fruit nectars with specific sensorial characteristics of sweet taste, acid taste and viscosity.

Keywords: thickeners, organic acids, homogenization, time-intensity analysis, rheology.

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SUMÁRIO

1. Introdução

Os aditivos alimentares representam o conjunto de substâncias adicionadas aos alimentos sem o propósito de nutrir, mas com o objetivo de modificar ou manter as características físicas, químicas, biológicas ou sensoriais desses produtos. Do ponto de vista tecnológico, os aditivos alimentares têm um papel importante no desenvolvimento de alimentos e justificam-se por razões tecnológicas, nutricionais ou sensoriais, desde que sejam utilizados aditivos autorizados e em concentrações de ingestão diária aceitável (BRASIL, 1997). Logo, os aditivos são substâncias agregadas aos alimentos com fins específicos em complementação aos benefícios inerentes dos demais ingredientes.

Os espessantes são aditivos alimentares com as funções de espessar e estabilizar, mesmo em baixas concentrações são capazes de aumentar a viscosidade de soluções, emulsões e suspensões, melhorando a textura dos produtos (HONG *et al*., 2012). As gomas são compostos poliméricos que, quando dissolvidos ou dispersos em água, formam soluções ou dispersões viscosas. Tem a função de conferir viscosidade, espessar e em alguns casos formar gel. A importância das gomas baseia-se nas suas habilidades de controlarem as características reológicas de sistemas aquosos por meio de estabilização de emulsões, suspensão de partículas, controle de cristalização e inibição da sinérese (LUCCA; TEPPER, 1994). As gomas são empregadas comercialmente em diversos setores industriais, com grande aplicação no ramo alimentício, onde podem ser utilizadas como substitutos de gordura e açúcar, em produtos com baixo teor de lipídios e reduzido valor calórico (LETHUAUT *et al*., 2003; ALEXANDER, 1997; THEBAUDIN *et al*., 1997).

Por sua vez, os acidulantes são substâncias capazes de aumentar a acidez de um alimento ou conferir sabor ácido (BRASIL, 1997). Os ácidos orgânicos são substâncias naturalmente presentes em vegetais e podem ser utilizados como acidulantes e reguladores de acidez. Os ácidos orgânicos podem ainda exercer ação conservante nos alimentos pela redução do pH, protegendo os mesmos contra o crescimento de microrganismos; podem também melhorar sua palatabilidade (DAMODARAN; PARKIN; FENNEMA, 2010).

Pesquisas utilizando tecnologias não térmicas têm sido estudadas em produtos de frutas (ZHOU *et al*., 2017; GUAN, *et al*., 2016; NAVARRO *et al*., 2014; PATRIGNANI *et al*., 2013; CALLIGARIS *et al*., 2012; MCKAY *et al*., 2011), tanto para o desenvolvimento de produtos ou para melhorar processos tecnológicos existentes. No homogeneizador de alta pressão, o fluido é bombeado passando através de uma válvula de orifício estreito, induzindo um aumento da velocidade seguida de despressurização com consequente cavitação e alta

tensão de cisalhamento (AUGUSTO *et al*., 2013). Esses efeitos promovem mudanças nas propriedades reológicas e físicas dos produtos. Portanto, esse tipo de tecnologia tem sido utilizada para a preparação ou estabilização de emulsões e suspensões, assim como para promover alterações físicas no produto, por exemplo, a alteração da viscosidade (DIELS & MICHIELS, 2006). Destaca-se também que o processamento de alimentos através de tecnologias não-térmicas tem se destacado pela preservação e modificação das propriedades funcionais dos componentes alimentares e pela manutenção dos atributos de qualidade dos alimentos.

Desse modo, o processamento em homogeneizador de alta pressão apresenta-se como uma alternativa para modificar as propriedades reológicas em sistemas modelos. Este estudo propõe avaliar as alterações e as percepções sensoriais nos sistemas modelos submetidos à homogeneização. Assim, soluções de espessantes com ácidos orgânicos e sacarose foram submetidas ao processamento em homogeneizador de alta pressão. A preferência pelos espessantes (goma guar, xantana e gelana) foi em função da viscosidade, disponibilidade para a indústria, características sensoriais e aspectos legais. Para os acidulantes (ácido cítrico, málico e tartárico), a escolha foi em função do poder acidulante e por serem os principais ácidos orgânicos utilizados em alimentos.

A análise sensorial tem sido utilizada como ferramenta para avaliar o desenvolvimento de novos produtos e aceitabilidade entre os consumidores, tendo relevante importância no processo industrial, com diversas técnicas que avaliam o produto quanto à sua qualidade sensorial. Os métodos sensoriais podem ser usados para determinar se os alimentos diferem em sabor, odor, suculência, textura, entre outros atributos; em que medida diferem os alimentos e verificar as preferências dos consumidores (VACLAVIK; CHRISTIAN, 2014).

A análise tempo-intensidade é um método que quantifica as intensidades percebidas das caracterísitcas sensoriais de um produto em função do tempo. Tem sido utilizada para a caracterização de alimentos e no desenvolvimento de novos produtos (LAWLESS; HEYMANN, 2010). A percepção do sabor, aroma e textura dos alimentos é modificada continuamente em função do tempo. Com isso, a análise tempo-intensidade tem sido utilizada em alimentos com o propósito de medir a velocidade, duração e a intensidade percebida por um único estímulo (AMERINE; PANGBORN; ROESSLER, 1965). Várias pesquisas têm utilizado essa técnica e em diferentes produtos (AZEVEDO *et al*., 2017; RODRIGUES *et al*., 2015; MORAIS *et al*., 2014; SOUZA *et al*., 2013; SOKOLOWSKY & FISCHER, 2012; CADENA & BOLINI, 2011), principalmente para avaliar o perfil temporal do gosto doce, ácido e amargo.

Portanto, considerando a relevância da utilização de tecnologias não térmicas e da grande diversidade de espessantes e acidulantes que podem ser aplicados em alimentos, torna-se de extrema importância o desenvolvimento de pesquisas que avaliem as características temporais e reológicas de sistemas modelos para néctar de fruta. Assim, esta pesquisa é inovadora e consiste numa ferramenta útil para o desenvolvimento de produtos tipo néctares com características sensoriais específicas de gosto doce, gosto ácido e viscosidade.

2. Objetivos

2.1. Objetivo Geral

Avaliar a influência do processo de homogeneização nas propriedades reológicas e sensoriais em diferentes soluções de espessantes (goma guar, xantana e gelana) acidificadas com ácidos orgânicos (ácido cítrico, málico e tartárico) e adicionadas de sacarose.

2.2. Objetivos Específicos

1. Desenvolver os sistemas modelos para aplicação em néctares de frutas.
2. Determinar o efeito das pressões de homogeneização no perfil sensorial dos sistemas modelos.
3. Determinar o perfil temporal dos sistemas modelos através do método tempo-intensidade para múltiplos estímulos (gosto ácido, gosto doce e viscosidade).
4. Determinar o comportamento reológico, o índice de consistência (k) e o índice de comportamento (n) nos sistemas modelos para néctar de fruta.
5. Avaliar o efeito das diferentes pressões e a influência do processo de homogeneização na viscosidade aparente dos sistemas modelos.
6. Avaliar o impacto de diferentes viscosidades na percepção de outros atributos sensoriais.
7. Relacionar os resultados sensoriais e reológicos dos sistemas modelos.

3. Revisão Bibliográfica

3.1. Espessantes

Na indústria de alimentos, os hidrocoloides são utilizados em funções específicas como agentes modificadores de textura, inibição da cristalização, estabilização de emulsões e espumas, encapsulação de sabores e aromas. Essas propriedades fazem dos polissacarídeos importantes aditivos em processamento e armazenamento de alimentos (DAMODARAN; PARKIN; FENNEMA, 2010; BELITZ; GROSCH; SCHIEBERLE, 2009). Além disso, alguns hidrocoloides podem produzir géis dependendo de fatores como pH, concentrações de sais, açúcares e disponibilidade de água da solução, por isso apresentam grande aplicação na indústria de alimentos. Sendo assim, os hidrocoloides modificam e controlam as propriedades reológicas dos sistemas aquosos onde são inseridos (ROJAS, 2014).

Os hidrocoloides polissacarídicos são polímeros de cadeia longa e elevada massa molar. Podem ser extraídos de plantas terrestres ou aquáticas, ou ainda, serem produzidos por síntese microbiana. Por serem hidrofílicos, dissolvem-se ou dispersam-se em água, dando o efeito espessante e estabilizante, e em alguns casos, gelificante (BOBBIO; BOBBIO, 1992, DAMODARAN; PARKIN; FENNEMA, 2010). As propriedades conferidas pelos espessantes devem se manter mesmo sob extremos de temperatura, pH e força iônica, presença de sais e de outros componentes do alimento ao qual serão adicionados (BOBBIO; BOBBIO, 1992).

Os espessantes são substâncias que aumentam a viscosidade de um alimento. Por sua vez, esses podem agir como estabilizantes, ou seja, são substâncias que torna possível a manutenção de uma dispersão uniforme de duas ou mais substâncias imiscíveis em um alimento (BRASIL, 1997). São compostos hidrossolúveis e hidrofílicos usados para estabilizar, dispersar e evitar a sedimentação de substâncias em suspensão. A sua principal propriedade é a facilidade de hidratação para formar suspensões aquosas de alta viscosidade a baixos níveis de concentração (VELÁSQUEZ, 2009).

A seleção das gomas para a aplicação em alimentos envolve dentre outros fatores, a aparência do produto final, compatibilidade com os constituintes funcionais do sistema, conservação, aspectos legais, custo, estabilidade, umidade, odor, propriedade emulsificante, sensação bucal, tipo de aplicação e a viscosidade (PENNA, 2002).

Os hidrocoloides são aplicados como emulsificantes e estabilizadores de emulsões (DICKINSON, 2009), em produtos que incluem bebidas carbonatadas (TAN, 2004), sorvete (GOFF, 1997), molhos e temperos (SIKORA *et al*., 2008). Os espessantes são utilizados

como substitutos de gorduras e de carboidratos de baixo peso molecular, em alimentos de baixo teor de lipídios e reduzido valor calórico.

3.1.1. Goma Guar

A goma guar é um polissacarídeo natural, obtida do endosperma das sementes da leguminosa *Cyamopsis tetragonolobus*. Possui alto peso molecular, é formada de cadeia linear de manose (β-1,4) com resíduos de galactose como cadeias laterais, na proporção de uma unidade de galactose para duas de manose. Quanto maior a relação molar galactose/manose, maior a solubilidade em água. A goma guar é um polissacarídeo linearmente ramificado, isto é, um polímero de cadeia longa e com cadeias laterais curtas, essas características são responsáveis pela alta viscosidade da solução de goma guar (BELITZ; GROSCH; SCHIEBERLE, 2009; IZYDORCZYK; CUI; WANG, 2005).

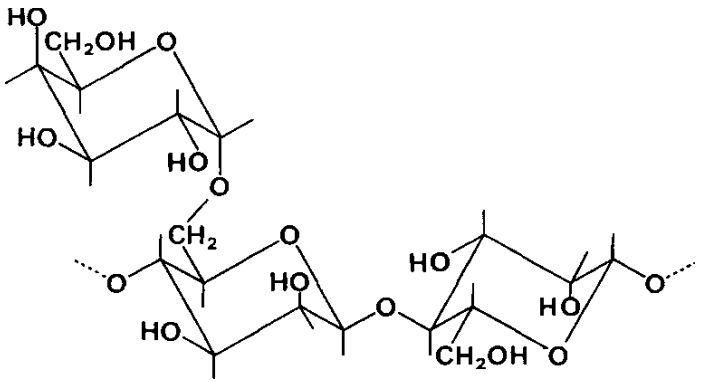

Figura 1 – Estrutura da Goma Guar
Fonte: (BELITZ; GROSCH; SCHIEBERLE, 2009).

A goma guar tem a capacidade de se hidratar em água fria, formando dispersões viscosas, cuja viscosidade depende da taxa de cisalhamento. Apresenta cadeias longas e bastante rígidas que fornecem soluções viscosas a baixas concentrações (0,05 - 0,25 %) (BeMILLER; WHISTLER, 1996). Ela é considerada de baixo custo, além de ser capaz de formar soluções com capacidade de retenção de umidade e estabilizar suspensões. Os estabilizantes evitam que com o tempo os ingredientes se separem em diferentes fases, onde atuam promovendo uma interação homogênea entre os ingredientes.

A goma guar é compatível com outras gomas, amidos, hidrocoloides e agentes gelificantes, aos quais pode ser associada para enriquecer a sensação tátil bucal, textura e para

modificar e controlar o comportamento da água em alimentos (MARUYAMA *et al*., 2006). A goma guar é utilizada em produtos como sopas, alimentos com baixa caloria, molhos para saladas, bebidas, sucos de frutas e produtos de panificação (IZYDORCZYK; CUI; WANG, 2005).

Wang *et al*. (2000) avaliaram a estabilidade de goma guar em soluções diluídas à diferentes temperaturas e pH. Os autores observaram estabilidade das soluções em pH 3,5 na temperatura de 50 ºC e em pH 2,0 na temperatura de 25 ºC. Lv *et al*. (2017) avaliaram o efeito da goma guar nas propriedades físicas e estabilidade de suco de laranja. E os resultados indicaram que a goma guar pode ser usada para substituir parcialmente o carboximetilcelulose (CMC) e melhorar a estabilidade e as propriedades físicas do suco de laranja.

3.1.2. Goma Xantana

A xantana é um heteropolissacarídeo hidrossolúvel com extrema importância comercial (GARCÍA-OCHOA *et al*., 2000), de origem microbiana, produzida pela bactéria *Xanthomonas campestris*, através de fermentação aeróbia. É constituída por uma cadeia principal de unidades de D-glucose unidas entre si por ligações β-1,4; a cadeia lateral é formada por resíduos alternados de D-manose e ácido D-glucurônico, na proporção molar de 2:1, possui ainda grupos acetil e pirúvico (KATZBAUER, 1998; MARCOTTE *et al*., 2001).

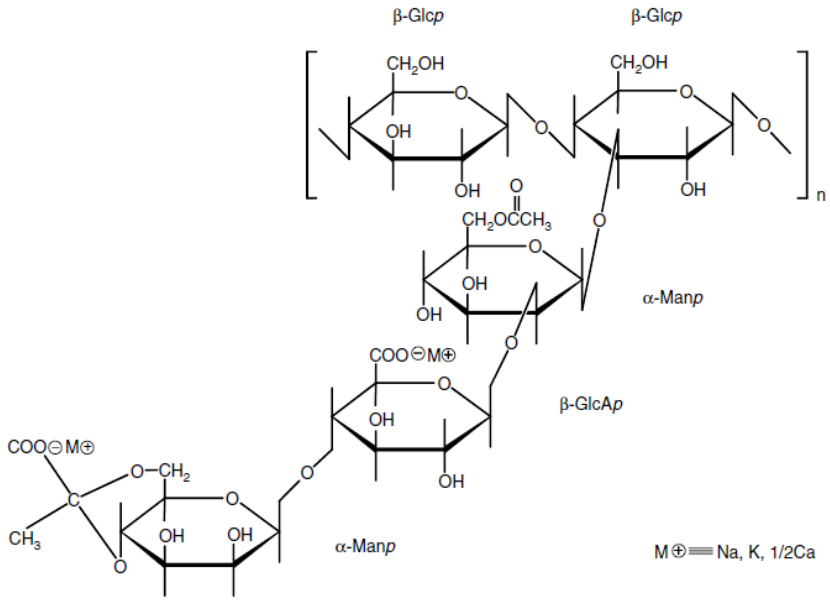

Figura 2 – Estrutura da Goma Xantana.

Fonte: (IZYDORCZYK; CUI; WANG, 2005).

A preferência por biopolímeros microbianos se deve às suas propriedades funcionais, somadas às vantagens de suas propriedades físico-químicas reprodutíveis, além de apresentar fontes estáveis. A produção de polímeros por microrganismos permite o controle dos parâmetros de produção, tais como tempo, temperatura, pH, teor de oxigênio, entre outros (MORRIS, 1995).

A utilização da xantana se deve principalmente a suas propriedades reológicas, que permitem a formação de soluções viscosas a baixas concentrações (0,05 - 1,0 %). Além disso, a goma é extremamente solúvel, tanto em água quente quanto fria, este comportamento está relacionado com a natureza polieletrólita da molécula (GARCÍA-OCHOA *et al*., 2000).

O valor calórico da goma xantana é aproximadamente 0,6 kcal/g, não apresenta digestibilidade, tem sido utilizada para baixar o valor calórico dos alimentos e melhorar o trânsito gastrointestinal (KATZBAUER, 1998). É estável em temperaturas de 0 a 100°C, o que a torna única entre as gomas utilizadas em alimentos, solubilidade e estabilidade em sistemas ácidos, mesmo na presença de sais; interação com outras gomas, capacidade de estabilizar dispersões, suspensões e emulsões aquosas (BeMILLER; WHISTLER, 1996). Apresenta excelente estabilidade a variações de pH (1 a 13), a cisalhamento prolongado e temperaturas elevadas (GARCÍA-OCHOA *et al*., 2000; KATZBAUER, 1998).

Segundo Sanderson (1981), a conformação das cadeias da goma xantana quando dispersa em solução é responsável pela alta viscosidade em repouso e baixa viscosidade em cisalhamento. Essa conformação é responsável pelo comportamento pseudoplástico da goma, ou seja, diminui a viscosidade com o aumento da taxa de deformação. A pseudoplasticidade melhora as características sensoriais pela percepção de menor viscosidade ao paladar e realça o sabor. A sua estrutura ramificada e o alto peso molecular conferem à goma xantana uma alta viscosidade, mesmo em baixas concentrações. A rede tridimensional formada por associações das cadeias de goma xantana tem eficiente estabilidade para suspensões e emulsões (KATZBAUER, 1998).

A goma xantana em associação com outras gomas proporciona textura lisa e cremosa a alimentos líquidos (CÂNDIDO; CAMPOS, 1995). As aplicações da goma xantana incluem molhos para salada, bebidas de frutas, produtos de panificação, misturas para bolo, geleias, sobremesas, coberturas, produtos lácteos, produtos cárneos e enlatados, confeitos e sopas (KATZBAUER, 1998). As propriedades pseudoplásticas facilitam a produção de queijos e patês. Em alimentos congelados, a adição de xantana confere estabilidade devido à ligação da água livre, evitando assim a sinérese, comum a estes produtos. Em bebidas, a goma xantana é eficaz para suspender polpa de fruta, atua também como espessante e realça o sabor

das bebidas. Isso é interessante para bebidas de baixa caloria, em que os açúcares são totalmente ou parcialmente substituídos por edulcorantes, resultando em uma bebida menos viscosa (KATZBAUER, 1998). Desse modo, a goma xantana tem sido utilizada como agente espessante, estabilizante e emulsificante, possibilitando a criação de novos produtos com texturas diferenciadas.

Weber, Queiroz & Chang (2008) estudaram os efeitos das gomas guar e xantana na estabilidade de diferentes géis de amido durante os processos de congelamento e descongelamento. Segundo esses autores, a goma xantana foi mais efetiva em reduzir a sinérese nos géis. Portanto, a goma xantana tem alto potencial de aplicação em alimentos, sendo utilizada em várias pesquisas e em diferentes tipos de produtos.

3.1.3. Goma Gelana

A goma gelana é um polissacarídeo aniônico extracelular secretado pela bactéria *Sphingomonas elodea*, e sua estrutura linear é baseada na unidade de repetição de tetrassacarídeos: 1,3 β-D-glicose; 1,4 β-D-ácido glucurônico; 1,4 β-D-glicose e 1,4 α-L-ramnose (GARCÍA *et al*., 2011; NODA *et al*., 2008; YAMAMOTO; CUNHA, 2007).

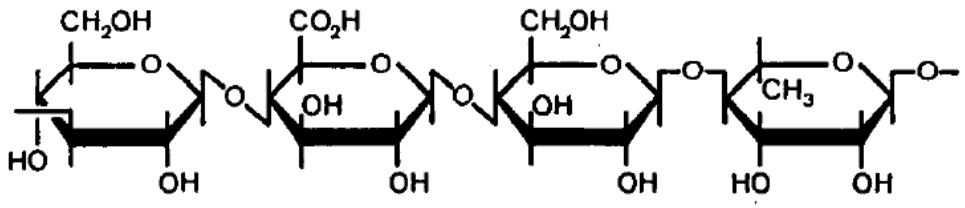

Figura 3 - Unidade de repetição química de tetrassacarídeos de goma gelana.
Fonte: (MORRIS, 1991).

A goma gelana é um hidrocoloide multifuncional com aplicação gelificante, espessante e estabilizante. As propriedades funcionais são manifestadas em concentrações muito baixas, ao nível de 0,05 % forma soluções aquosas de baixa viscosidade a elevada temperatura, que sob resfriamento origina géis. Uma das principais vantagens da goma gelana é ser capaz de formar géis com características diferentes. Gelana com maior quantidade de grupo acilo normalmente produz géis elásticos, flexíveis, não quebradiços e opacos, assim como gelana com menos grupo acilo formam géis não elásticos, firmes, quebradiços e

translúcidos (OGAWA *et al*., 2006; PHILLIPS; WILLIAMS, 2009). Muitos fatores podem afetar as propriedades de gelificação dos polissacarídeos, dentre os quais podemos citar a estrutura molecular, massa molar, temperatura, qualidade do solvente, presença de polieletrólitos, pH e força iônica (BeMILLER; WHISTLER, 1996).

O processo de gelificação consiste na formação de duplas-hélices ordenadas (transição conformacional), seguida pela interação entre as hélices (transição sol – gel). A transição conformacional é dependente do aumento da temperatura da solução, da concentração do polímero e da composição da solução. Ademais, a interação entre as duplas-hélices depende da presença de cátions na solução que promovem a formação de zonas de junção, com consequente formação da rede. Valores baixos de pH reduzem a mobilidade das cadeias de gelana, permitindo a agregação e gelificação. A redução do pH gera dissociação dos grupos carboxílicos laterais das moléculas de gelana, tornando-a um polieletrólito menos aniônico. Com isso, a redução da repulsão eletrostática intermolecular favorece a formação de duplas-hélices pela associação de duas moléculas, com a presença de pontes de hidrogênio entre o ácido glucurônico de uma cadeia e a glicose e a ramnose de outra cadeia. Além do mais, íons H^+ se ligam à superfície das hélices individuais, reduzindo assim a barreira eletrostática para a agregação das hélices, tornando possível a formação de zonas de junção. Interações, incluindo pontes de hidrogênio, ligam as cadeias às zonas de junção, resultando na gelificação (YAMAMOTO, 2006; PERFEITO, 2014).

Os géis de gelana promovem a liberação do sabor das frutas, são límpidos, estáveis em ampla faixa de pH sob aquecimento. A redução de pH de dispersões de gelana até valores próximos a 4,0 resulta no aumento da força dos géis, independentemente do tipo de acidificação empregada (MORITAKA *et al*., 1995; YAMAMOTO; CUNHA, 2007).

A goma gelana é um polissacarídeo que tem ampla utilização na indústria de alimentos e em aplicações biotecnológicas por formar um gel transparente que resiste ao aquecimento e a ácidos. Devido à gelana formar géis a baixas concentrações, juntamente com suas propriedades funcionais versáteis, permite a obtenção de novas texturas e, consequentemente, a geração de novos produtos. O uso desse polissacarídeo em combinação com outros hidrocoloides pode melhorar a estabilidade, a formação da estrutura e a liberação de sabor em sistemas alimentícios (YAMAMOTO, 2006). As aplicações da goma gelana estão relacionadas às propriedades espessantes, estabilizantes e gelificantes em produtos alimentícios, incluindo glacês, geleias, compotas, recheios de tortas, confeitos, doces, barrinhas de frutas, sorvetes, iogurtes, hidrogéis para elaboração de molhos e manjares

(DANALACHE *et al*., 2015; NODA *et al*., 2008; LAU; TANG; PAULSON, 2000; TANG *et al*., 1996).

3.2. Acidulantes

Os ácidos orgânicos são substâncias naturalmente presentes em vegetais e podem ser utilizados como aditivos acidulantes e reguladores de acidez. Acidulante é a substância capaz de aumentar a acidez de um alimento ou conferir a ele gosto ácido. E regulador de acidez é a substância que altera ou controla a acidez ou alcalinidade dos alimentos (BRASIL, 1997).

Os ácidos utilizados em alimentos podem ser encontrados *in natura*, produzidos a partir da fermentação ou por síntese. No primeiro caso, por exemplo, encontram-se os ácidos cítrico e tartárico; por fermentação são obtidos os ácidos cítrico, láctico e acético. Por meio de síntese são fabricados os ácidos málico, acético e fosfórico. Os ácidos orgânicos mais comuns são os ácidos carboxílicos cuja acidez está associada ao grupo carboxila (COOH), sendo geralmente considerados ácidos fracos (THERON; LUES, 2011). Contudo, os acidulantes, apresentam perfis gustativos muito distintos.

Pesquisas revelam que os ácidos orgânicos cítrico, málico e tartárico são promotores da biodisponibilidade de ferro e redutores de pH. Essa redução contribui para a conservação dos alimentos, diminuindo a resistência térmica dos microrganismos ou inibindo a proliferação dos mesmos. Além disso, os ácidos orgânicos melhoram a palatabilidade dos alimentos (DAMODARAN; PARKIN; FENNEMA, 2010; GUPTA; SHIMRAY; RAO, 2012; LAKSHMI; GUPTA; PRAKASH, 2006).

Os ácidos orgânicos comumente usados na indústria de alimentos são: acético, lático, cítrico, málico, fumárico, succínico e tartárico (LINDSAY, 1996). Entre outros produtos em que os acidulantes são empregados, pode-se destacar: geleias artificiais, sorvetes, néctares, pós para coberturas e produtos de confeitaria. Várias pesquisas têm sido desenvolvidas utilizando ácidos orgânicos em diferentes matrizes alimentares. Dentre as quais, podemos citar os doces (MENEZES *et al*., 2012); geleias (FERREIRA *et al*., 2004); produtos de panificação (GUPTA; SHIMRAY; RAO, 2012); sucos (HUMAYUN *et al*., 2014); vinhos (NIU *et al*., 2012).

3.2.1. Ácido Cítrico

O ácido cítrico ($C_6H_8O_7$) está amplamente presente na natureza, sendo derivado dos frutos cítricos. Porém, devido a grande demanda passou a ser extraído por *Aspergillus niger* através da fermentação de soluções de carboidratos de baixo peso molecular. O ácido cítrico também pode ser produzido por síntese química, embora o custo seja maior do que com a fermentação. O ácido cítrico apresenta gosto ácido de curta duração e valores de pKa (3,09; 4,74 e 5,41) (BELITZ; GROSCH; SCHIEBERLE, 2009; THERON; LUES, 2010).

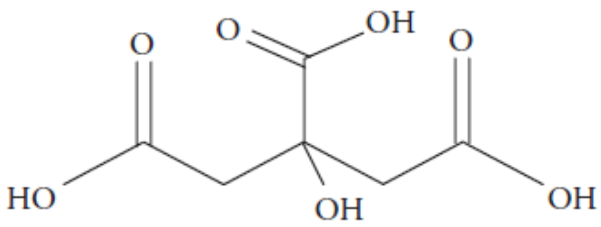

Figura 4 - Estrutura do Ácido Cítrico.
Fonte: (THERON; LUES, 2010).

O ácido cítrico é considerado um ácido orgânico fraco bastante utilizado como acidulante, tamponante e sequestrante, que tem por característica alta solubilidade e ação sequestrante de íons. Como acidulante reduz os valores de pH prevenindo a atividade microbiana. A sua utilização em alimentos confere a sensação ácida dos frutos e intensifica o sabor. Em combinação com seus sais tem ação tamponante, estabilizando o pH durante o processamento de alimentos e no produto final (ARAÚJO, 2004).

Devido à propriedade acidulante, palatabilidade, facilidade de assimilação pelo organismo humano, e baixa toxicidade, o ácido cítrico é um dos mais utilizados como acidulante e antioxidante na indústria de alimentos (GITIRANA, 2007). Cerca de 70 % da produção deste ácido é utilizada pela indústria de alimentos, 12 % pela indústria farmacêutica e 18 % por outras indústrias (THERON; LUES, 2010).

O ácido cítrico apresenta rápida percepção sensorial para o gosto ácido. Humayun *et al*. (2014) avaliaram o efeito do ácido cítrico e málico nas características sensoriais de suco de laranja e observaram que o ácido cítrico apresentou percepção sensorial imediata para o gosto ácido, enquanto que o ácido málico foi responsável pelo gosto ácido mais suave e

duradouro. Os resultados também indicaram sinergismo organoléptico entre os ácidos orgânicos estudados.

Na indústria alimentícia, o ácido cítrico é utilizado como aditivo na fabricação de refrigerantes, bebidas, néctares, vinhos, sobremesas, doces, compotas e geleias, dentre outros. Esse ácido orgânico consegue prevenir a turbidez, auxiliar na retenção da carbonatação, potencializar os conservantes, intensificar o sabor "frutal", prolongar a estabilidade da vitamina C e reduzir alterações na cor (ZHU *et al*., 2014).

3.2.2. Ácido Málico

O ácido málico (D-,L-) ($C_4H_6O_5$) é o principal ácido presente na maçã, sua síntese é obtida pelo processo de hidratação dos ácidos fumárico e maleico. Além da maçã, outros frutos como o marmelo, a melancia, o caqui e a ameixa também possuem ácido málico, porém, em menores proporções. Na indústria alimentícia, o ácido málico é importante devido sua propriedade acidulante, ou seja, é capaz de neutralizar o gosto doce de alimentos e bebidas, tornando-o mais ácido. Esse ácido orgânico também tem a função de realçar o sabor dos alimentos, assim como mascarar os sabores indesejáveis. O ácido málico apresenta valores de pKa (3,40 e 5,05) (BELITZ; GROSCH; SCHIEBERLE, 2009).

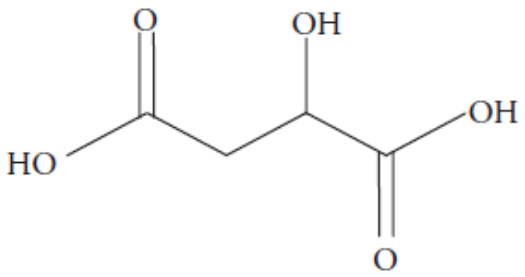

Figura 5 - Estrutura do Ácido Málico.
Fonte: (THERON; LUES, 2010).

O ácido málico tem um sabor suave e persistente. Apresenta adstringência, porém, ela não é tão intensa quanto à do ácido cítrico, contudo, é mais duradoura. O ácido málico tem sido utilizado para mascarar o sabor residual amargo de adoçantes sintéticos, tornando-os mais aceitáveis em bebidas. Ainda que utilizados adoçantes como o aspartame, verificou-se que o crescimento mais lento para o pico de adstringência não sobrepôs os adoçantes e, assim,

menos adoçante torna-se necessário. No entanto, em alguns produtos, como bebidas cítricas, a intensidade inicial associada ao ácido cítrico é preferível (THERON; LUES, 2010).

Em comparação com o ácido cítrico, o málico tem um maior potencial realçador de sabor nos alimentos, portanto, tem sido utilizado como acidulante em marmeladas, bebidas, sobremesas, geleias, conservas e produtos à base de frutas, objetivando a redução de custo (BELITZ; GROSCH; SCHIEBERLE, 2009; THERON; LUES, 2010).

3.2.3. Ácido Tartárico

O ácido tartárico ($C_4H_6O_6$) é um ácido orgânico obtido a partir da fermentação do vinho e bagaço, que contém uma mistura de tartarato hidrogenado de potássio e tartarato de cálcio. Esta mistura é convertida em tartarato de cálcio, a partir do qual o ácido tartárico é liberado usando ácido sulfúrico. O ácido tartárico racêmico é obtido por cis-epoxidação do ácido maleico, seguido de hidrólise. Principalmente devido aos seus baixos valores de pKa (2,98 e 4,34), o ácido tartárico é utilizado com frequência como acidulante. O perfil de adstringência do ácido tartárico é mais suave do que o ácido cítrico e tem um pico de acidez mais alto do que o cítrico e é também mais duradouro. O ácido tartárico está presente em frutos como uvas, abacaxi, tamarindo e nos subprodutos da fermentação do vinho (BURDOCK, 1997; BELITZ; GROSCH; SCHIEBERLE, 2009; THERON; LUES, 2010).

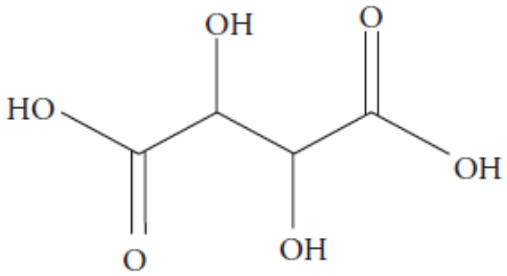

Figura 6 - Estrutura do Ácido Tartárico.
Fonte: (THERON; LUES, 2010).

Os acidulantes reduzem o pH do meio, favorecendo a conservação dos alimentos, conferem estabilidade por prevenir reações oxidativas e têm o potencial de intensificar o sabor dos alimentos. O ácido tartárico apresenta potencial de aplicação nas indústrias de sucos, refrigerantes, geleias, caramelos, sorvetes, doces e vinhos, atuando também como

intensificador de sabor de frutas em alimentos processados (BURDOCK, 1997; RODRIGUES, 2006; BELITZ; GROSCH; SCHIEBERLE, 2009). O ácido cítrico e o tartárico tem sido utilizado como acidulante na fabricação de bebidas à base de frutas, sendo os principais ácidos orgânicos realçadores de sabor nesses produtos.

No estudo sobre o efeito da goma xantana e do ácido tartárico nas características físicas, químicas e sensoriais de cobertura de framboesa, Pereira (2009) verificou que o ácido tartárico exerceu maior redução nos valores de pH e influenciou positivamente os atributos sensoriais, mantendo o equilíbrio ácido/doce e a cor da cobertura, quando comparado ao ácido cítrico. Assim, a interação entre espessante e ácido proporciona uma estrutura com estabilidade física em função da viscosidade, e química devido à redução da atividade de água e do pH, além de preservar suas características sensoriais, fator relevante à aceitação do consumidor (RODRIGUES, 2006).

3.3. Análise Sensorial

A análise sensorial é definida como um método científico utilizado para evocar, medir, analisar e interpretar reações para características de alimentos e materiais que possam ser percebidas pelos sentidos da visão, olfato, tato, paladar e audição (STONE; BLEIBAUM; THOMAS, 2012), através de elementos da psicofísica (MEILGAARD; CIVILLE; CARR, 2016). A análise sensorial é realizada em função das respostas transmitidas pelos indivíduos às várias sensações que se originam de reações fisiológicas e são resultantes de estímulos, gerando a interpretação das propriedades intrínsecas dos produtos.

A percepção do gosto é produzida por conjuntos de quimiorreceptores na boca. Neurônios receptores especializados, agrupados em cavidades dentro das papilas gustativas, geram as percepções do gosto, especialmente ácido, doce, salgado e amargo (NIU *et al*., 2012; SÁENZ-NAVAJAS *et al*., 2010). A partir do estudo da fisiologia e anatomia dos sistemas, sabemos que cada modalidade dos sentidos tem receptores únicos e caminhos neurais para estruturas maiores e mais complexas no cérebro (STONE; BLEIBAUM; THOMAS, 2012).

Os métodos sensoriais podem ser usados para fornecer informações importantes para a indústria de alimentos, como por exemplo, verificar as preferências dos consumidores e aceitabilidade de um produto, controle de qualidade, armazenamento, processamento, desenvolvimento, reformulações de produtos, verificar se os alimentos diferem em sabor, odor, suculência, textura, entre outros atributos (VACLAVIK; CHRISTIAN, 2014; ROSS, 2009). São três os principais tipos de testes sensoriais comumente usados, cada um com um

objetivo diferente. Os testes discriminativos são considerados testes sensoriais simples, que avaliam se existem diferenças perceptíveis entre dois ou mais tipos de produtos; os testes descritivos determinam o perfil sensorial de um produto e identificam os principais atributos sensoriais, pois descrevem as características sensoriais específicas das amostras e suas intensidades; e os testes afetivos avaliam a aceitação ou preferência de um produto em relação a outro (VACLAVIK; CHRISTIAN, 2014; STONE; BLEIBAUM; THOMAS, 2012; LAWLESS; HEYMANN, 2010).

3.3.1. Análise Tempo-Intensidade

O método tempo-intensidade é considerado útil, pois difere da análise descritiva convencional, possibilitando a verificação de mudanças na percepção de um atributo ao longo do tempo (PALAZZO *et al.*, 2011). A análise tempo-intensidade tem como objetivo medir a velocidade, a duração e a intensidade percebida por um único estímulo (AMERINE; PANGBORN; ROESSLER, 1965). Sua relevância está relacionada à percepção do sabor e textura dos alimentos como um fenômeno dinâmico e não estático, portanto, a percepção da intensidade dos atributos sensoriais é modificada ao longo do tempo. Assim sendo, o gosto, o aroma, a textura, o sabor e as sensações térmicas mostram mudanças dinâmicas em intensidade durante todo o tempo de contato com a mucosa oral, com um aumento da percepção até atingir uma intensidade máxima e um declínio até a extinção (LAWLESS; HEYMANN, 2010).

Ao realizar uma análise de tempo-intensidade, podem-se obter informações detalhadas através dos parâmetros das curvas, como o tempo de intensidade máxima, intensidade máxima percebida, tempo de duração da intensidade máxima, a taxa de crescimento, área sob a curva, taxa de decrescimento, tempo onde a intensidade máxima começa a declinar, tempo total de duração do estímulo (LAWLESS; HEYMANN, 2010). Segundo Cardello e Faria (1999), através da associação da percepção humana com os recursos da informática, informações podem ser obtidas sobre características sensoriais pré-estabelecidas das amostras avaliadas.

Vários estudos foram realizados utilizando a análise tempo-intensidade para diferentes tipos de alimentos e bebidas (AZEVEDO *et al*., 2017; PALAZZO; BOLINI, 2014; MORAIS *et al*., 2014; SOUZA *et al*., 2013; MORAIS; CRUZ; BOLINI, 2013). Segundo Cardello, Silva e Damásio (2003), o software desenvolvido para a análise tempo-intensidade

(Time-Intensity Analysis of Flavors and Tastes – TIAFT) - (UNICAMP, 2012) corresponde às expectativas na coleta de dados e na obtenção dos parâmetros das curvas.

Na análise tempo-intensidade ocorre à seleção e o treinamento dos assessores (STONE; BLEIBAUM; THOMAS, 2012) de acordo com sua capacidade em discriminar diferenças nas propriedades sensoriais entre as amostras avaliadas. A partir dos resultados, determina-se a representação das médias de cada curva para os parâmetros aos quais foram treinados. Os resultados obtidos também podem ser avaliados através da técnica multivariada de Análise de Componentes Principais (ACP), que objetiva reduzir a dimensionalidade de um grupo de resultados de variáveis inter-relacionadas e manter o máximo possível de variabilidade presente neste grupo de resultados (JOLLIFFE, 2002).

Através do software TIAFT e dos assessores treinados, pode-se avaliar a interação da intensidade do gosto doce, ácido e viscosidade; e, esses resultados podem ser viáveis em pesquisas com alimentos e desenvolvimento de novos produtos. Portanto, a análise tempo-intensidade é um método que fornece a caracterização temporal de atributos importantes, assim como a percepção de semelhanças e diferenças entre as amostras.

3.4. Homogeneização

Durante o processamento de preservação dos alimentos, a segurança e a qualidade dos produtos devem ser consideradas. Com isso, o aumento na demanda por alimentos com qualidade nutricional e sensorial, apresentando características de produtos *in natura*, tem viabilizado a utilização de tecnologias não térmicas na indústria alimentícia. Essas tecnologias apresentam vantagens como o processamento em baixas temperaturas por um curto período de tempo, exercendo assim, mudanças mínimas ou inexistentes nos sabores e nutrientes essenciais (ZHANG *et al*., 2019; BIRMPA; SFIKA; VANTARAKIS, 2013; RAWSON *et al*., 2011).

Pesquisas utilizando tecnologias não térmicas têm sido realizadas com o objetivo de combinar a mesma eficiência na inativação microbiana e enzimática que possuem os processos térmicos convencionais, porém com maior retenção das características sensoriais e nutricionais dos produtos se comparadas aos processos tradicionais (BETORET *et al*., 2015; LEITE, 2013).

As tecnologias tradicionais para o processamento de alimentos, como a pasteurização e a esterilização, são utilizadas para obtenção de produtos com estabilidade e segurança microbiológica. Contudo, os processamentos térmicos causam efeitos indesejáveis

nos alimentos, como a redução de compostos bioativos e vitamínicos, o escurecimento não enzimático e a formação de sabores indesejáveis (CALLIGARIS *et al*., 2012; LIMA *et al*., 2014; PEREIRA; VICENTE, 2010).

Logo, o processamento em homogeneizador de alta pressão é capaz de preservar a qualidade nutricional e as propriedades dos alimentos quando comparados aos tratamentos térmicos tradicionais, pois o produto é processado em curto período de tempo e com temperatura mais baixa. Entende-se que sob a influência da pressão, pequenas moléculas, como os compostos voláteis, pigmentos, aminoácidos e vitaminas não são afetados, devido às suas estruturas relativamente simples. Em contraste, moléculas maiores, como proteínas, enzimas, polissacarídeos e ácidos nucléicos, podem sofrer alterações (BETORET *et al*., 2015; ALEXANDRE; BRANDÃO; SILVA, 2012; MAHALIK; NAMBIAR, 2010; CAMPOS; DOSUALDO; CRISTIANINI, 2003; BALCI; WILBEY, 1999).

Em contrapartida, no processamento em homogeneizador de alta pressão, o fluido é bombeado por intensificadores de pressão sendo forçado a passar por um orifício estreito em uma válvula de alta pressão, onde é submetido a uma rápida aceleração. Como consequência, fenômenos como cavitação, cisalhamento e turbulência são simultaneamente induzidos, levando ao aumento da temperatura cuja magnitude depende da intensidade da pressão. Esses fenômenos causam alterações físicas nos produtos e modificam as características reológicas, por exemplo, a viscosidade. As pressões normalmente utilizadas na indústria são entre 20 e 50 MPa, mas atualmente os homogeneizadores disponíveis atingem maiores pressões (400 MPa) (FLOURY *et al*., 2004; FLOURY; LEGRAND; DESRUMAUX, 2004; PASQUIN, 1999; CALLIGARIS *et al*., 2012).

A principal aplicação industrial utilizando homogeneizador de alta pressão está relacionada à estabilidade de emulsões. Por esta razão, o homogeneizador tem sido utilizado em indústria farmacêutica, cosmética e alimentícia (CALLIGARIS *et al*., 2012). Várias pesquisas em alimentos avaliaram o efeito do processamento em homogeneizador de alta pressão na inativação de microrganismos (MCKAY *et al*., 2011; BELLOCH *et al*., 2012; AUGUSTO; TRIBST; CRISTIANINI, 2011; TRIBST *et al*., 2009; DIELS; MICHIELS, 2006), na inativação enzimática (NAVARRO *et al*., 2014; CARBONELL *et al*., 2013; VELÁZQUEZ-ESTRADA *et al*., 2012), nas alterações microestruturais e em polissacarídeos (LEITE; AUGUSTO; CRISTIANINI, 2015; LEITE; AUGUSTO; CRISTIANINI, 2014; VILLAY *et al*., 2012; FLOURY *et al*., 2002). Outros estudos relataram que esse tipo de processamento preservou as características nutricionais em sucos de frutas, como a retenção

de compostos bioativos (GUAN *et al*., 2016; YU *et al*., 2016; YU *et al*., 2014; VELÁZQUEZ-ESTRADA *et al*., 2013).

Algumas pesquisas (CARBONELL *et al*., 2013; CERDÁN-CALERO; IZQUIERDO; SENTANDREU, 2013) avaliam as alterações e percepções sensoriais em produtos submetidos ao processamento em homogeneizador de alta pressão. No entanto, poucos estudos analisam características sensoriais e reológicas. Assim, o processo de homogeneização apresenta-se como uma alternativa para modificar as propriedades reológicas em sistemas modelos para néctar de fruta, tornando essa pesquisa inovadora por avaliar alterações nos perfis sensoriais e reológicos.

Portanto, a aplicação de tecnologias não térmicas em processamento de produtos de frutas é importante, sendo possível obter produtos com qualidade nutricional e retenção de compostos bioativos, assim como diferentes perfis sensoriais, mantendo o sabor mais próximo ao produto natural.

3.5. Reologia

A reologia é o estudo do comportamento de um material à aplicação de uma tensão ou deformação (TOLEDO, 1991). O comportamento reológico dos fluidos classifica-se como newtoniano e não-newtoniano. Os fluidos newtonianos apresentam uma relação linear entre tensão de cisalhamento e a taxa de deformação, dependendo, portanto, da temperatura e da composição do fluido, enquanto que os fluidos não-newtonianos, se apresentam dependentes ou independentes do tempo, de forma que, os independentes não são afetados pelo histórico anterior de cisalhamento, sendo caracterizados como fluidos pseudoplásticos. Devido à complexidade da composição e estrutura dos alimentos, estes podem apresentar mais de um tipo de comportamento, dependendo de modificações, origem e concentração (NAGY; CHEN; SHAW, 1993; RAO, 2014).

Para descrever o comportamento reológico são utilizados modelos matemáticos que relacionam tensão de cisalhamento e taxa de deformação. Os modelos de Ostwald-de-Waele (Lei da Potência), Herschel-Bulkley e Casson, são os mais aplicados para descrever o comportamento reológico em sucos de frutas (SILVA; GUIMARÃES; GASPARETTO, 2005; HOLDSWORTH, 1993).

Em experimentos reológicos simples, as amostras são submetidas à taxa de cisalhamento, sendo obtida a curva de escoamento (viscosidade aparente como função da taxa de cisalhamento). Em alimentos, as soluções salinas, óleos, emulsões, suspensões e soluções

poliméricas diluídas podem apresentar características de fluidos newtonianos. Nesses casos, a viscosidade aparente é o parâmetro mais indicado para ser caracterizado (RAO, 1999). Sistemas de dispersões mais concentradas, apresentam propriedades de fluidos não-newtonianos, tais como, os fluidos pseudoplásticos ou dilatantes, assim como podem apresentar mudança na viscosidade dependente do tempo (FONSECA, 2014).

Assim, a viscosidade é a resistência de um líquido ao fluir causada por sua fricção interna. A viscosidade dos fluidos newtonianos é dependente da temperatura, enquanto que os fluidos não newtonianos dependem do tempo e/ou da taxa de cisalhamento. A viscosidade aparente é modificada quando diferentes taxas de cisalhamento são aplicadas, portanto, quanto maior o cisalhamento, menor a viscosidade (KATZBAUER, 1998).

A viscosidade é considerada uma medida direta da qualidade do fluido, e através dela pode-se entender importantes mudanças na estrutura do fluido durante o processamento. Testes reológicos utilizando baixas tensões de cisalhamento devem ser realizados para avaliar as características de uma solução ou controle de qualidade de um produto alimentício, assim como, altas tensões de cisalhamento são utilizadas para realizar estudos reológicos das condições do processamento de soluções ou produtos (DIAZ; VENDRUSCOLO; VENDRUSCOLO, 2004; SHAW, 1975).

A viscosidade de uma solução aquosa com espessante indica a sua qualidade e o seu potencial de aplicação industrial. Essa viscosidade tem relação direta com a composição química, estrutura e concentração do polímero sintetizado. Portanto, o aumento da concentração do polímero aumenta a viscosidade da solução. Este comportamento pode ser atribuído às interações intermoleculares e entrelaçamentos intramoleculares, devido às dimensões da macromolécula e o peso molecular (XUEWU *et al*., 1996; RAO, 1999; GARCÍA-OCHOA *et al*., 2000).

Através das medidas reológicas podem-se observar os efeitos na viscosidade das soluções ocasionados pelo cisalhamento nas estruturas, como por exemplo, a orientação das moléculas, o estiramento e a deformação das estruturas, esses efeitos fazem com que a viscosidade diminua dependendo da taxa de cisalhamento aplicada (DIAZ; VENDRUSCOLO; VENDRUSCOLO, 2004).

As soluções de goma xantana apresentam alta viscosidade em baixas concentrações, assim como, alta pseudoplasticidade e estabilidade frente a variações de temperatura, pH e presença de sais (SUTHERLAND, 1983). A característica pseudoplástica da goma xantana é desejável em alguns produtos, pois aumenta as qualidades sensoriais (liberação de sabor, sensação bucal) em alimentos e apresenta boa capacidade de

bombeamento, mistura e fluidez (KATZBAUER, 1998). Do ponto de vista sensorial, os polissacarídeos que possuem comportamento pseudoplástico provocam menor sensação de gomosidade na boca do que aqueles com comportamento newtoniano. A taxa de cisalhamento na deglutição é considerada entre 50 - 200s^{-1} (MORRIS, 1984). A xantana apresenta baixa viscosidade nessa taxa de cisalhamento (50 - 200s^{-1}), assim, realça o sabor e confere menor viscosidade ao produto alimentício (CHALLEN, 1993).

Importante destacar que a correlação entre os dados reológicos e sensoriais é fundamental para otimização do processo industrial, pois, através da interação entre a percepção do consumidor e as análises físicas do produto, pode-se avaliar melhor a qualidade (SILVA, 2011). Desse modo, torna-se importante determinar o comportamento reológico e sensorial dos sistemas modelos para néctar de fruta, visto que foram submetidos ao processo de homogeneização.

Artigo 1- APPLICATION OF TIME-INTENSITY ANALYSIS IN MODEL SYSTEM SUBMITTED TO HOMOGENIZATION

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ABSTRACT

The use of the high pressure homogenizer has been studied in fruit juices, but researches in model system for application in fruit nectar are scarce. Therefore, it is necessary to evaluate the application of these technologies and how the homogenization pressure (P_H) can interfere in the sensorial profile of the samples. To prepare the solutions we used guar gum (0.1%), organic acids (0.3%) and sucrose (10%), which were later homogenized (0 - control, 25 and 50 MPa) at 25 ºC. The rheological behavior and the temporal profile of the samples were evaluated. The model systems presented pseudoplastic behavior and were fitted to the Ostwald-de-Waele model. The consistency index reduced and the flow behavior index increased with processing. Apparent viscosity also decreased due to homogenization. In the time-intensity sensorial analysis, it was observed that the samples differed among the evaluated parameters, demonstrating that the samples with tartaric acid presented higher intensity for the sour taste. However, for sweetness, no change was observed. In the viscosity attribute, the model systems presented similar temporal profiles. Therefore, it was noted that the homogenization process favored a greater temporal profile of sour taste, making sensory perception more lasting in a model system for fruit nectar.

Keywords: homogenization, rheology, temporal analysis, guar gum, organic acids.

1. Introduction

Food additives have a high potential for application in the food industry and play an important role in the development of new products. Thickeners are food additives with thickening and stabilizing functions that provide desired texture to food and high viscosity, and are able, even at low concentrations, to increase the viscosity of solutions, emulsions and suspensions, improving the texture of the products (Hong et al., 2012).

Guar gum is obtained from the endosperm of *Cyamopsis tetragonolobus*, being formed by chain linear mannose (β-1,4) with galactose residues as side chains, in the ratio of one unit of galactose to two units of mannose. Guar gum has as main property the feature of hydrating rapidly in cold water and form viscous solutions with pseudoplastic behavior. It is considered as a low cost product, besides being compatible with other gums, starches, hydrocolloids and gelling agents (Nieto and Akins, 2011).

In the food industry, organic acids are extensively used as acidulant additives and acidity regulators. They influence directly the taste and the quality of the processed foods, improve digestibility and exert a preservative action by lowering the pH (Theron and Lues, 2011; Damodaran et al., 2010).

Emerging non-thermal technologies, such as high pressure homogenization, have been studied as partial or total substitute for food thermal processing, mainly in fruit products (Augusto et al., 2012), whether for the development of new products or to improve existing technological processes. In the use of a high pressure homogenizer, the fluid is forced to flow through a homogenizing valve under high pressure conditions in the order of micro seconds, with consequent increase in speed due to sudden decrease in volume (gap), followed by depressurizing, resulting in high shear stress, turbulence and cavitation (Dumay et al., 2013).

Research in the fields of non-thermal food processing technologies have stood out for their focus on the preservation and modification of the functional properties of food components while maintaining the quality attributes of the food. Processing in high pressure homogenizer has been applied in different fruit juices (Zhou et al., 2017; Guan et al., 2016; Leite et al., 2015; Calligaris et al., 2012), as well as in polysaccharides (Wang et al., 2011; Harte and Venegas, 2010). Thus, this study is of great interest since it evaluates the effect of P_H on the rheological and sensory behavior in guar gum model systems with applicability for fruit nectars.

Through rheological behavior one can determine the functionality of an ingredient in product development, as well as correlate the data obtained by sensory analysis

(Holdsworth, 1993). Time-intensity sensory analysis provides information about the perceived sensations in food over time. Therefore, they describe the mean velocity, duration and intensity perceived for a single stimulus (Amerine et al., 1965). Time-intensity analysis has been used in research on different types of products (Freitas et al., 2015; Palazzo and Bolini, 2014; Palazzo and Bolini, 2009).

Thus, the aim of this study was to evaluate guar gum model systems acidified with organic acids, and with sucrose additives, determining the influence of the P_H in the time profile and in rheological behavior.

2. Materials and Methods

2.1. Material

To elaborate the model systems, we used the guar gum thickener (AtiViva®, Barretos, Sao Paulo, Brazil) at a concentration of 0,1% (w/v) (Brasil, 2013), and the organic acids (0.3%) (w/v) of citric, malic and tartaric acid (Synth®, Diadema, Sao Paulo, Brazil) were used separately in each thickener solution. The model systems were sweetened with refined sucrose sugar (10%) (w/v) (União®, São Paulo, Brazil).

2.2. Preparation of the model systems

To prepare the model systems, the guar gum was mixed to sucrose and then dissolved in deionized water at room temperature, under stirring (Shaker Fisatom, 713D) for 3 hours. Simultaneously with the preparation of the thickeners, the organic acid was added. The procedures were carried out under stirring, with inlet temperature of 24.5 ± 2 °C and a pH of 2.65 ± 0.02. All rheological and sensory analyses were performed in triplicate.

2.3. Homogenization

After preparing the samples, the homogenization process was carried out at 0 (control), 25 and 50 MPa, in a high pressure homogenizer (Panda Plus, GEA Niro Soavi, Italy). The samples were processed at 25 °C, and immediately after stored in plastic bottles (330mL) under refrigeration (7 ± 2 °C) for 21 days. Afterwards, the rheological and sensorial analyzes were conducted at room temperature (24 ± 2 °C). At the end of the process, we

obtained 9 samples, each with a different acid (citric acid, malic acid and tartaric acid) and processed at 0, 25 and 50 MPa.

2.4. Rheological Analysis

The rheological characteristics of the samples were studied and analyzed in a voltage-controlled rheometer (σ) (AR2000ex, TA Instruments), with cone-plate geometry (60 mm diameter, 2°). During the analyses the temperature was kept constant (25 °C) using a Peltier system. The rheological properties were measured at steady state with decreasing shear rate (0.1 s^{-1} to 300 s^{-1}) for 5 min, and 30 points readings for each curve. After obtaining the behavior to the flow, the model system was adjusted using the Ostwald-de-Waele model (Eq. (1)).

$$\sigma = k \cdot \dot{\gamma}^{\,n} \quad (1)$$

The model parameters of Eq. (1) were modeled as a function of the P_H using the CurveExpert Professional Software v.1.6.3, and a 95% significative probability level.

2.5. Sensory Analysis

The sensory tests were conducted at the Laboratory of Sensory Science and Consumer Studies from the Food and Nutrition Department (FEA/UNICAMP). The samples were presented in a monadic way through complete balanced blocks (Macfie et al., 1989), with three repetitions and in individual booths with white light. The guar gum solutions were presented in (disposable) plastic cups encoded with three-digit numbers. The assessors were advised to use water between the samples to clean the palate.

This research project was submitted and approved by the Research Ethics Committee of UNICAMP, CAAE: 52934315.7.0000.5404. The Free and Informed Consent Form containing information about the research was prepared and presented to the assessors.

2.5.1. Time-Intensity Analysis

The individuals were recruited among UNICAMP undergraduate and graduate students and staff by invitation and posters set at the university. The volunteers who showed interest in participating in the project were trained about the importance of the research and the methodology to be applied.

In the time-intensity analysis, it is necessary pre-select, train and subsequently select the individuals. Thus, to evaluate the discriminative power of each volunteer, triangular tests were performed. A control sample and two samples of solutions of thickeners acidified with organic acids, with a 0.1% level significant difference in relation to viscosity, were evaluated by the participants. Posteriorly the Wald's sequential analysis was applied to the data (Amerine et al., 1965). For pre-selection, using Wald's sequential analysis, values of ρ_0 = 0.45, ρ_1 = 0.70, α = 0.05 and β = 0.05 were used.

2.5.2. Training Session

At this stage, the assessors were trained to use the data collection program Time-Intensity Analysis of Flavors and Tastes – TIAFT – (State University of Campinas, UNICAMP, 2012), developed at the Laboratory of Sensory Science and Consumer Studies at the Faculty of Food Engineering (UNICAMP).

Thus, the evaluators used the TIAFT software, and formed a sensory memory with the extremes of the scale for the sweetness, sourness and viscosity attributes. At this stage, it is also necessary that the evaluators should have a good discriminability between the samples, the repeatability of their results and agreement with the team (Damásio and Costell, 1991).

With the parameters (maximum intensity (Imax); time at which the maximum intensity was perceived (Timax); total time of attribute duration (Ttot); and area under the curve (Area)) analyzed through TIAFT, the time-intensity curve is obtained.

For the temporal analysis of the sweetness and sourness attributes we standardized the initial waiting time (5 s); residence time in the mouth (25 s); time after ingestion (10 s) and scale (10).While, for the viscosity attribute were expected initial (5 s); residence time in the mouth (10 s); time after ingestion (40 s) and scale (10).

2.5.3. Selection of assessors for time-intensity analysis

At this stage, a training was performed using the time-intensity method with the pre-selected individuals (16). 11 of them were selected with discriminative power between the samples and repeatability of their results (Damásio and Costell, 1991). Three experimental samples with different concentrations of thickeners were used to select the assessors.

The participants evaluated samples for the sweetness, sourness and viscosity attributes. The samples were presented in monadic form and evaluated with three repetitions. The intensity of each analyzed attribute was recorded as a function of the time used with the mouse, in a eleven points scale with numbers from 0 to 10. In the time-intensity scale, 0 corresponds to none (left side), 5 corresponds to moderate (center) and 10 corresponds to strong/very (right side).

To evaluate the sweetness and sourness attributes, the following steps were used: at the first warning issued by the computer (5 s) one ought to press start, the assessor put the sample in their mouth and indicated the intensity of the sensory attribute on the scale using the mouse. When hearing the second warning tone (25 s), the evaluator swallowed the sample, and a third warning (10 s) indicated the end of the test. To evaluate the viscosity stimulus, when the first warning was issued by the computer (5 s) one ought to press start, the assessor put the sample in their mouth and indicated the intensity of the sensory attribute on the scale using the mouse. When hearing the second warning tone (10 s), the evaluator swallowed the sample, and a third warning (40 s) indicated the end of the test.

For the selection of the assessors, a two factor (sample and repetition) analysis of variance (ANOVA) was performed with respect to each parameter of the curve obtained. The 11 selected assessors, showed ability to discriminate the samples ($pF_{sample} < 0.30$), repeatability of the results ($pF_{repeat} > 0.05$) and consensus with the team (Damasio and Costell, 1991).

2.5.4. Evaluation of the attributes by time-intensity analysis

The temporal analyses of the guar gum model systems for the sweetness, sourness and viscosity attributes were carried out by the assessors in monadic form and with three repetitions.

Sweetness was defined as the one characteristic of a sucrose solution and sourness as the one characteristic of a citric acid solution. Viscosity was defined as the internal resistance that the particles of a substance present when slipping on top of each other.

Thus, the assessors recorded their temporal perception of the attributes analyzed for the parameters for which they were trained (Imax, Timax, Area, Ttot). Based on the results, the mean values of each curve were determined and the curves were then overlapped.

2.6. Statistical analysis

The results of the effect of homogenization process and the parameters obtained through the time-intensity curves were evaluated through analysis of variance (ANOVA) and Tukey's mean test ($p \leq 0.05$) using the program *Statistical Analysis System* – SAS 9.4. With the data of the time-intensity analysis we also performed the Principal Component Analysis (PCA).

3. Results and Discussion

3.1. Time-intensity analysis

Table 1 shows the mean values of the guar gum model systems with acidulants for the parameters of the curves of the time-intensity analysis, corresponding to the sourness stimulus. We can observe that the homogenization process did not differ significantly ($p < 0.05$) for the time of maximum intensity and total time of perception of the sourness attribute between the control model systems and the homogenized ones (25 to 50 MPa). The time of perceived maximum intensity presented mean values between 8.47 and 9.32 s. The sample (G50T) showed the highest total time (23.34 s) of sourness perception, as well as the highest maximum intensity value (5.00) and area (68.42). We observed that the guar gum model systems with tartaric acid (GCT, G25T and G50T) showed higher values in all parameters, indicating that tartaric acid was considered by the assessors as being the most intensive for the sourness attribute.

In the evaluation of the parameters maximum intensity and area, the samples acidified with tartaric acid did not differ statistically from each other ($p < 0.05$), whereas GCM and G 25M did not differ from the citric acid or tartaric acid samples. On the other hand, the tartaric acid solutions were statistically different from the citric acid solutions. The homogenized model systems at 50 MPa showed a statistical difference ($p < 0.05$) among them, and the G50T sample showed a higher value of maximum intensity and area.

Shallenberger (1996) report that sour taste is related to the concentration of hydrogen ions. However, other studies claim that the sour taste intensity of organic acid solutions is not simply related to the concentration of hydrogen ion. Thus, in addition to hydrogen ions, the concentration of protonated organic acids executes a role in the

determination of the sour taste intensity of organic acids (Da Conceição Neta et al., 2007; Johanningsmeier et al., 2005).

Table 1 – Mean values of the parameters of time-intensity curves for sourness attribute in model systems of guar gum[1].

Samples	Timax*	Imax	Ttot*	Area
GCC	8.86^{a}	3.57^{d}	20.88^{a}	43.94^{cd}
GCM	8.98^{a}	4.06^{bcd}	21.09^{a}	48.71^{bcd}
GCT	9.31^{a}	4.54^{ab}	22.90^{a}	58.49^{ab}
G25C	8.93^{a}	3.78^{cd}	21.13^{a}	46.37^{cd}
G25M	8.64^{a}	4.08^{bcd}	21.40^{a}	50.28^{bcd}
G25T	9.32^{a}	4.54^{ab}	22.41^{a}	58.47^{ab}
G50C	8.47^{a}	4.27^{bc}	21.47^{a}	51.61^{bc}
G50M	8.95^{a}	3.43^{d}	20.52^{a}	39.21^{d}
G50T	9.27^{a}	5.00^{a}	23.34^{a}	68.42^{a}
MDS[2]	1.19	0.66	2.83	11.28

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]Minimal Significant Difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area - area under the curve. GCC – citric control guar; GCM – malic control guar; GCT – tartaric control guar; G25C – citric 25MPa guar; G25M – malic 25MPa guar; G25T – tartaric 25MPa guar; G50C – citric 50MPa guar; G50M – malic 50MPa guar; G50T – tartaric 50MPa guar. *Time in seconds.

The different organic acids used and the homogenization process did not affect the sweetness perception in guar gum model systems, as they did not show significant difference ($p < 0.05$) in the parameters time of maximum intensity, maximum intensity and total time (data not shown). The model systems needed between 8.65 and 9.71 s to reach the time of maximum intensity and between 3.82 and 4.49 s for maximum intensity, while the total time of perception of sweetness was between 18.86 and 21.34 s. It was also observed that the areas of the samples with citric acid (GCC, G25C and G50C) presented higher values, indicating that sweetness was better perceived by the assessors in these samples. This can occur because citric acid is considered a weaker acid (Theron and Lues, 2011).

In the time-intensity analysis for the viscosity attribute (Table 2), we observed that the GCC sample differed from G50C for the parameter of maximum time of intensity and that the other samples did not differ among themselves ($p < 0.05$). The homogenized sample (G50C) reached the maximum intensity in the shortest time (7.56 s). The maximum intensity of the control samples presented values of 3.12 and 3.31. Whereas, the guar gum model

systems processed in the high pressure homogenizer showed maximum intensity for viscosity, with values between 2.48 and 3.04. Although these values are slightly lower, we noted from Table 2 that the sample (G50T) differed significantly ($p < 0.05$) from the control samples (GCC, GCM, GCT) and G25C. Nevertheless, no significant difference was observed among the other samples ($p < 0.05$).

Table 2 – Mean values of the parameters of time-intensity curves for viscosity attribute in model systems of guar gum[1].

Samples	Timax*	Imax	Ttot*	Area
GCC	9.62^{a}	3.31^{a}	26.22^{a}	62.51^{a}
GCM	8.78^{ab}	3.31^{a}	24.75^{a}	59.19^{ab}
GCT	8.47^{ab}	3.12^{a}	24.87^{a}	55.24^{abc}
G25C	8.77^{ab}	3.04^{a}	24.56^{a}	53.67^{abc}
G25M	8.36^{ab}	2.99^{ab}	24.89^{a}	52.29^{abc}
G25T	8.80^{ab}	2.87^{ab}	24.99^{a}	51.63^{abc}
G50C	7.56^{b}	3.00^{ab}	25.34^{a}	55.37^{abc}
G50M	8.05^{ab}	2.86^{ab}	24.34^{a}	49.86^{bc}
G50T	8.34^{ab}	2.48^{b}	25.43^{a}	44.18^{c}
MDS[2]	1.72	0.56	2.24	11.52

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]Minimal Significant Difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area - area under the curve. GCC – citric control guar; GCM – malic control guar; GCT – tartaric control guar; G25C – citric 25MPa guar; G25M – malic 25MPa guar; G25T – tartaric 25MPa guar; G50C – citric 50MPa guar; G50M – malic 50MPa guar; G50T – tartaric 50MPa guar. *Time in seconds.

The total viscosity time was between 24 and 26 s, but the samples did not differ ($p < 0.05$). Thus, even occurring breakdown of polysaccharides during the process in high pressure homogenizer (Wang et al., 2011; Harte and Venegas, 2010; Lagoueyte and Paquin, 1998), the guar gum model systems exhibited a similar total time of sensory perception of viscosity. In the area parameter, the sample (GCC) presented a higher value (62.51) and differed statistically from the samples (G50M and G50T), which presented lower values.

Through the time-intensity analysis one can verify changes in the sensorial perception of a certain attribute over time. For a more comprehensive view of how these changes occur, this study graphically presented the multiple temporal perceptions of the attributes analyzed. Therefore, multiple time-intensity analysis (MTIA) graphically represents

the temporal profile of the curves of two or more sensory attributes of a given sample (Palazzo and Bolini, 2009).

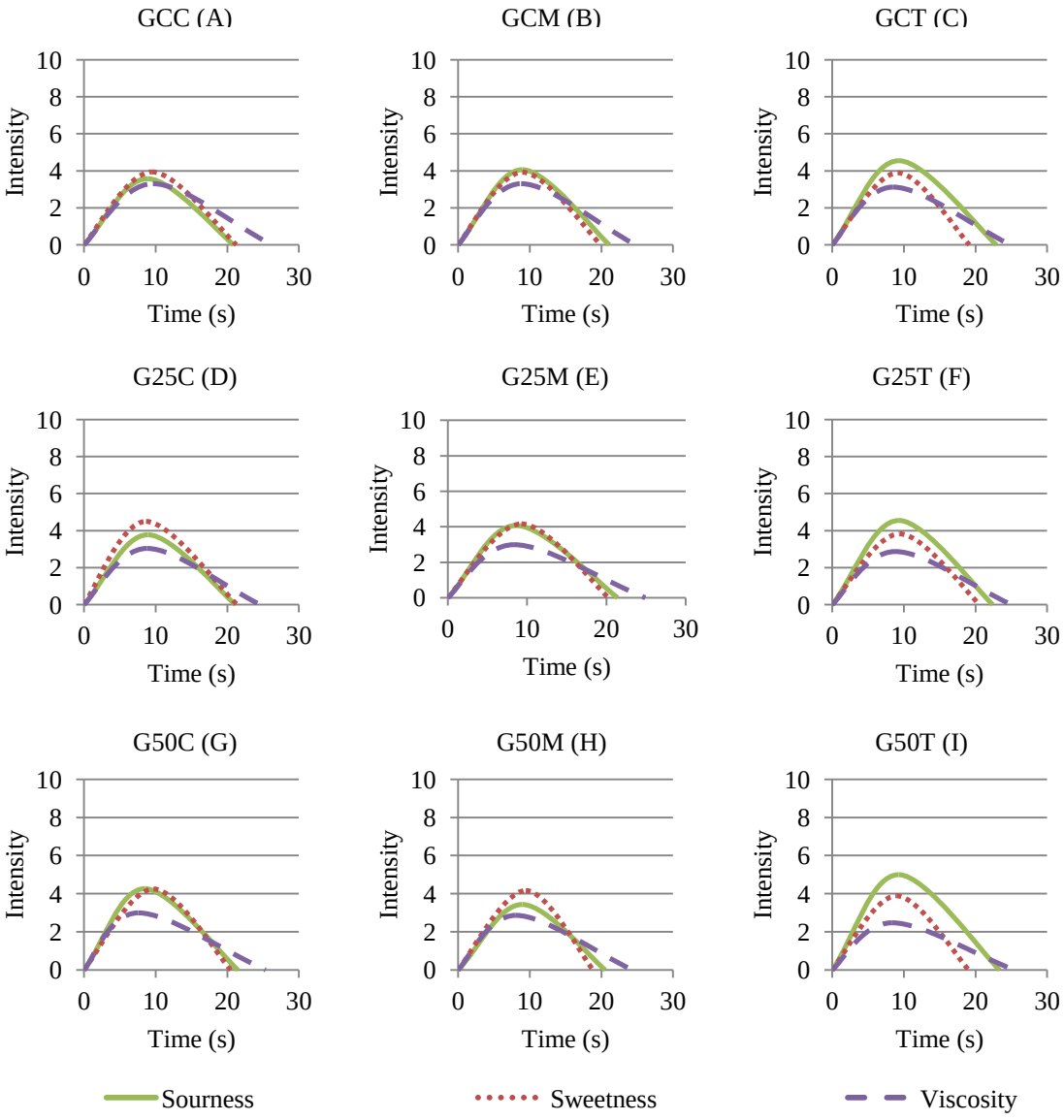

Figure 1 - Multiple time-intensity curves in model systems of guar gum. GCC - citric control guar (A); GCM - malic control guar (B); GCT - tartaric control guar (C); G25C - citric 25MPa guar (D); G25M - malic 25MPa guar (E); G25T - tartaric 25MPa guar (F); G50C - citric 50MPa guar (G); G50M - malic 50MPa guar (H); G50T - tartaric 50MPa guar (I).

Figure 1 represents the curves of the MTIA for the three attributes (sweetness, sourness and viscosity) as a function of time. Through the curves it can be observed that the

guar gum model systems show similar temporal profiles for the sweetness and sourness. The samples with tartaric acid (GCT, G25T and G50T) had an intensity and total perception time of the sourness slightly higher than the other samples. This contributes to meet the expectations of a product with a more accentuated and lasting sourness, and less prolonged sweetness. The guar gum model systems also presented similar sensory profiles for the viscosity stimulus, despite the homogenization process reducing viscosity, this factor did not present significant difference in the temporal profile of the samples. Thus, the use of different organic acids and the processing in high pressure homogenizer (25 and 50 MPa) present a viable alternative for use in fruit nectars, since the evaluated guar gum model systems showed similar temporal profiles. The use of model systems in scientific research has the advantage of having experimental reproducibility and being economical, without significantly changing the products (Augusto et al., 2011; Berto et al., 2003).

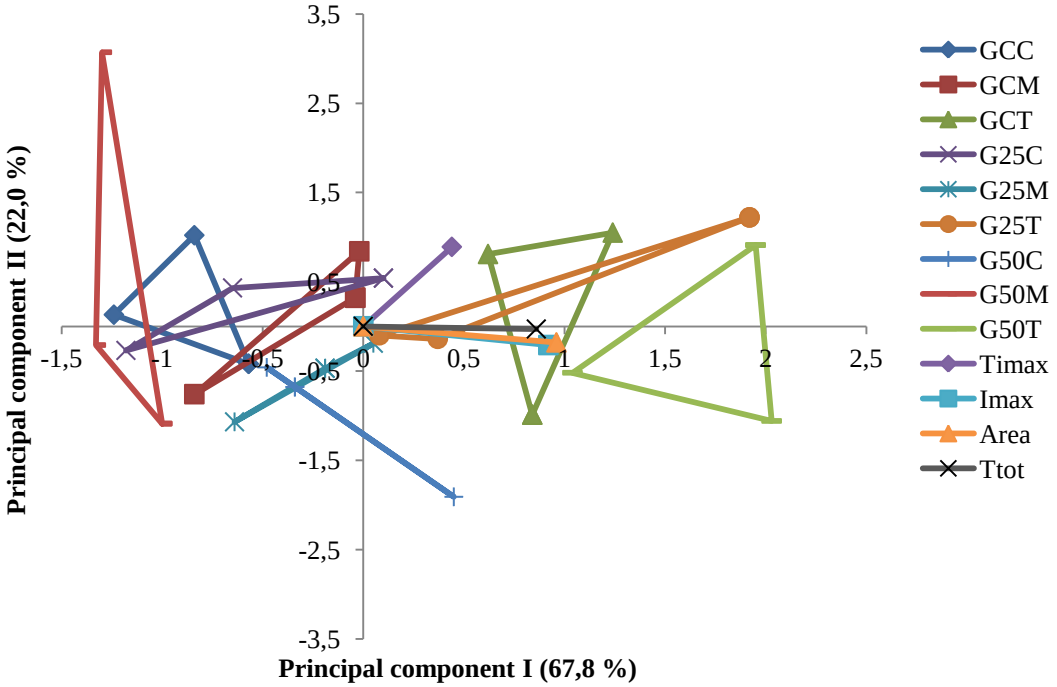

Figure 2 – Principal Component Analysis of sourness in guar gum model systems.

Figure 2 shows the analysis of principal component analysis (PCA) of the guar gum model systems with organic acids for sourness. In this figure, the parameters evaluated in the time-intensity analysis are represented by vectors. Similar model systems occupy close regions in the graph and are characterized by vectors that are close to them. The two main components accounted for 89.8 % of the total variability observed between the samples. It is noted that the vectors (Imax, Ttot and Area) are turned to the samples with tartaric acid (GCT,

G25T and G50T), characterizing them as having the highest intensity for the sourness attribute. However, the other guar gum model systems are all are opposed to the vectors of maximum intensity, total time and area, revealing that they have lower sourness intensity when compared with the samples containing tartaric acid.

PCA of the sweetness attribute presented an explanation of 83.3 % (data not shown), it is noticed that the positioning and the proximity of the samples may indicate that they have similar temporal profiles in guar gum model systems for sweetness stimulus. The G25C sample was positioned far from the others, being characterized by the area parameter.

Time-intensity analysis has been widely used in different food matrices, such as fruit nectar (Freitas et al., 2015), chocolate (Palazzo and Bolini, 2014), fruit jelly (Souza et al., 2013) and white wine (Sokolowsky and Fischer, 2012), mainly to verify sensory perception of sweet, acid and bitter taste, viscosity, and melting in various products.

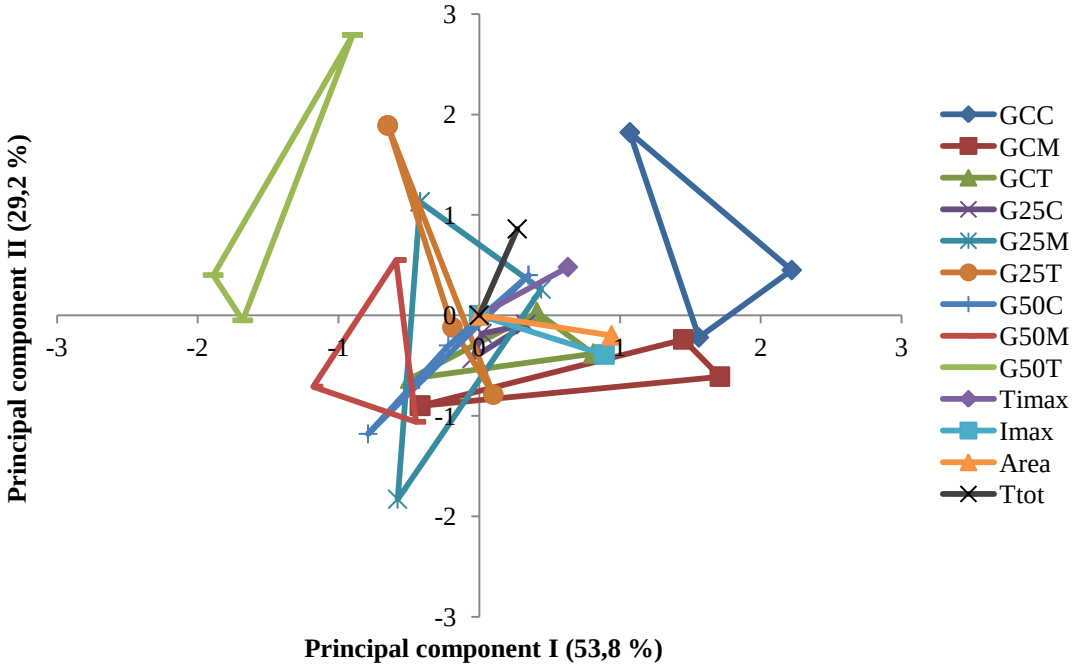

Figure 3 – Principal Component Analysis of viscosity in guar gum model systems.

In Figure 3, we can observe that the two main components were used and jointly explained 83.0 % of the total variability observed among the samples. The principal component analysis allows the visualization of the relations between the parameters and the samples. Thus, each sample can be characterized more intensely by some specific parameters. The control model systems (GCC, GCM and GCT) were characterized by the vectors of maximum intensity and area, indicating higher viscosity in relation to the samples

homogenized at 25 and 50 MPa. Conversely, the GCC sample was characterized by the four analyzed parameters (Timax, Imax, Ttot and Area). The G50T sample is contrasted to the vectors of maximum intensity and area, revealing that it has a lower viscosity.

3.2. Rheological behavior

The homogenization process altered the flow behavior of the guar gum model systems with organic acids. In Figure 4, it can be seen that the homogenized samples at 25 and 50 MPa exhibited a decrease of the behavior to the flow, as we observed that at a fixed shear rate the shear stress values are lower in the homogenized samples. The effects of the P_H and the rheological behavior were similar between the different organic acids used.

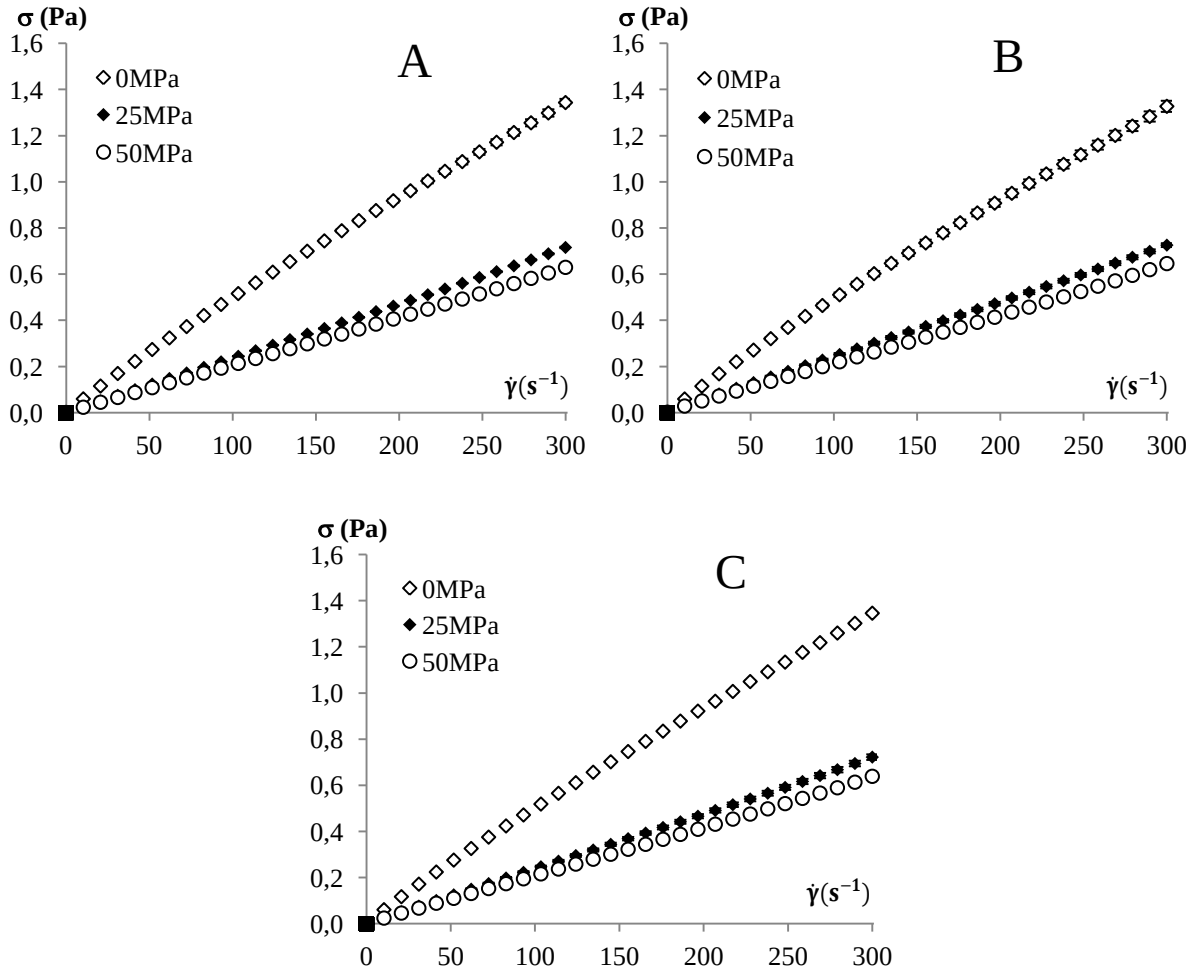

Figure 4 – Flow behavior at 25 °C in model systems of guar gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

Similarly, in Figure 5, it can be observed that the apparent viscosity reduced with the homogenization process, as the non-homogenized model systems presented higher values, independently of the shear rate. The apparent viscosity of the guar gum model systems presented a reduction of approximately 1.5 to 2.0 % of their initial value when the samples were processed. Research shows that the current standard of oral shear rate has been reported as $50s^{-1}$ (Ong et al., 2018; Ferry et al., 2006). Changes in viscosity of non-Newtonian control and homogenized model systems were observed to have an apparent viscosity of approximately 0.006 and 0.002 Pa.s, respectively, when measured at the shear rate of $50s^{-1}$. Relating these data to those of viscosity through time analysis, we observed that, in Table 2, the maximum intensity values were slightly higher for the control model systems (GCC, GCM and GCT), characterizing them with greater intensity for the viscosity attribute. It was also noticed that the homogenized samples presented values of maximum intensity close to those of the control samples. Thereby, the guar gum model systems showed no difference ($p < 0.05$) in the maximum intensity parameter, except for G50T which differed significantly ($p < 0.05$) from the control samples (GCC, GCM and GCT) and from G25C. This can be considered favorable since there was no significant difference between the control model systems and the homogenized ones.

According to Augusto et al. (2012), the rheological properties of serum are slightly affected by processing in high pressure homogenizer. They evaluated a serum model for application in tomato juice and observed a reduction in the viscosity of the fluid.

Therefore, we can state that P_H changed the flow behavior, but this was not sensorially perceived by the assessors. Also, the different organic acids used did not affect the sensory perception of the samples. In the food area, the correlation between rheological and sensory data is critical to determining the functionality of an ingredient in product development, for applications in projects and evaluations of processes, quality control and shelf-life testing (Tabilo-Munizaga and Barbosa-Canovas, 2005; Holdsworth, 1993).

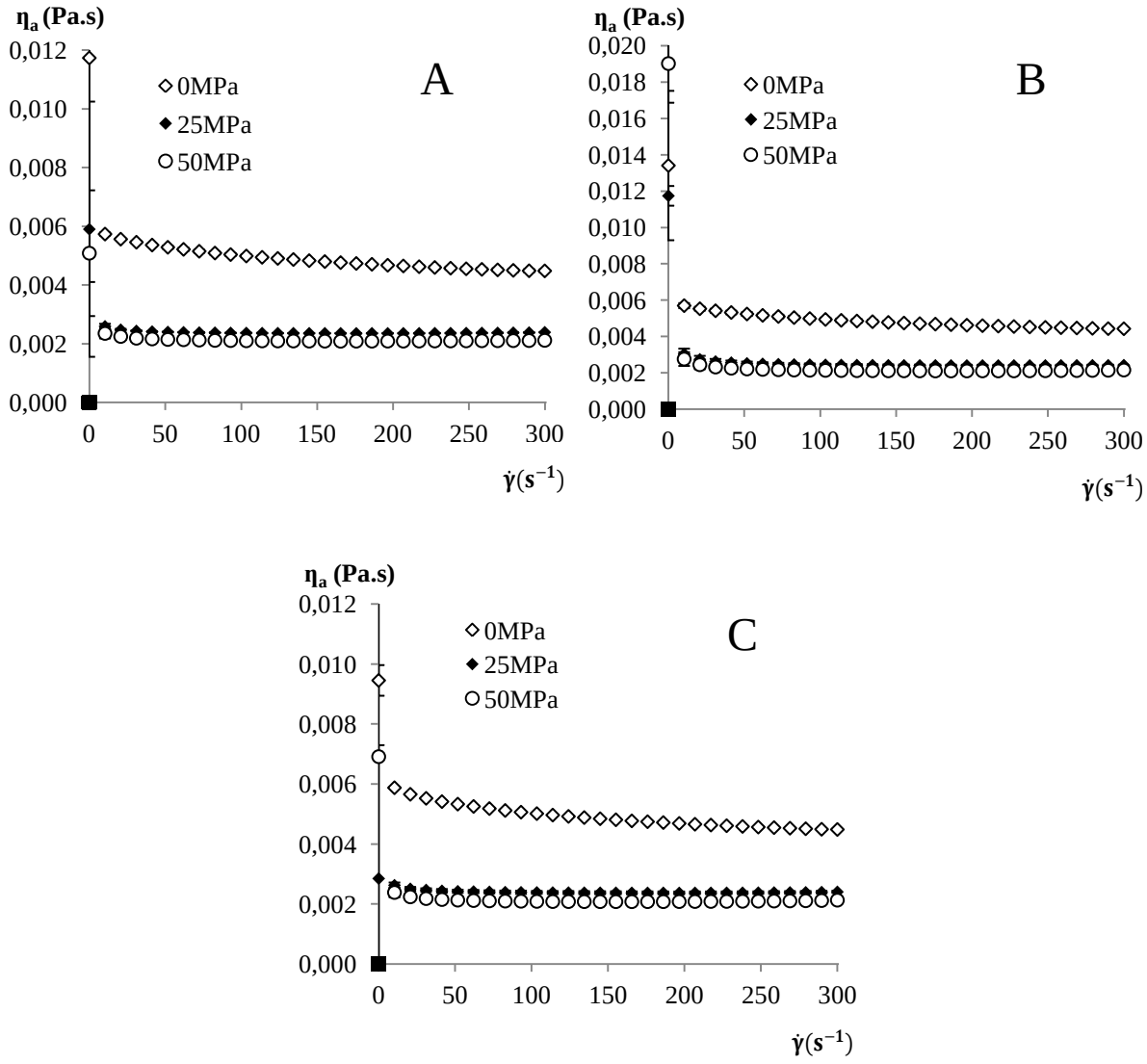

Figure 5 – Apparent viscosity at 25 °C in model systems of guar gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

The Ostwald-de-Waele model (Power Law) was used to describe the flow behavior of guar gum model systems with organic acids. Model systems were characterized as non-Newtonian fluids with pseudoplastic characteristics. In Table 3, the effect of P_H on the parameters of the Ostwald-de-Waele model can be observed for the analyzed model systems. The Ostwald-de-Waele model has been widely used to describe the rheological behavior in juices and nectars (Leite et al., 2014; Faraoni et al., 2013).

Table 3 – Effect of homogenization pressure (P_H) on flow properties in guar gum model systems: Parameters of the Ostwald-de-Waele model at 25 °C.

P_H	Citric acid			Malic acid			Tartaric acid		
	k (Pa.s^n)	*n* (-)	R^2	*k* (Pa.s^n)	*n* (-)	R^2	*k* (Pa.s^n)	*n* (-)	R^2
0MPa	0.008 ± 0.0001	0.900 ± 0.003	0.989	0.007 ± 0.0002	0.899 ± 0.002	0.977	0.008 ± 0.0001	0.897 ± 0.001	0.987
25MPa	0.002 ± 0.0000	1.000 ± 0.000	0.994	0.003 ± 0.0002	0.986 ± 0.015	0.988	0.002 ± 0.0001	0.999 ± 0.002	0.993
50MPa	0.002 ± 0.0001	1.000 ± 0.000	0.992	0.002 ± 0.0004	0.990 ± 0.018	0.990	0.002 ± 0.0001	1.000 ± 0.000	0.989

It can be observed that the consistency index (*k*) had a slight reduction with the increase of pressure, and the flow behavior index (*n*) had a slight increase with the homogenization process. For the consistency index parameter, values ranging from 0.008 to 0.002 Pa.s^n were obtained, and for the flow behavior index, results ranging from 0,897 to 1,000 (Table 3). Values of the flow behavior index (*n*) equal to 1, show that the flow alignment effect was practically null in the processed samples. It was also observed that there was a reduction in the pseudoplastic characteristics of the homogenized model systems, thus, these samples presented a behavior close to the Newtonian. A general trend can be considered, as the different organic acids used did not interfere in the rheological behavior of the flow, since the guar gum model systems showed statistically equal values of the consistency index ($p < 0.05$); similarly, the flow behavior values were similar. Research on different polysaccharides (Floury et al., 2002; Lagoueyte and Paquin, 1998) also showed that with the increase of homogenization pressure there was a reduction in the consistency index (*k*) and an increase in the flow behavior index (*n*).

The processing using high pressure homogenizer has been much studied in different food matrices, among them fruit juices (Zhou et al., 2017; Leite et al., 2014; Calligaris et al., 2012; Tribst et al., 2011), dairy beverages (Martínez-Monteagudo et al., 2017), dietary supplements (Martínez-Sánchez et al., 2016), as well as in different polysaccharides (Villay et al., 2012; Wang et al., 2011; Harte and Venegas, 2010). Hence, the knowledge of rheological behavior is fundamental for the development of new products, not only as a quality measure, but also in the design, evaluation and operation of food processing equipment such as pumps, agitation systems and pipes (Ibarz et al., 1996).

4. Conclusions

The processing of guar gum model systems in a high pressure homogenizer is a viable alternative for application in fruit nectar. Through the sensorial time-intensity analysis and the rheological behavior we can indicate new perspectives on the intensity of tastes and viscosity. Therefore, the viscosity in the control and homogenized samples presented similar temporal profiles, and the tartaric acid model systems showed higher maximum intensity for the acid taste, presenting potential of application for nectars with characteristics of acid taste more intense and prolonged.

Notation

$\dot{\gamma}$ shear rate [s^{-1}]
η_a apparent viscosity (= $\sigma/\dot{\gamma}$) [Pa·s]
σ shear stress [Pa]
k consistency index, Ostwald-de-Waele model (Eq. (1)) [Pa·s^n]
n flow behavior index, Ostwald-de-Waele model (Eq. (1)) [–]

Acknowledgments

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Declaration of conflicting interests

The authors declare that there is no conflict of interest.

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Artigo 2 - EFFECT OF HOMOGENIZATION PRESSURE AND TEMPORAL PROFILE IN MODEL SYSTEM

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ABSTRACT

The effect of homogenization pressure (P_H) was studied in a model system for the application in fruit nectar. Xanthan gum (0.2%), organic acids (0.3%) and sucrose (10%) were used to prepare the solutions, which were then homogenized (0 – control, 25 and 50 MPa) at 25 °C. Rheological analyses of flow behavior and sensorial time-intensity were performed for the sweetness, sourness and viscosity attributes. The model system presented pseudoplastic behavior and was fitted to the Ostwald-de-Waele model. The P_H reduced the consistency index and increased the flow behavior index, as well as reduced apparent viscosity. The temporal analysis differed between the studied parameters, demonstrating a greater temporal profile of the sourness and that the processing of the samples reduced the viscosity perception. Therefore, researches on the effect of P_H on the rheological and sensorial behavior in a model system aids in the development of products and processes.

Keywords: homogenization pressure; rheology; viscosity; time-intensity analysis; xanthan gum; organic acid.

1. Introduction

Food additives represent the set of substances added to the food without the purpose of nourishing, but with the objective to modify or maintain the physical, chemical and biological or sensory properties of these products (Brasil 1997). Thickeners are food additives that function thickening and stabilizing, providing functional technological functions such as texture and viscosity to the food.

Xanthan gum shows high solubility and stability in wide temperature range and pH, even in the presence of salts. It is a thickener with a wide variety of applications in the food industry, due to its rheological characteristics and properties. One of the main properties of xanthan gum is the capacity of forming highly viscous solutions in low concentrations (García-Ochoa et al. 2000). This feature provides a great advantage for its use in food because the concentration required to convey the desired properties is small and does not affect the taste of the final product. The use of xanthan is mainly due to its rheological properties as its pseudoplastic behavior benefits the food industry, since the viscosity of the solutions decreases with the increase of the shear rate, allowing the mixing, pumping and flow capacity in the pipes (Preichardt and Klaic 2016).

Acidulants are substances capable of increasing the acidity of an aliment or conferring an acid taste (Brasil 1997). Organic acids are substances naturally present in plants and can be used as acidulant additives and acidity regulators. They exert a preservative action in foods by lowering the pH, increasing the digestibility and improve the palatability of food (Damodaran et al. 2010). In the food industry, thickeners and organic acids are of significant importance since they are used for the manufacture and development of various products, among them, fruit nectars.

In this study a high pressure homogenizer was used, which consists of a process in which a fluid is pumped and forced to flow through a narrow orifice valve, inducing an increase in speed after depressurization with consequent cavitation and high shear stress (Augusto et al. 2013). These effects promote changes in the rheological and physical properties of the products, such as viscosity change. Thus, the use of the high pressure homogenizer presents an alternative to modify the rheological properties in model system. It has been used as auxiliary technique to assist food preservation, mainly fruit products, and is considered a very promising non-thermic treatment, as it limits thermal damage, avoiding flavor, aroma, color and nutritional losses (Augusto et al. 2012; Calligaris et al. 2012).

The use of the high pressure homogenizer has been studied in several fruit juices (Zhou et al. 2017; Leite et al. 2015; Leite et al. 2014; Velázquez-Estrada et al. 2013; Calligaris et al. 2012) with the objective of reducing viscosity, enzymatic and microbial inactivation; and in different polysaccharides (Augusto et al. 2012; Wang et al. 2011; Harte and Venegas 2010) to evaluate the viscosity and functionality of the ingredients. The present study, in turn, is innovative for evaluating the influence of homogenization on the rheological and sensorial properties in a model system for fruit nectar.

Sensorial analysis has been used as a tool to evaluate the development of products, having a relevant importance in the industrial process. The evaluation of a product through the time-intensity analysis aims to measure speed, duration and intensity perceived by a single stimulus (Amerine et al. 1965). Multiple time-intensity analysis (MTIA) is a graphical representation of the temporal profile of the curves of two or more sensory attributes of a sample (Palazzo and Bolini 2009). Several studies have been carried out using time-intensity analysis for different types of food and beverages (Azevedo et al. 2015; Freitas et al. 2015; Morais et al. 2013; Palazzo and Bolini 2014). Therefore, the study of the rheological properties and the time-intensity profile of a model system for application in fruit nectar can be a useful tool for both research and potential industrial application.

Thus, the objective of this study was to evaluate the effect of the homogenization in the time profile and in the rheological behavior in model system of xanthan gum, acidified with organic acids and with added sucrose.

2. Materials and Methods

2.1. Material

For the preparation of the model system we used the thickener xanthan gum (Keltrol®, CPKelco, Limeira, Sao Paulo, Brazil) in a 0.2 % (w/v) concentration (Brasil 2013), the acidulants citric acid (0.3 %), malic acid (0.3 %) and tartaric acid (0.3 %) (w/v) (Synth®, Diadema, Sao Paulo, Brazil) were used separately for each thickener solution. The solutions were sweetened with refined sucrose sugar (10 %) (w/v) (União®, Sao Paulo, Brazil).

2.2. Preparation model system - fruit nectar

For the preparation of the solutions of thickeners acidified with organic acids, xanthan gum was mixed with sucrose and subsequently dissolved in deionized water at room temperature, under stirring (Fisatom Shaker, 713D) for 3 hours. Simultaneously to the preparation of the thickeners, the organic acid was added. All procedures were performed under agitation with inlet temperature of 24.5 °C and a pH of 2.67. All rheological and sensorial analyses were performed in triplicate.

2.3. Homogenization

The homogenization process was performed at 0 - control, 25 and 50 MPa in a high pressure homogenizer (Panda Plus, GEA Niro Soavi). The samples were processed at 25 °C simulating processing conditions for fruit nectar. After processing, the samples were stored under refrigeration (7 ± 2 °C) in bottles, and the rheological and sensorial analyses conducted at room temperature (24 ± 2 °C). In total, there were 9 samples, each with a different acid (citric, malic and tartaric acid) and processed at 0, 25 and 50 MPa.

2.4. Rheological Analysis

The rheological characteristics of the samples were analyzed in a controlled voltage rheometer (AR2000ex, TA Instruments) with cone-plate geometry (60 mm diameter, 2°). The samples were analyzed at constant temperature (25 °C) through a Peltier system. The rheological analyses were obtained at steady state with decreasing shear rate (0.1-300 s^{-1}) for 5 min. The data obtained in the rheological analysis were adjusted to the Ostwald-de-Waele model (Power law) (Eq. (1)).

$$\sigma = k \cdot \dot{\gamma}^{\,n} \quad (1)$$

The parameters of the model of Eq. (1) were modeled as a pressure function of P_H using the CurveExpert Professional Software v.1.6.3, and a significant probability level of 95%.

2.5. Sensory Analysis

Sensory analysis was performed in the Laboratory for Sensory Science and Consumer Studies in the Food and Nutrition Department (FEA/UNICAMP). The model systems were presented in disposable cups, coded with three-digit numbers. The samples were

presented in monadic form in complete balanced blocks (MacFie et al. 1989), with three repetitions and in individual booths. The assessors were instructed to use water between the samples to clean their palate.

This research project was submitted and approved by the Research Ethics Committee of UNICAMP, CAAE: 52934315.7.0000.5404. The Free and Informed Consent Form containing information about the research was prepared and presented to the assessors.

2.5.1. Time-Intensity Analysis

The individuals were recruited among employees, undergraduate and graduate students of UNICAMP. They were instructed on the importance of the study and methodology to be used. For the time-intensity analysis it is necessary that the individuals should be pre-selected, trained and subsequently selected. Therefore, the triangular test was applied in which the testers had to identify differences between two samples. A control sample and two samples of solutions of thickeners acidified with organic acids, with a significant difference of 0.1% in relation to viscosity, were evaluated by the assessors; subsequently, Wald's sequential analysis was applied (Amerine et al. 1965). Each volunteer performed the test with 12 repetitions. The values used for pre-selection in the Wald sequential analysis were: $\rho_0 = 0.45$, $\rho_1 = 0.70$, $\alpha = 0.05$ and $\beta = 0.05$.

2.5.2. Training Session

The data collection for the time-intensity analysis was performed in computer, through the program Time-Intensity Analysis of Flavors and Tastes, TIAFT, (Universidade Estadual de Campinas, UNICAMP, 2012), developed at the Laboratory of Sensory Science and Consumer Studies at the School of Food Engineering (UNICAMP).

At this stage, the assessors used the TIAFT software, and formed a sensory memory with the extremes of the scale for the analysed attributes. The maximum intensity references for sensory memory were: sweetness (sucrose solution 12%), sourness (citric acid solution 0.4%) and viscosity (xanthan gum solution 0.3%). Thus, the individuals were instructed to use the software, and it was necessary to discriminate the samples and repeat their results in agreement with the team (Damásio and Costell 1991).

Through the TIAFT program, we obtained the time-intensity curve, with the maximum intensity parameters (Imax), the time when the maximum intensity was recorded

(Timax), the total duration time of the assessed stimulus (Ttot) and the area under the time-intensity curve (Area) (Palazzo and Bolini 2014).

The standard conditions for the time-intensity analysis for the viscosity attribute were: initial wait (5 s); time with the sample in the mouth (10 s); time after ingestion (40 s) and scale (10). While, for the temporal analysis of the sweetness and sourness attributes we standardized the initial waiting time (5 s); residence time in the mouth (25 s); time after ingestion (10 s) and scale (10).

2.5.3. Selection of the Assessors for time-intensity analysis

With the preselected individuals (16), a training was carried out, using the time-intensity method, with experimental samples (3 samples with different concentrations of thickeners), to select the assessors capable of presenting discriminative power between the samples and the repeatability of the results in agreement with the team (Damásio and Costell 1991). Eleven advisors were selected to make up the sensory team.

The assessors analyzed the samples for the sweetness, sourness and viscosity attributes. The samples were presented in monadic form, with three repetitions, recording the intensity of the attribute regarding the time passed, with the use of the mouse, in linear scale from 0 to 10. In the time-intensity scale, 0 corresponds to none (left side), 5 corresponds to moderate (middle) and 10 to strong/very (right side).

At the first warning issued by the computer (5 s), start was pressed, the assessor put the sample in the mouth and using the mouse they indicated on the scale the intensity of the viscosity attribute. At the second warning tone (10 s), the participant swallowed the sample and a third warning (40 s) indicated the end of the test. While, to evaluate the sweetness and sourness attributes, at the first warning issued by the computer (5 s), start was pressed, the assessor put the sample in the mouth and using the mouse they indicated on the scale the intensity of the attribute assessed. At the second warning tone (25 s), the participant swallowed the sample and a third warning (10 s) indicated the end of the test.

In order to select the assessors, an analysis of variance (ANOVA) was used for each assessor in relation to each parameter of the curve obtained. The 11 selected advisors were able to discriminate the samples ($p < 0.30$) and reproducibility of the results ($p > 0.05$) (Damásio and Costell 1991).

2.5.4. Evaluation of the attributes through the time-intensity analysis

The selected assessors analyzed of the sweetness, sourness and viscosity attributes of the samples (model systems) which were presented in a monadic fashion, with three repetitions. Viscosity was defined as the internal resistance that the particles of a substance present when slipping on top of each other. Sweetness was defined as the one characteristic of a sucrose solution and sourness as the one characteristic of a citric acid solution. In this way, the assessors recorded the temporal perception of the attributes analyzed for the parameters to which they were trained (Imax, Timax, Area, Ttot). From the results, the representation of the means of each curve was determined.

2.6. Statistical analysis

The data of the effect of homogenization pressure (P_H) and the sensory data were subjected to the analysis of variance (ANOVA) and Tukey's mean test ($p \leq 0.05$). With the data obtained from the time-intensity analysis, the Principal Component Analysis (PCA) was also performed. For statistical analysis of the data, we used the program Statistical Analysis System – SAS 9.4.

3. Results and Discussion

3.1. Time-intensity analysis

The homogenized samples presented alteration in the sensorial perception of sourness. Table 1 shows that the mean values of the time-intensity analysis parameters in the xanthan gum model systems presented a significant difference ($p < 0.05$). The time to reach maximum intensity did not differ among most samples, except between XCC and X25M. The mean values for this parameter were between 12 and 14 s.

It was observed that the xanthan gum model systems homogenized at 25 and 50 MPa showed higher values in the analyzed parameters (Imax, Ttot and Area), indicating that the homogenized samples were considered by the assessors as being the ones with the highest intensity for the sourness attribute. Thus, the perception of sourness was prolonged and this is related to the changes that occurred in the structures of the cells and in the interaction among them.

The XCC, X50C and X50T samples differed statistically ($p < 0.05$) in the total time, presenting values between 26.45 and 30.53 s. In the area parameter, a significant difference was observed between the control model systems and the homogenized ones. The control samples had lower values of areas.

Table 1 – Mean values of the parameters of time-intensity curves for sourness in model systems of xanthan gum[1].

Samples	Timax*	Imax	Ttot*	Area
XCC	12.59^{b}	3.62^{e}	26.45^{b}	51.38^{e}
XCM	13.28^{ab}	3.92^{de}	27.37^{ab}	60.53^{de}
XCT	13.77^{ab}	4.29^{cde}	28.22^{ab}	66.71^{cde}
X25C	13.55^{ab}	4.67^{cd}	28.82^{ab}	76.62^{bcd}
X25M	14.26^{a}	5.04^{bc}	29.83^{ab}	81.05^{abc}
X25T	13.72^{ab}	5.79^{ab}	29.53^{ab}	90.88^{ab}
X50C	13.46^{ab}	5.53^{ab}	30.24^{a}	89.54^{ab}
X50M	13.00^{ab}	6.05^{a}	29.06^{ab}	91.97^{ab}
X50T	12.90^{ab}	5.93^{a}	30.53^{a}	97.04^{a}
MDS[2]	1.63	0.83	3.61	19.96

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]MDS – Minimum significant difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area – area under the curve. XCC – citric control xanthan; XCM – malic control xanthan; XCT – tartaric control xanthan; X25C – citric 25MPa xanthan; X25M – malic 25MPa xanthan; X25T – tartaric 25MPa xanthan; X50C – citric 50MPa xanthan; X50M – malic 50MPa xanthan; X50T – tartaric 50MPa xanthan. *Time in seconds.

The time-intensity analysis was also performed for the sweet taste attribute, however, it was observed that the different organic acids and the processing used did not interfere in the sweetness perception of the samples, since they did not present a significant difference ($p < 0.05$) (data not shown) in any of the evaluated parameters. The xanthan gum model systems showed maximum intensity for sweet taste between 4.81 and 5.31, and total time between 23.33 and 24.82 s.

Through time-intensity analysis we obtained the values of the parameters analyzed in model systems of xanthan gum (Table 2), in which it is shown that the time to reach the maximum perceived intensity for the viscosity attribute differed significantly ($p <$

0.05) between sample X25T, and samples XCM, XCT, X25C, X25M. The time of maximum perceived intensity presented mean values between 8.35 and 10.90 s.

Table 2 – Mean values of the parameters of time-intensity curves for viscosity in model systems of xanthan gum[1].

Samples	Timax*	Imax	Ttot*	Area
XCC	9.45^{ab}	7.39^{a}	29.00^{a}	154.95^{a}
XCM	10.90^{a}	7.18^{ab}	28.13^{a}	141.82^{ab}
XCT	10.57^{a}	6.59^{b}	28.57^{a}	130.85^{b}
X25C	10.52^{a}	3.36^{cd}	25.28^{b}	59.88^{cd}
X25M	10.88^{a}	3.95^{c}	25.09^{bc}	68.76^{c}
X25T	8.35^{b}	3.33^{cd}	24.76^{bc}	55.67^{cd}
X50C	9.10^{ab}	3.14^{d}	23.92^{bc}	50.30^{d}
X50M	9.25^{ab}	2.88^{d}	23.37^{c}	47.69^{d}
X50T	9.47^{ab}	2.93^{d}	23.67^{bc}	46.57^{d}
MDS[2]	1.92	0.65	1.80	14.71

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]MDS – Minimum significant difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area – area under the curve. XCC – citric control xanthan; XCM – malic control xanthan; XCT – tartaric control xanthan; X25C – citric 25MPa xanthan; X25M – malic 25MPa xanthan; X25T – tartaric 25MPa xanthan; X50C – citric 50MPa xanthan; X50M – malic 50MPa xanthan; X50T – tartaric 50MPa xanthan. *Time in seconds.

Greater peak intensity was observed in the control samples, that is, the P_H reduced the viscosity of the solutions (Table 2). In the maximum intensity attribute, the control samples differed significantly ($p < 0.05$) from samples homogenized at 25 and 50 MPa, these showed a decrease of approximately 50% in the values for the analyzed attribute. In the samples subjected to the homogenization process at 25 MPa, we observed maximum intensity values between 3.33 and 3.95, for samples with tartaric acid and malic acid, respectively. And in those processed at 50 MPa, we found values of 2.88 to 3.14 for samples with malic and citric acid, respectively. Nonetheless, they showed no significant difference ($p < 0.05$) among them.

The control samples had a longer viscosity perception time, making the viscous sensation more prolonged. We observed that the total time of viscosity perception was higher in the control samples, where it ranged from 28.13 to 29.00 s, for the malic and citric acid

samples respectively (Table 2). In the case of the samples at 25 and 50 MPa, we observed that the total time was approximately 25 and 23 s, respectively. This is due to the effect of the P_H, because during the process the rupture of polysaccharides occurs, the molecules are broken into smaller particles (Wang et al. 2011; Harte and Venegas 2010; Lagoueyte and Paquin 1998), thus making the samples less viscous, yielding, consequently, a lower total time of perception of the viscosity attribute. Thus, it can be observed that the control samples differed significantly ($p < 0.05$) from the homogenized samples. Consequently, the control samples obtained a larger area, since they presented higher maximum intensity and total time (Table 2). Therefore, we remarked that the P_H reduced the viscosity of the model systems of xanthan gum acidified with organic acids. We also observed that the different organic acids used had no significantly effect on the parameters of maximum intensity, total time and area of the model systems for the viscosity stimulus.

The mean parameters of each sample were used to construct the MTIA curves for the attributes (sourness, sweetness and viscosity) as a function of time (Figure 1). Through the curves it can be observed that the xanthan gum model systems submitted to homogenization presented higher maximum intensity and total time of perception for the sourness attribute. The samples with tartaric acid (XCT, X25T and X50T) presented a greater area among their respective treatments. It is also noted, for the same attribute, that the homogenized samples (25 and 50 MPa) presented similar temporal profiles. This favors to meet the expectations of a product with more acidic characteristics, and perception of sourness for a longer period. All xanthan gum model systems presented similar temporal profiles for the sweet taste stimulus, that is, the homogenization process did not affect the sensorial profile of the samples in the respective attribute.

In Figure 1, it can be seen that the control samples showed higher maximum intensity and total time in the perception of viscosity, characterizing these samples as being more viscous. Among the control samples, we also observed that the XCC sample had the highest maximum intensity and area, this made it differ significantly ($p < 0.05$) from the XCT sample, as can be observed in Table 2. Conversely, among the samples homogenized at 25 MPa, the tartaric acid solutions had lower values for all analyzed parameters (Timax, Imax, Ttot and Area). The solutions of xanthan with citric acid obtained greater total time of perception of the viscosity between the control solutions, 25 and 50 MPa. Whereas, those of tartaric acid (XCT, X25T and X50T) presented smaller area between their respective treatments. The control model systems (0MPa) presented similar behavior and those subjected

to homogenization (25 and 50 MPa) also showed similarities and small variations among each other for the viscosity stimulus.

The processing of xanthan gum model systems (25 and 50 MPa) in high pressure homogenizer is a possibility for application in fruit nectars with a characteristic acid taste and a lower viscosity.

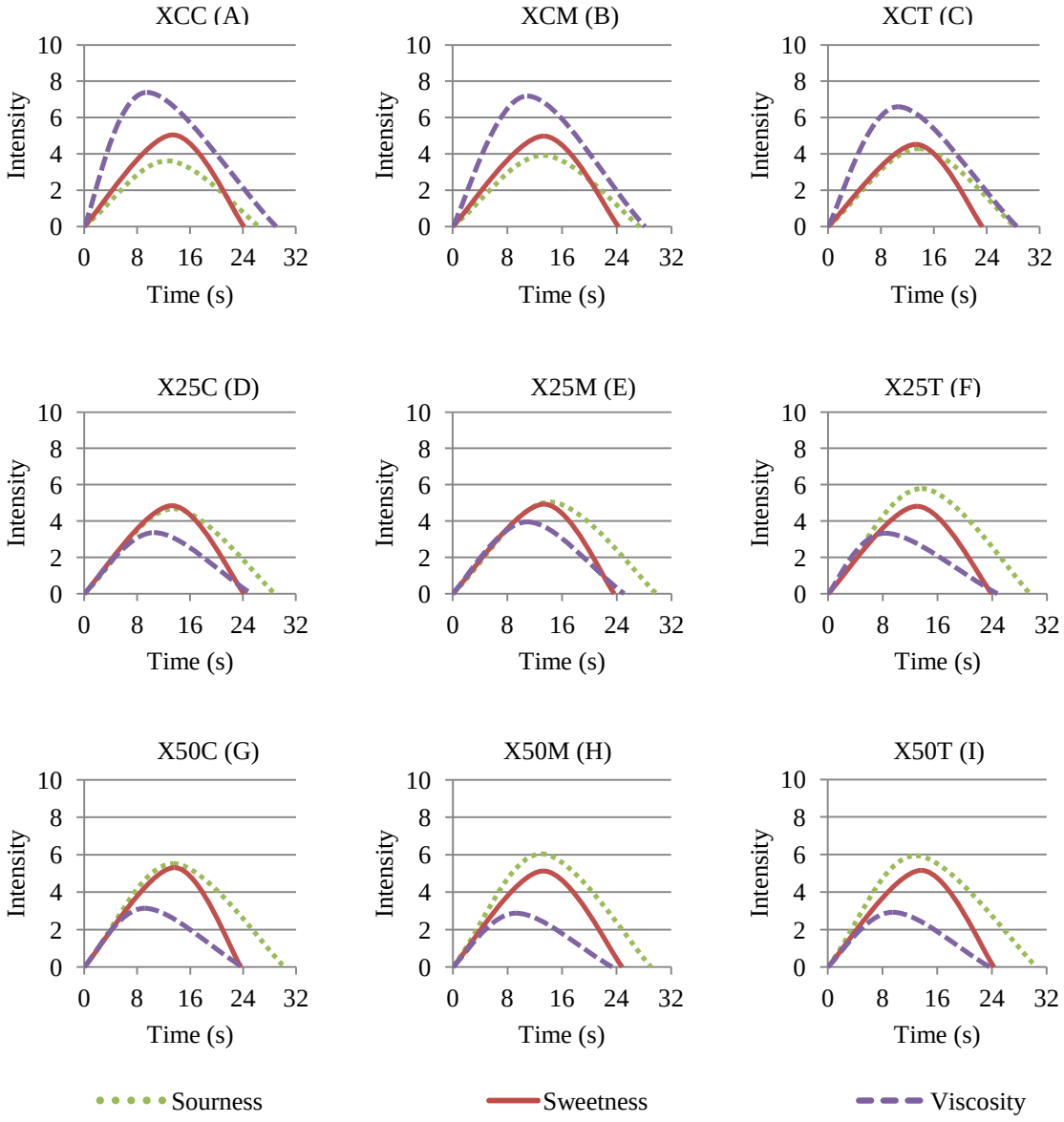

Figure 1 – Multiple time-intensity curves in model systems of xanthan gum with acidulants. XCC – citric control xanthan (A); XCM – malic control xanthan (B); XCT – tartaric control xanthan (C); X25C – citric 25MPa xanthan (D); X25M – malic 25MPa xanthan (E); X25T – tartaric 25MPa xanthan (F); X50C – citric 50MPa xanthan (G); X50M – malic 50MPa xanthan (H); X50T – tartaric 50MPa xanthan (I).

Palazzo and Bolini (2009) analyzed raspberry gelatin through MTIA, which graphically represents the dynamic profiles by overlapping curves of two or more sensory attributes of a specific sample. Through MTIA, it is possible to verify the beginning of the peak intensity or decrease of important attributes to consumers in specific products. These results contribute to the production of foods with sensorial characteristics of intensity and duration time of sensory characteristics that may have an influence on the consumer's decision of purchase and choice for several products (Palazzo and Bolini 2014).

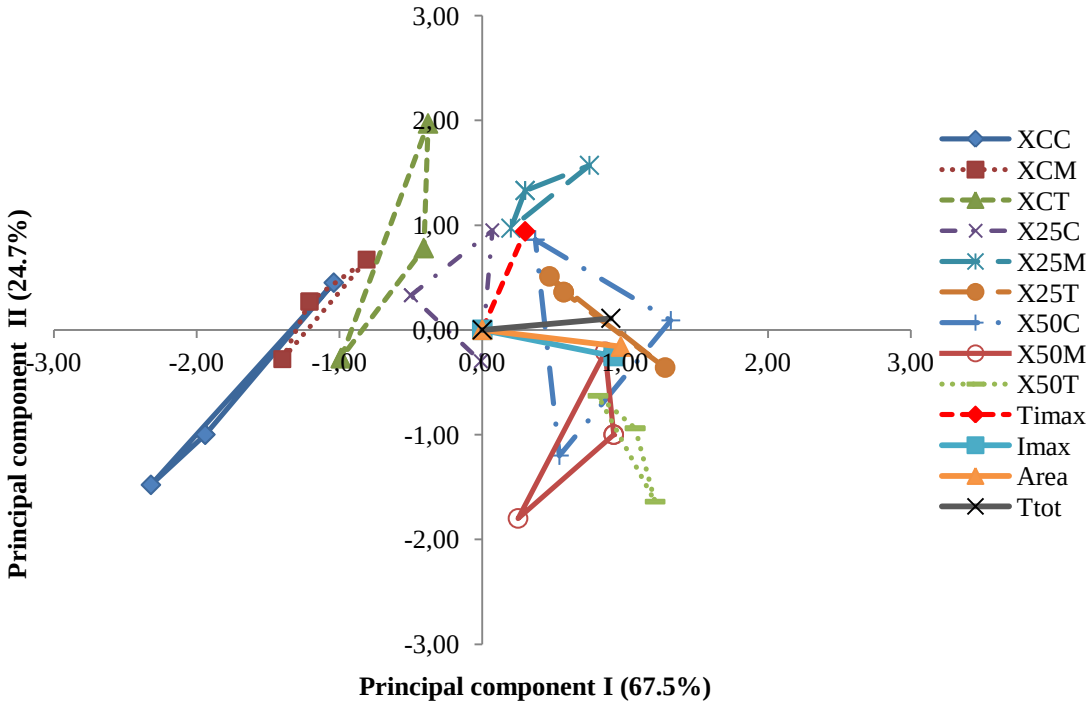

Figure 2 – Principal Component Analysis of sourness in xanthan gum model systems.

With the data collected from each sample, the Principal Component Analysis (PCA) was performed for the evaluated attributes. For the sourness attribute, the two major components accounted for 92.2% of the total variability observed between the samples (Figure 2). Similar samples occupy regions near the graph and are characterized by the vectors that are close to them. Therefore, the vectors (Imax, Ttot and Area) are focused on the samples submitted to processing in high pressure homogenizer (X25T, X50C, X50M and X50T), characterizing them as being of greater intensity for the acid stimulus. Samples XCC and X25M were characterized by the vector (Timax). It can be observed that the control model systems are in opposition to all the vectors, revealing that they have a lower intensity of sourness when compared to the samples submitted to homogenization.

For the sweet taste stimulus, we observed that the two main components explained 83.4% of the total variation among the samples (data not shown). The proximity among the samples indicates that they have similar temporal profiles in xanthan gum model systems for the sweet taste attribute. Samples XCT and X25T showed to be opposite to the vectors, being less characterized by the evaluated parameters.

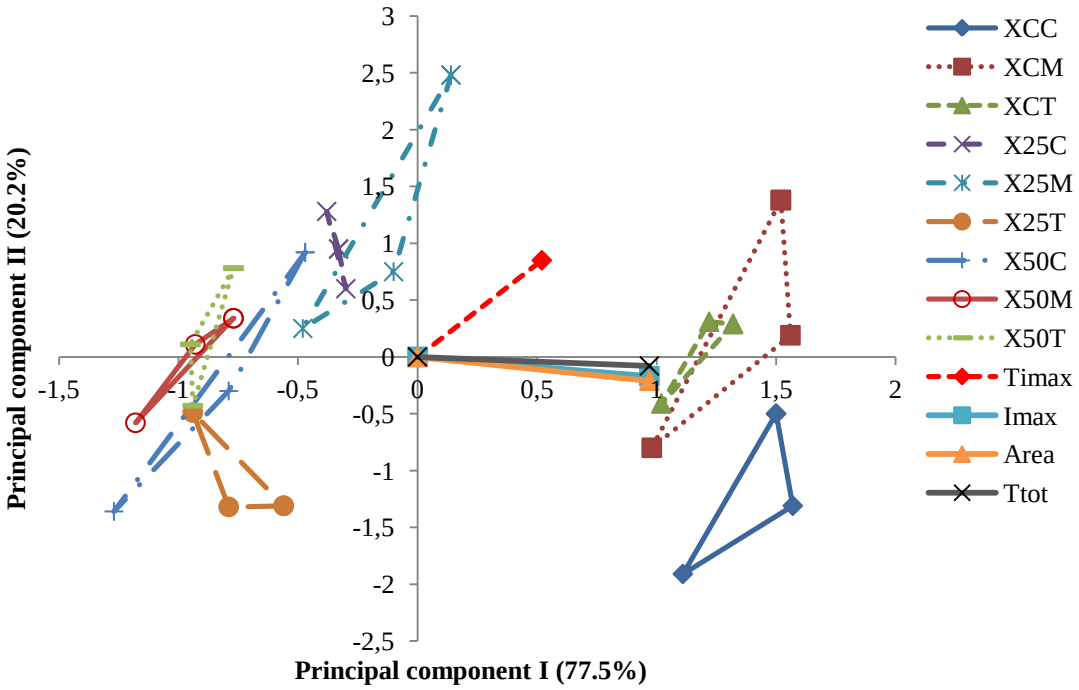

Figure 3 – Principal Component Analysis of viscosity in xanthan gum model systems.

In Figure 3, it can be seen that 77.5% of the total variation verified between the samples in relation to the viscosity attribute was explained by the first axis (main component I), and the parameters Imax, Ttot and Area contributed to the variability associated with this axis. The total percentage of the variation explained by the analysis of principal components is 97.7%. It was also observed that the control samples (XCC, XCM and XCT) are very close to each other, and are characterized by the vectors of Imax, Ttot and Area, indicating them as of greater intensity for the viscosity attribute. We also found that model systems homogenized at 25 and 50 MPa, are all opposed to the vectors of maximum intensity, total time of stimulus perception and area, revealing that these samples are less viscous compared to the control samples. The proximity between the samples indicates a similarity in the temporal profile. Thus, the positioning of the control samples (XCC, XCM and XCT) may indicate that they have similar temporal profiles for the viscosity stimulus. The samples submitted to the

homogenization process (25 and 50 MPa) are close to each other, indicating similarity, and are apart from the other ones (0 MPa) in the graphical representation, indicating that their temporal profile is distinct from them. According to Augusto et al. (2011), one of the advantages of using model systems for scientific research is the experimental reproducibility, which minimizes the effects of inherent variations in the characteristics of the food.

3.2. Rheological behavior

Figure 4 shows the flow behavior of the xanthan gum model systems with acidulants adjusted to the Ostwald-de-Waele model. This model has been extensively used to describe the rheological behavior of juices and nectars (Faraoni et al. 2013), for being simple and of wide technological application (Branco and Gasparetto 2003). Model systems have been characterized as non-Newtonian fluids with pseudoplastic characteristics, i.e. when at rest their molecules are in a cluttered state and when subjected to shear stress, their molecules tend to orient themselves toward the flow stream.

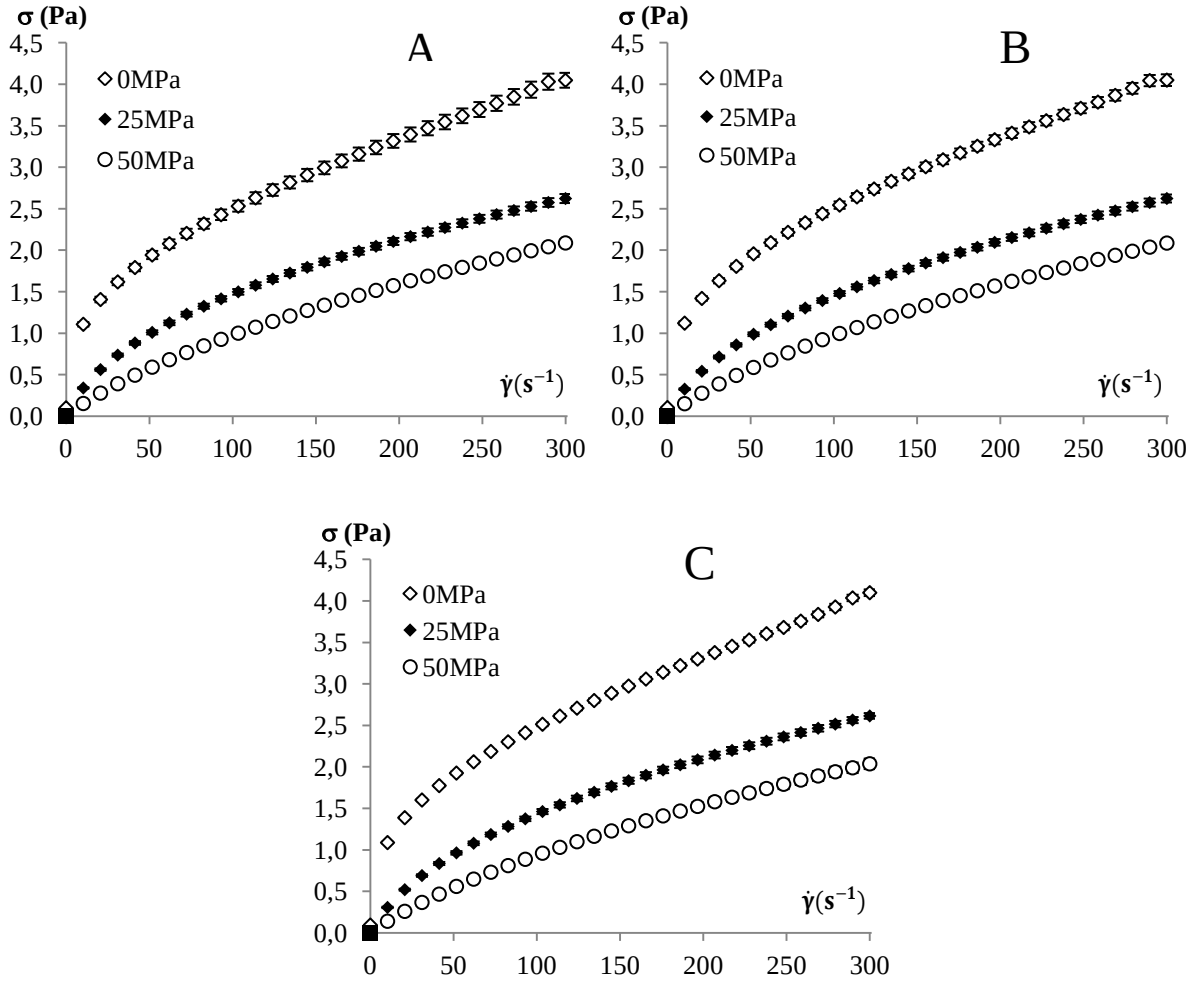

Figure 4 – Flow behavior at 25 °C in model systems of xanthan gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

According to Sanderson (1981), the conformation of xanthan gum when dispersed in solution is responsible for the high viscosity at rest and low shear viscosity, as a consequence of the weak molecular interactions at low concentrations of the gum. This conformation is responsible for the pseudoplastic behavior of the gum, that is, it decreases the viscosity with the increase of the deformation rate.

The behavior of the flow was affected by the P_H as can be observed in Figure 4, there was a decrease in the homogenized samples. It is noted that at a specific shear rate, the shear stress values are lower in the samples homogenized at 25 and 50 MPa. Regarding apparent viscosity (Figure 5), we also observed that the non-homogenized samples had higher values, independent of the shear rate. The P_H affected the apparent viscosity of the model

systems of xanthan gum, since it decreased approximately 98% of its initial value when the samples were processed.

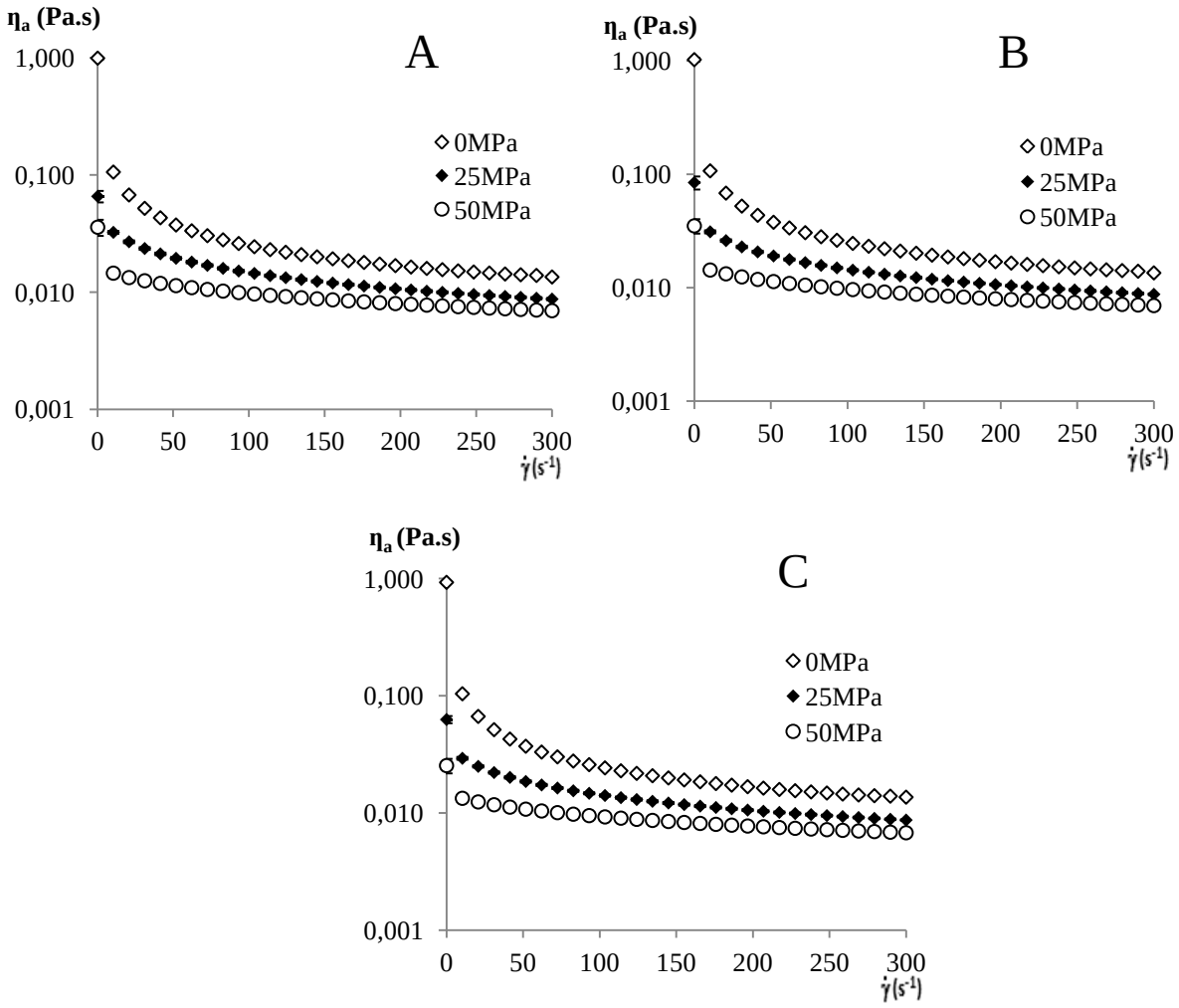

Figure 5 – Apparent viscosity at 25 °C in model systems of xanthan gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

We also observed that the rheological behavior to the flow and the effect of P_H were similar among the samples with the different organic acids used. Augusto et al. (2012) evaluated a serum model for application in tomato juice and observed reduction in fluid viscosity and that the rheological properties of the serum are slightly affected by processing in high pressure homogenizer.

The control and homogenized model systems had an apparent viscosity of approximately 0.04 and 0.02 Pa.s, respectively, when measured at the shear rate of $50s^{-1}$.

Researches report that changes in the viscosity of non-Newtonian liquids with $50s^{-1}$ shear rate has been adopted as swallowing standard (Ong et al. 2018; Kim et al. 2017). A relationship between the rheological properties of the flow and the sensory results was also observed. As seen previously in Table 2, the values of maximum intensity, total time of perception of the attribute and area, were higher for the control samples (0 MPa), characterizing them as being more viscous. The control model systems (XCC, XCM and XCT) differed significantly from the samples processed in high pressure homogenizer ($p < 0.05$) (Table 2). On the other hand, the homogenized samples (25 and 50 MPa) presented lower viscosity, equivalently to the rheological results, since homogenization process altered the flow behavior and reduced the apparent viscosity models of xanthan gum with organic acids. According to Holdsworth (1993), the study of rheological behavior has several purposes, such as determining the functionality of an ingredient in product development, quality control, product life and correlation with data obtained by sensory analysis, besides being essential for the design of equipment and processes (Rao 2014).

The time-intensity analysis showed no significant difference ($p < 0.05$) in the viscosity of the homogenized solutions at 25 and 50 MPa. Therefore, it can be stated that P_H altered the viscosity perception between the control samples and the processed ones, but did not interfere between the homogenized (25 and 50 MPa) nor between the different organic acids used.

Table 3 shows the effect of P_H on the parameters of the Ostwald-de-Waele model for the xanthan gum model systems with organic acids. The consistency index (*k*) decreased with increasing pressure, but the flow behavior index (*n*) increased with the homogenization process. That is, samples with lower viscosity had a lower consistency index and higher flow behavior index. It can be stated that the different organic acids used did not interfere in the flow behavior, since the xanthan gum model systems presented very close consistency index value and flow behavior index values. With the increase of the P_H from 0 to 50 MPa, the citric acid model systems presented decreasing values for the consistency index of 0.374 to 0.036 $Pa.s^n$ and increasing values for the flow behavior index of 0.413 to 0.714. These variations were observed among all analyzed model systems. These results represent a trend in works with different types of polysaccharides (Wang et al. 2011; Floury et al. 2002; Lagoueyte and Paquin 1998).

Table 3 – Effect of homogenization pressure (P_H) on flow properties in xanthan gum model systems: Parameters of the Ostwald-de-Waele model at 25 °C.

P_H	Citric acid		Malic acid		Tartaric acid	
	k (Pa.s^n)	*n* (-)	*k* (Pa.s^n)	*n* (-)	*k* (Pa.s^n)	*n* (-)
0MPa	0.374 ± 0.011	0.413 ± 0.003	0.381 ± 0.009	0.412 ± 0.002	0.362 ± 0.003	0.420 ± 0.001
25MPa	0.115 ± 0.004	0.549 ± 0.003	0.108 ± 0.004	0.560 ± 0.003	0.101 ± 0.004	0.570 ± 0.005
50MPa	0.036 ± 0.001	0.714 ± 0.001	0.035 ± 0.001	0.714 ± 0.003	0.032 ± 0.002	0.727 ± 0.003

Food research in model systems using emerging technologies is important because of the potential of industrial application and process optimization. The use of a high pressure homogenizer has been studied in different food matrices, therefore studying rheological behavior is of fundamental importance, as it is useful not only in relation to product quality, but also in relation to operational projects and process optimization (Augusto et al. 2012).

4. Conclusions

The P_H altered the rheological behavior of the flow in the model systems of xanthan gum with acidulants for fruit nectar. With the increase of P_H, the model systems showed reduction in the apparent viscosity and pseudoplastic behavior, with reduction in the consistency index and increase in the flow behavior index. The different organic acids used did not affect the rheological properties of the model systems. The technology used contributed to a greater temporal profile of sourness, while the sensory perception of sweet taste was not affected by the process in high pressure homogenizer. The control model systems presented higher maximum intensity for the viscosity attribute and the homogenized samples (25 and 50 MPa) lower intensity, that is, they were less viscous, being sensorially similar to each other. These results contribute to further research and potential industrial applications.

Notation

$\dot{\gamma}$ shear rate [s^{-1}]

η_a apparent viscosity (= $\sigma/\dot{\gamma}$) [Pa·s]

σ shear stress [Pa]

k consistency index, Ostwald-de-Waele model (Eq. (1)) [Pa·s^n]

n flow behavior index, Ostwald-de-Waele model (Eq. (1)) [–]

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Disclosure statement

The authors declare that there is no conflict of interest.

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Artigo 3 - EFFECT OF THE HOMOGENIZATION PROCESS ON THE SENSORY AND RHEOLOGICAL PROPERTIES IN MODEL SYSTEM

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ABSTRACT

The use of non-thermal technologies such as the high pressure homogenization has been studied in several food products. However, its use in model systems for fruit nectar is innovative. This study aimed to evaluate the effect of the processing in high pressure homogenizer on the rheological behavior and the sensory attributes in model system of gellan gum. Gellan gum (0.05%), organic acids (0.3%), and sucrose (10%) were used to prepare the solutions, which were subsequently homogenized (0 - control, 25 and 50 MPa) at 25 °C. Rheological and sensory analyses were performed. The samples presented pseudoplastic behavior and were characterized by the Ostwald-de-Waele model. The homogenization pressure (P_H) altered the viscosity of the model systems, reduced the consistency index and apparent viscosity, and increased the flow behavior index. The stimuli sour taste and viscosity differed among the parameters evaluated in the time-intensity analysis. No differences were observed for the maximum intensity of viscosity between the treated samples (25 and 50 MPa), which exhibited a similar temporal profile. Therefore, studies on the rheological and sensory behavior are fundamental to product development and process optimization.

Keywords: homogenization; time-intensity analysis; rheological behavior; gellan gum; acidulants.

1. Introduction

The use of non-thermal technologies in food processing has stood out for improving the technological processes and food quality attributes. In the high pressure homogenizer, the fluid is forced to flow rapidly through a narrow orifice valve, with a consequent increase in speed, followed by depressurization, resulting in high shear stress, turbulence, and cavitation (Dumay et al., 2013; Augusto et al., 2013).

Several authors have studied the food behavior with the use of emerging technologies (Zhou et al., 2017; Yu et al., 2014; Calligaris et al., 2012; Maresca et al., 2011; Tribst et al., 2011). The homogenization process promotes changes in the rheological and physical properties of the products, such as viscosity change. Thus, the use of the high pressure homogenizer presents an alternative to modify the rheological properties in model system. However, this study is innovative because little is known about the sensory and rheological characteristics in model systems for fruit nectar.

Organic acids are substances naturally present in plants and can be used as acidulants and acidity regulators. The acidulants reduce the pH of the medium favoring food preservation, confer stability, and inhibiting oxidative reactions, besides the potential to enhance the food flavor (Damodaran et al., 2010).

In turn, gellan gum is an extracellular polysaccharide secreted by the microorganism *Pseudomonas elodea*. At low concentrations (0.05%), this polysaccharide forms low-viscosity solutions at high temperatures, forming a gel under cooling. Gellan gum has been applied in the food industry and biotech applications for forming a transparent gel that resists heating and acids. Due to the ability of gellan to form gels at low concentrations, together with its versatile functional properties, it allows the production of new textures and, consequently, the development of new products (Sanderson & Ortega, 1993; Yamamoto, 2006). Therefore, research using gellan gum associated with non-thermal technologies can be an effective alternative for the development of new products.

The knowledge of the rheological behavior is fundamental to determine the functionality of an ingredient for the development of products, and to correlate with sensory data, as well as the evaluation and operation of food processing equipment (Ibarz et al., 1996; Holdsworth, 1993).

The time-intensity analysis determines the relationship between the perceived intensity of a stimulus and the duration perception. This sensory method has been used to characterize food and to develop new products, besides contributing to the analysis of

attributes that undergo changes during processing (Lawless & Heymann, 2010; Noble et al., 1991). Whereas the sensory characteristics of a food product are important for the consumer's perception, the study of these characteristics is fundamental, especially when an innovative process is used.

Thus, this study aimed to evaluate the effect of the processing in high pressure homogenizer on the rheological and sensory properties in model system of gellan gum.

2. Materials and Methods

2.1. Material

In the model systems, the thickener gellan gum (Kelcogel HF-B®, CPKelco, Limeira, São Paulo, Brazil) was used at the concentration of 0.05% (w/v) (Brasil, 2013) and the organic acids (0.3%, w/v) citric, malic and tartaric (Synth®, Diadema, São Paulo, Brazil) were used separately in each thickener solution. The model systems were sweetened with refined sugar sucrose (10%) (w/v) (União®, São Paulo, Brazil).

2.2. Preparation of the samples

For the preparation of the model systems acidified with organic acids, gellan gum was mixed with sucrose and then dissolved in deionized water at 70 °C under stirring (Shaker Fisatom, 713D, 1640 rpm). The dissolution lasted 3 hours, followed by cooling under room temperature. Simultaneously to the preparation of the thickeners, the organic acid was added. The whole process was carried out under agitation, at pH 2.65. The rheological and sensory evaluation was performed in triplicate.

2.3. Homogenization

A high-pressure homogenizer (Panda Plus, GEA Niro Soavi) was used to treat the samples (0 - control, 25, and 50 MPa). The samples were processed at 25 °C and then stored under refrigeration (7 ± 2 °C) for 21 days. The rheological and sensory analyses were performed at room temperature (24 ± 2 ºC). Nine model systems were developed, each containing a different organic acid (citric, malic, and tartaric acid).

2.4. Rheological Analysis

A controlled voltage rheometer (AR2000ex, TA Instruments) with cone-plate geometry (60 mm diameter, 2 °) was used for the rheological analysis. The model systems were analyzed at a constant temperature (25 °C) through a Peltier system. The analyses were obtained in steady state with decreasing shear rate (0.1-300 s^{-1}) for 5 min. Data were described by the Ostwald-de-Waele model (Eq. (1)).

$$\sigma = k \cdot \dot{\gamma}^{n} \quad (1)$$

The model parameters in Eq. (1) were modeled as a function of P_H using the CurveExpert Professional software v.1.6.3, at a 95% significance.

2.5. Sensory Evaluation

The sensory evaluation was performed in individual booths at the Laboratory of Sensory Science and Consumer Studies at the Department of Food and Nutrition (FEA/UNICAMP). The samples were presented in disposable cups, coded with three-digit numbers, in complete balanced blocks (MacFie et al., 1989), in monadic form, with three replications. Mineral water was provided to the assessors as a palate cleanser.

This research project was approved by the Research Ethics Committee of UNICAMP, CAAE: 52934315.7.0000.5404. The Free and Informed Consent Form containing information about the research was presented to the assessors.

2.5.1. Time-Intensity Analysis

Individuals who were interested in participating in the project were recruited among undergraduate and graduate students, and staff at UNICAMP. For the time-intensity analysis, a pre-selection was performed, and the candidates were subjected to training sessions and subsequently selected. During the pre-selection stage, the individuals were evaluated through triangular tests using the Wald sequential analysis (Amerine et al., 1965) to select the assessors with discriminative ability. A control sample and two thickener samples acidified with organic acids were used, with a significant difference at 0.1% in relation to the

viscosity. The parameters used for pre-selection in the sequential analysis were: $\rho_0 = 0.45$, $\rho_1 = 0.70$, $\alpha = 0.05$ e $\beta = 0.05$.

2.5.2. Training Session

The time-intensity analysis was performed in the Time-Intensity Analysis of Flavors and Tastes (TIAFT) data collection program (University of Campinas - UNICAMP, 2012), developed at the Laboratory of Sensory Science and Consumer Studies at the Faculty of Food Engineering (UNICAMP).

The assessors were trained to use the TIAFT software and to form a sensory memory with the extremes of the scale for the stimuli sweetness, sourness, and viscosity. A discrepancy between the samples, repeatability of results and agreement with the team was required before the test application (Damásio & Costell, 1991).

The parameters maximum intensity (Imax), time of maximum intensity perceived (Tmax), total duration of perception (Ttot) and area under the curve (Area) were obtained using the TIAFT for the time-intensity curve (Palazzo & Bolini, 2014).

For the stimuli sweetness and sourness, the initial waiting time (5 s); residence time in the mouth (25 s); time after ingestion (10 s) and scale (10) was used; for the stimulus viscosity, the initial wait was (5 s); residence time in the mouth (10 s); time after ingestion (40 s), and scale (10).

2.5.3. Selection of the assessors

Sixteen individuals were pre-selected and trained to use the time-intensity method. Experimental samples (3 samples with different thickener concentrations) were used to select the assessors with the ability to discriminate the samples and repeatability of results in agreement with the team (Damásio & Costell 1991). The sensory team was composed of 11 selected assessors.

The stimuli sweetness, sourness and viscosity were evaluated in a monadic manner, in three replicates. The intensity of the stimulus was recorded as a function of the time, using a mouse, in a linear scale ranging from 0 to 10, in which 0 corresponded to none (left side), 5 corresponded to moderate, and 10 corresponded to a lot (right side).

At the first warning issued by the computer (5 s) the start button was pressed, the assessor should put the sample in the mouth and indicate the intensity of the attribute

viscosity in the scale, using the mouse. At the second beep (10 s), the assessor should swallow the sample, and a third beep (40 s) indicated the end of the test. For the attribute sweetness and sourness, at the first beep (5 s) the start button was pressed, the assessor should put the sample in the mouth and indicate the intensity of the attribute in the scale. Upon hearing the second beep (25 s), the assessor should swallow the sample, and a third warning (10 s) indicated the end of the test.

Analysis of variance (ANOVA) was used to select each assessor in relation to each parameter. The 11 selected assessors showed ability to discriminate the samples ($p < 0.30$) and reproducibility of results ($p > 0.05$) (Damasio & Costell 1991).

2.5.4. Time-intensity analysis of the attributes

The attributes sweetness, sourness and viscosity of the model systems were analyzed by the assessors. The samples were presented in a monadic form, in three replicates. The selected assessors recorded the temporal perception of the stimuli analyzed for the parameters Imax, TImax, Area, and Ttot. Viscosity was defined as the internal resistance that the particles of a substance present when slipping on top of each other. The sweet taste was defined as the characteristic of a sucrose solution, and sour taste as the characteristic of a citric acid solution. The averages of each curve were determined from the results.

2.6. Statistical analysis

The results were submitted to analysis of variance (ANOVA) and Tukey's test ($p \leq 0.05$). The time-intensity analysis data were submitted to Principal Component Analysis (PCA). The Statistical Analysis System - SAS 9.4 program was used.

3. Results and Discussion

3.1. Time-Intensity Analysis

Table 1 shows the mean values of the parameters analyzed in the gellan gum model systems. For the maximum intensity of sourness, the model systems subjected to processing in high pressure homogenizer presented the highest values. The processing did not interfere in the sour taste intensity between the samples containing the same organic acid.

Thus, no significant differences ($p < 0.05$) were observed between the samples (GECC, GE25C and GE50C), that is, between the control and the treated samples containing the same organic acid. This behavior was also observed in models using malic acid and tartaric acid, evidencing that the processing in high pressure homogenizer may be a viable alternative for application in model systems using fruit nectar, once the intensity of sourness did not differ ($p < 0.05$) among the samples using the same organic acid.

Table 1 – Results of the parameters of time-intensity curves for sourness attribute[1].

Samples	Timax*	Imax	Ttot*	Area
GECC	12.61^{a}	4.14^{d}	26.55^{a}	59.07^{c}
GECM	12.53^{a}	4.70^{cd}	25.53^{a}	65.91^{abc}
GECT	12.93^{a}	5.07^{abc}	28.23^{a}	75.03^{abc}
GE25C	12.22^{a}	4.84^{bcd}	26.93^{a}	67.68^{abc}
GE25M	12.53^{a}	5.39^{abc}	28.67^{a}	82.66^{ab}
GE25T	12.76^{a}	5.66^{ab}	28.99^{a}	85.65^{a}
GE50C	12.51^{a}	4.78^{cd}	25.82^{a}	64.90^{bc}
GE50M	12.52^{a}	5.01^{abc}	27.82^{a}	74.92^{abc}
GE50T	12.93^{a}	5.78^{a}	27.95^{a}	85.26^{a}
MDS[2]	1.42	0.84	3.49	20.18

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]MDS – Minimum significant difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area – area under the curve. GECC – citric control gellan; GECM – malic control gellan; GECT – tartaric control gellan; GE25C – citric 25MPa gellan; GE25M – malic 25MPa gellan; GE25T – tartaric 25MPa gellan; GE50C – citric 50MPa gellan; GE50M – malic 50MPa gellan; GE50T – tartaric 50MPa gellan. *Time in seconds.

The models with tartaric acid (GECT, GE25T, and GE50T) showed higher maximum intensity values, indicating that it was perceived by the assessors, with greater intensity of sourness. This characteristic is related to the chemical nature of the organic acid. Some authors have reported that the intensity of sourness may be related both to the concentration of hydrogen ions in solution and the concentration of protonated (non-dissociated) organic acids (Da Conceição Neta et al., 2007; Johanningsmeier et al., 2005).

The model system GE50T presented the highest maximum intensity (5.78) for the stimulus sourness, differing from the samples GE50C, GE25C, GECC, and GECM. The processing in high pressure homogenizer did not affect the time of maximum intensity and

total duration of perception of sourness, with no significant differences ($p < 0.05$) between the control and the homogenized samples, with values ranging from 12.22 to 12.93 s, and 25.53 to 28.99 s, respectively.

In the parameter Area, the sample GECC presented a value of 59.07, thus it was characterized as the model system with the lower Area, with a significant difference ($p < 0.05$) from the samples GE25M, GE25T, and GE50T. The P_H did not affect the parameter Area of the samples containing the same organic acid, with no differences ($p < 0.05$) between the control and the homogenized samples (GECT, GE25T, and GE50T). This characteristic was also observed in the model systems using citric and malic acid. The samples with the addition of tartaric acid had higher Area values.

The use of high pressure homogenizer and the addition of different organic acids did not interfere in the perception of sweetness in the gellan gum model systems, with no significant difference ($p < 0.05$) among all parameters evaluated (data not shown). The values of maximum intensity, time of maximum intensity perceived, and total duration of perception of sweetness ranged from 4.77 to 5.41, 12.67 to 13.72 s, and 22.48 to 24.19 s, respectively, for the gellan gum model systems.

Table 2 – Results of the parameters of time-intensity curves for viscosity attribute[1].

Samples	Timax*	Imax	Ttot*	Area
GECC	9.63^{a}	3.93^{a}	25.14^{a}	66.48^{ab}
GECM	8.45^{abc}	4.35^{a}	24.98^{a}	75.37^{a}
GECT	9.22^{ab}	4.04^{a}	25.46^{a}	71.12^{a}
GE25C	8.99^{abc}	3.22^{b}	24.17^{a}	56.29^{bc}
GE25M	7.54^{c}	2.87^{b}	24.08^{a}	48.66^{c}
GE25T	8.12^{bc}	3.11^{b}	24.60^{a}	53.86^{c}
GE50C	8.26^{abc}	3.18^{b}	23.98^{a}	55.53^{c}
GE50M	9.11^{ab}	2.93^{b}	25.15^{a}	51.05^{c}
GE50T	9.10^{ab}	3.16^{b}	24.39^{a}	56.39^{bc}
MDS[2]	1.47	0.55	1.66	10.87

[1]Means followed by the same letter in the column, do not differ by Tukey test ($p \leq 0.05$). [2]MDS – Minimum significant difference. Imax - maximum intensity; Timax - time at which the maximum intensity was perceived; Ttot - total time of attribute duration; Area – area under the curve. GECC – citric control gellan; GECM – malic control gellan; GECT – tartaric control gellan; GE25C – citric 25MPa gellan; GE25M – malic 25MPa gellan; GE25T – tartaric 25MPa gellan; GE50C – citric 50MPa gellan; GE50M – malic 50MPa gellan; GE50T – tartaric 50MPa gellan. *Time in seconds.

As shown in Table 2, the processing in high pressure homogenizer altered the temporal profile of gellan gum model systems. Significant differences ($p < 0.05$) were observed for the time of maximum intensity, and the sample GECC presented the highest value (9.63 s), which was significantly different from the samples GE25M and GE25T.

With respect to the parameter maximum intensity, a significant difference ($p < 0.05$) was observed between the control and the homogenized samples (25 and 50 MPa). Thus, it can be stated that P_H altered the sensory perception of viscosity since the assessors attributed a higher maximum intensity to the control samples. No differences ($p < 0.05$) were observed between the homogenized model systems (25 and 50 MPa) for this parameter, which exhibited similar temporal profiles, with values ranging from 4.35 to 2.87 for the samples GECM and GE25M, respectively. This decrease in viscosity perception is probably due to the breakdown of the polysaccharide chain during processing in high pressure homogenizer (Wang et al., 2011; Harte & Venegas, 2010).

The total duration of perception ranged from 23.98 and 25.46 s, without significant differences ($p < 0.05$) between the samples. For the parameter Area, significant differences ($p < 0.05$) were observed between the control and the homogenized samples, with values ranging from 75.37 to 48.66 for GECM and GE25M, respectively, while no differences were observed between the homogenized samples ($p < 0.05$).

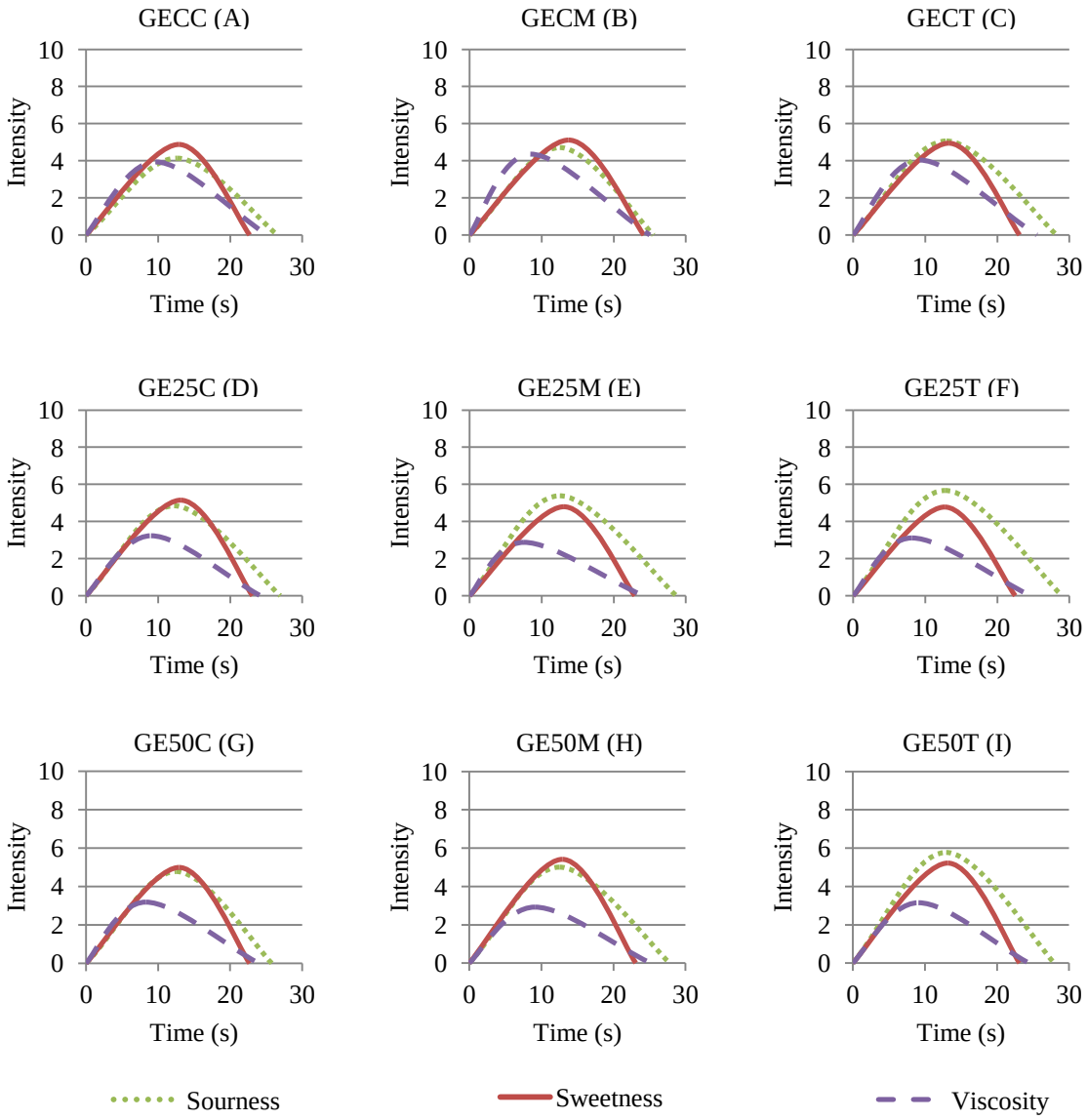

Figure 1 – Multiple time-intensity curves in model systems of gellan gum. GECC - citric control gellan (A); GECM - malic control gellan (B); GECT - tartaric control gellan (C); GE25C - citric 25MPa gellan (D); GE25M - malic 25MPa gellan (E); G25T - tartaric 25MPa gellan (F); GE50C - citric 50MPa gellan (G); GE50M - malic 50MPa gellan (H); GE50T - tartaric 50MPa gellan (I).

Time-intensity analysis allows investigating the dynamic evolution of sensory perceptions over time (Saint-Denis, 2018). Thus, the assessors are trained to assess the intensity of the sensory attributes continuously and over time. For a better understanding, Figure 1 shows the curves of the multiple time-intensity analysis (MTIA) of the sensory perceptions of the attributes analyzed in the gellan gum model systems.

The attributes sourness, sweetness and viscosity were analyzed as a function of time (Figure 1). It was observed that the model systems with tartaric acid presented higher maximum intensity of sour taste. The total duration of perception of sourness was similar for all samples studied. It is worth noting that P_H did not affect the maximum intensity of sourness of the model systems with the addition of the same organic acid. However, the process altered the viscosity of the model systems, with a different time profile between the control and the homogenized samples (25 and 50 MPa), with a higher intensity for the samples GECC, GECM, and GECT, which were more viscous. The homogenized model systems were less viscous and exhibited similar sensory profiles for the stimulus viscosity. The time-intensity curve of sweetness showed that the treated samples were not affected by P_H. Therefore, the processing in high pressure homogenizer (25 and 50 MPa) and the use of different organic acids can be an alternative for application in fruit nectars, with emphasis on sour taste and viscosity.

Principal Component Analysis (PCA) was performed for the attribute sourness in all model systems (Figure 2). The parameters evaluated in the temporal analysis are represented by vectors. It was observed that 90.2% of the variation between the samples were explained by the principal components I and II.

The sample GECT was mainly characterized by the vector (Timax), while the samples GE25M, GE25T, and GE50T were characterized by the vectors Imax, Ttot, and Area (Figure 2). Thus, it can be stated that the samples with the addition of tartaric acid showed higher intensity of sourness. The samples were close to the vectors that characterize them, and the samples with similarities between them were located next to each other in the PCA chart. It was also verified that the other model systems were opposite to the vector parameters (Timax, Imax, Ttot, and Area), revealing that these samples were less characterized by these parameters.

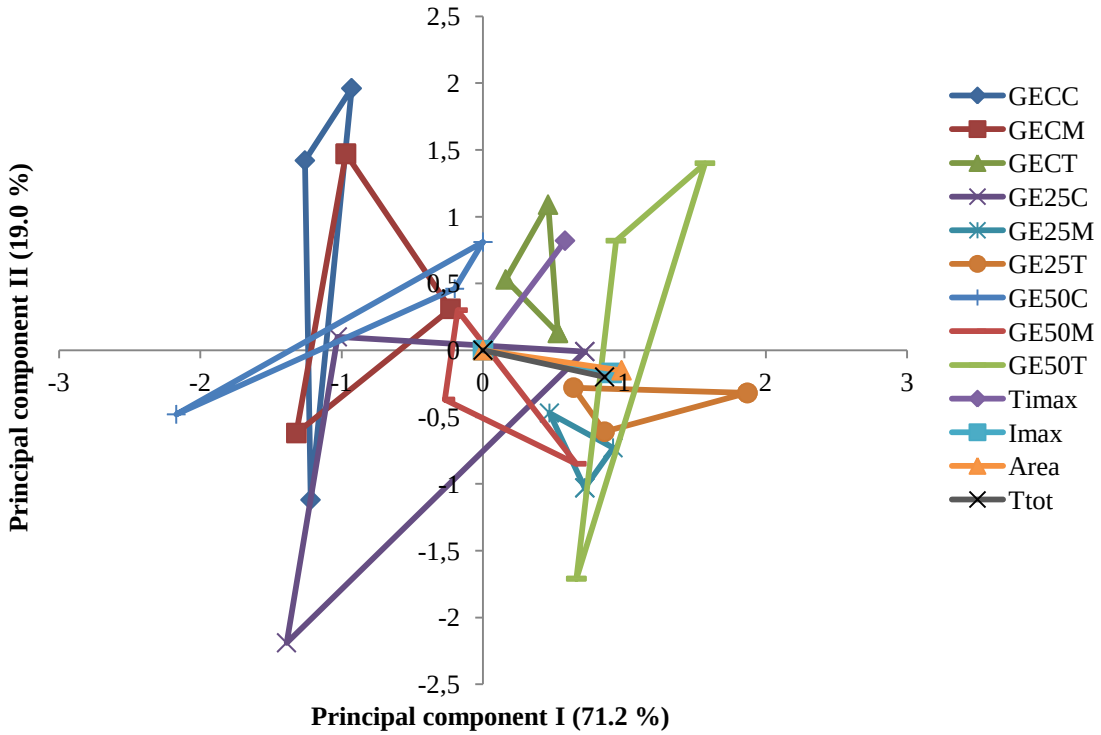

Figure 2 – Principal Component Analysis of sourness in gellan gum model systems.

The Principal Component Analysis of sweetness in the gellan gum model systems explained 87.8% (data not shown) of the variance between the samples. The control and the homogenized samples (25 and 50 MPa) showed proximities and similarities in the temporal profile, indicating that they were similar for the stimulus sweetness.

Time-intensity analysis has been applied in the sensory evaluation of several products (Azevedo et al., 2017; Rodrigues et al., 2015; Morais et al., 2014; Melo et al., 2007) for the temporal evaluation of the attributes and sensations perceived in food over time.

As shown in Figure 3, the total variance explained by PCA was 86.9%, and for viscosity, 66.7% was explained by the principal component I, and the parameters Imax, Ttot, and Area contributed to the variance associated with this axis.

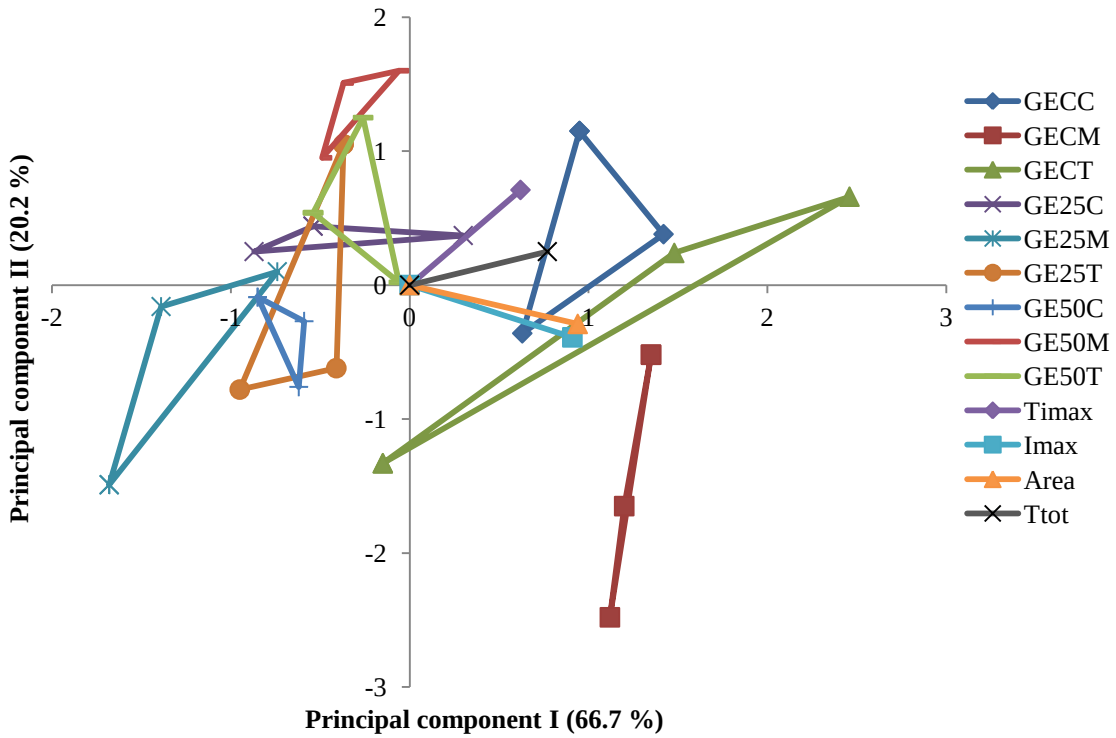

Figure 3 – Principal Component Analysis of viscosity in gellan gum model systems.

The control model systems showed proximity to each other, indicating a similar temporal profile for the attribute viscosity (Figure 3). The samples GECC and GECT were characterized by the vectors Timax, Imax, Ttot, and Area, while the sample GECM was mainly characterized by the vectors Imax and Area. Thus, it can be stated that the control model systems presented a higher maximum intensity of the attribute viscosity, being characterized as the most viscous sample.

In addition, the homogenized samples (25 and 50 MPa) were all opposed to the vectors, confirming that these samples were less characterized by the parameters evaluated when compared to the control (Figure 3). The homogenized model systems (25 and 50 MPa) have similar temporal profiles, thus they were similar for the attribute viscosity.

3.2. Rheological Analysis

As can be seen in Figure 4, the use of the high pressure homogenizer altered the flow rate of the gellan gum model systems. The homogenized samples (25 and 50 MPa) presented a decrease in the flow behavior, once lower shear stress values were observed at a fixed shear rate. However, a similar flow behavior and an impact of the processing was observed for the different organic acids.

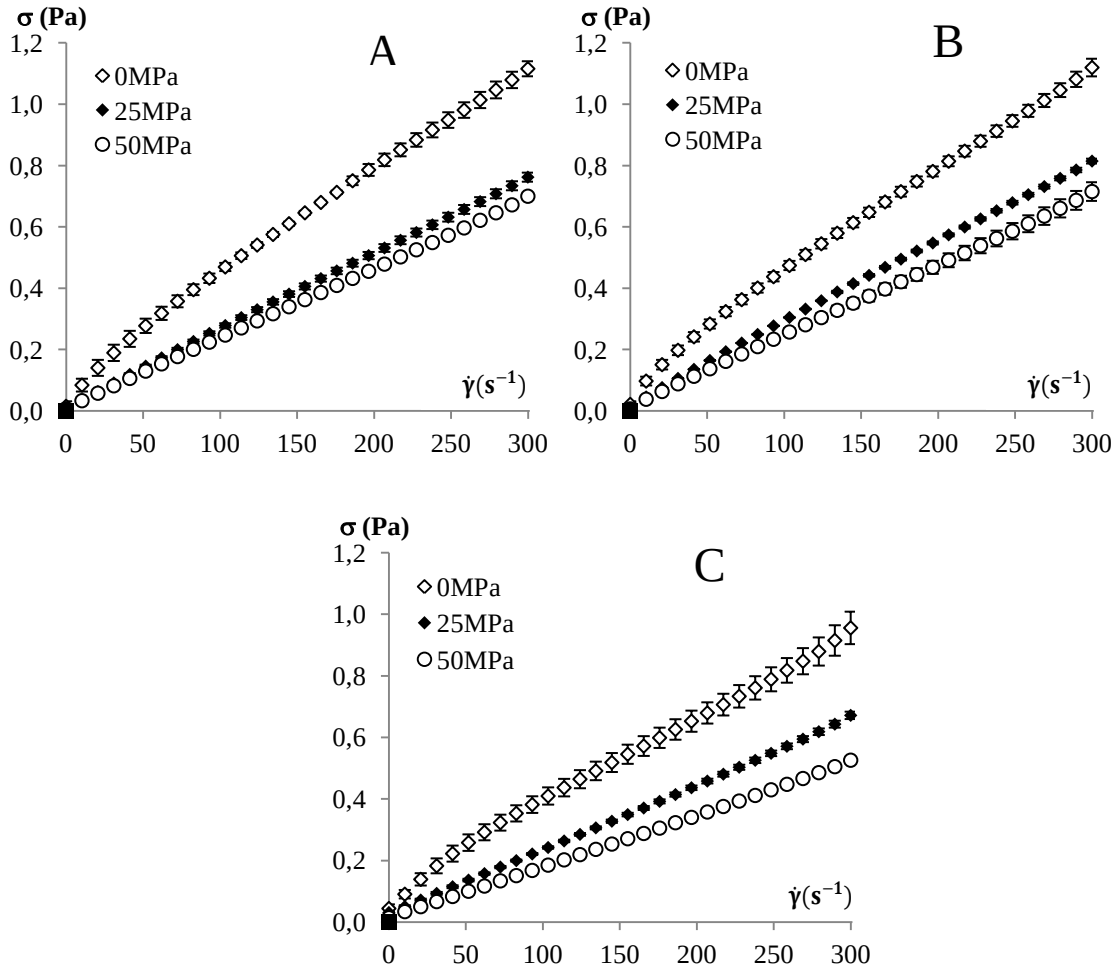

Figure 4 – Flow curve at 25 °C in model systems of gellan gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

As shown in Figure 5, the processing in high pressure homogenizer led to a reduction of the apparent viscosity of the homogenized samples (25 and 50 MPa), which presented lower values regardless of the shear rate, with a reduction of approximately 98.5 to 99.6% in relation to the control. Research shows that the current standard of oral shear rate has been reported as $50s^{-1}$ (Kim et al., 2017; Ferry et al., 2006). Changes in viscosity of non-Newtonian control and homogenized model systems were observed to have an apparent viscosity of approximately 0.006 and 0.002 Pa.s, respectively, when measured at the shear rate of $50s^{-1}$. Relating these data to those of viscosity through time analysis (Table 2) it was observed that, the maximum intensity of viscosity was higher in the control model systems (0 MPa), characterizing them as more viscous samples. Although the homogenized samples no

differences in viscosity (25 and 50 MPa), a difference was perceptible in the control samples. Thus, the processed samples presented lower viscosity, equivalently to the rheological results.

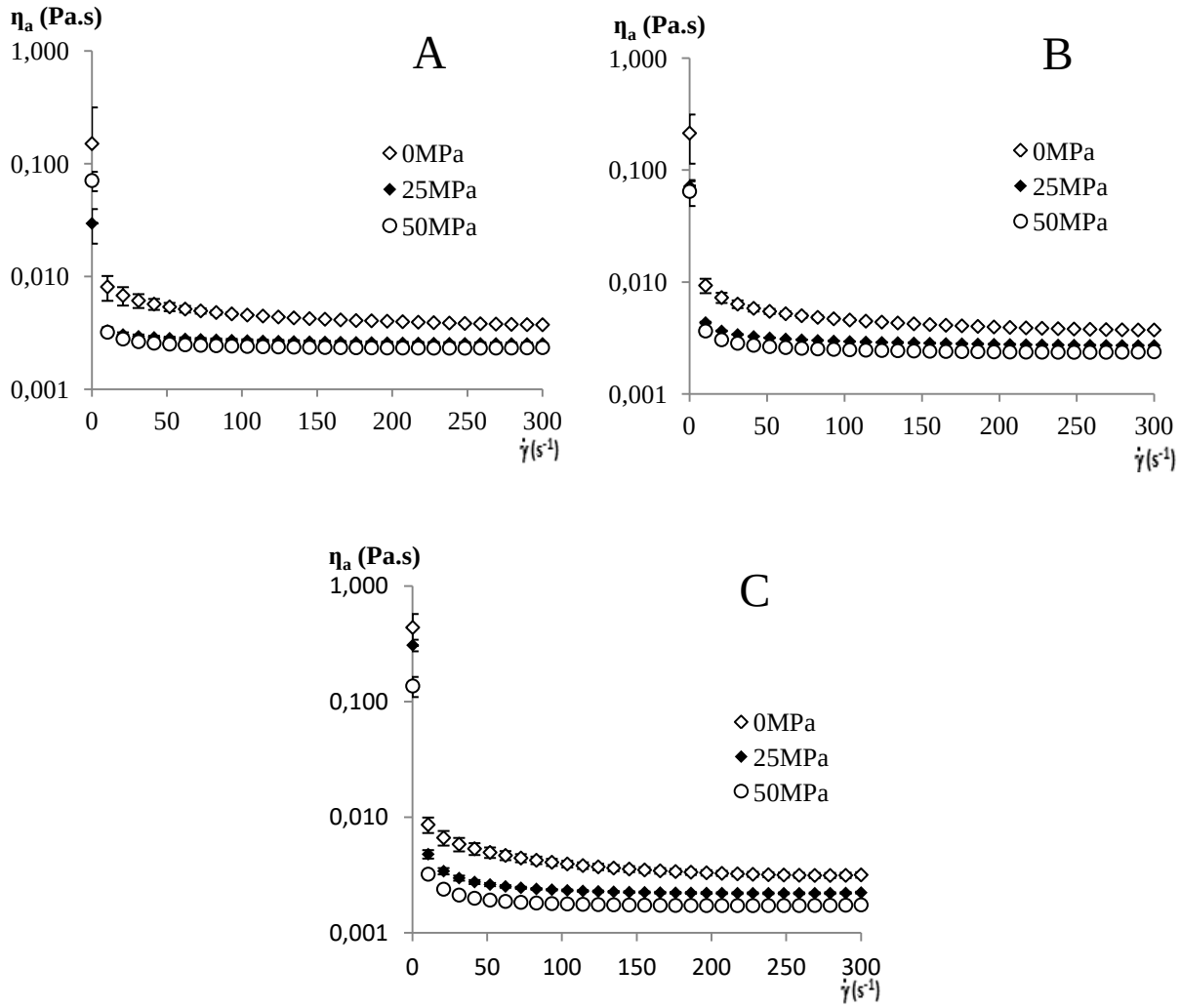

Figure 5 – Apparent viscosity at 25 °C in model systems of gellan gum with citric acid (A), malic acid (B) and tartaric acid (C) (the points are the mean values, and the vertical bars are standard deviation).

Therefore, a correlation between the rheological and sensory data is necessary for the development of new formulations and process technologies, once it can determine the functionality of an ingredient during the development, quality control, and shelf life of the products (Holdsworth, 1993).

The effect of P_H on the parameters of the Ostwald-de-Waele model can be seen in Table 3. The consistency index (*k*) decreased and the flow behavior index (*n*) increased with the increase of P_H. That is, samples with lower viscosity had a lower consistency index and

higher flow behavior index. It was observed that the model systems presented (*n*) lower values than 1, indicating fluids with pseudoplastic characteristics. The increase in P_H from 0 to 50 MPa led to decrease consistency index and increase flow behavior index, which ranged from 0.012 to 0.003 Pa.s and 0.786 to 0.946, respectively, in the malic acid model systems. It was observed that at a fixed P_H, the organic acids did not interfere in the rheological behavior of the fluid. Similar results were observed in other studies with different polysaccharides (Wang et al., 2011; Floury et al., 2002), that is, the increase in P_H led to a reduction of *k* values and an increase in the behavior index (*n*).

Research in the field of food using non-thermal technology has been applied in different products. However, the use of the high pressure homogenizer in model systems for application in fruit nectars is innovative. Thus, studies on the rheological and sensory behavior for better functionality of the thickeners and acidulants are required, which are important for product quality and process optimization (Augusto, Ibarz & Cristianini, 2012).

Table 3 – Parameters of the Ostwald-de-Waele model at 25 °C in gellan gum model systems.

P_H	Citric acid		Malic acid		Tartaric acid	
	k (Pa.s^n)	*n* (-)	*k* (Pa.s^n)	*n* (-)	*k* (Pa.s^n)	*n* (-)
0MPa	0.012 ± 0.003	0.799 ± 0.056	0.012 ± 0.001	0.786 ± 0.018	0.012 ± 0.003	0.755 ± 0.029
25MPa	0.004 ± 0.000	0.940 ± 0.008	0.004 ± 0.000	0.913 ± 0.006	0.003 ± 0.000	0.930 ± 0.003
50MPa	0.003 ± 0.000	0.966 ± 0.007	0.003 ± 0.000	0.946 ± 0.005	0.002 ± 0.000	0.963 ± 0.005

4. Conclusion

The homogenization process altered the rheological behavior of the gellan gum model systems, reducing the consistency index (*k*) and increasing the behavior index (*n*). A reduction of the apparent viscosity of the samples was also observed with the increase of P_H.

The temporal profile of the stimulus viscosity presented similarity between the homogenized samples (25 and 50 MPa). The control samples presented higher maximum intensity of the attribute viscosity, and the P_H did not affect the sensory perception of sweetness.

The P_H did not interfere in the maximum intensity of sourness of the samples containing the same organic acid. Thus, for this stimulus, similar temporal profiles were observed for the gellan gum model systems with the addition of the same organic acid.

Notation

$\dot{\gamma}$ shear rate [s^{-1}]

η_a apparent viscosity (= $\sigma/\dot{\gamma}$) [Pa·s]

σ shear stress [Pa]

k consistency index, Ostwald-de-Waele model (Eq. (1)) [$Pa \cdot s^n$]

n flow behavior index, Ostwald-de-Waele model (Eq. (1)) [–]

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Disclosure statement

The authors declare that there is no conflict of interest.

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4. Discussões Gerais

Os sistemas modelos de goma guar apresentaram similaridade nos parâmetros Timax e Ttot, independentemente das amostras controle ou homogenizadas (25 e 50 MPa) no atributo gosto ácido. O parâmetro Imax destacou-se nas amostras homogeneizadas, sendo os sistemas modelos com ácido tartárico de gosto ácido mais intenso. A viscosidade foi o atributo com maior efeito devido ao processo de homogeneização, pois os sistemas modelos controle apresentaram maior Imax para viscosidade, sendo então as amostras mais viscosas. De modo geral, entre as amostras processadas os perfis temporais foram semelhantes.

Importante destacar que os sistemas modelos de goma guar apresentaram menor percepção sensorial no Ttot entre os atributos sensoriais (gosto ácido, doce e viscosidade) avaliados, indicando menor perfil temporal destas amostras.

Nas amostras de goma gelana, a P_H não influenciou a percepção sensorial do gosto ácido no parâmetro Imax entre as amostras que foram utilizadas o mesmo ácido orgânico. Para o atributo viscosidade, a percepção temporal apresentou alteração entre as amostras controle e as homogeneizadas (25 e 50 MPa), pois durante o processamento ocorre a ruptura das ligações entre os polissacarídeos reduzindo a viscosidade. Os sistemas modelos controle obtiveram maior Imax para o atributo viscosidade. O parâmetro Ttot apresentou similaridade entre todas as amostras de goma gelana.

Os sistemas modelos de goma xantana homogeneizados (25 e 50 MPa) apresentaram maiores valores para o parâmetro Imax, demonstrando um perfil temporal mais intenso para o gosto ácido. As amostras de goma xantana destacam-se como as mais viscosas, esse comportamento está associado à concentração de espessante (0,2 %) no meio, entretanto essas amostras sofreram maior alteração na percepção da viscosidade devido ao processo de homogeneização. De modo geral, as amostras homogeneizadas apresentaram similaridade no parâmetro Imax.

Dentre os sistemas modelos avaliados, os de goma gelana e xantana demonstraram maior Timax e Ttot para o estímulo gosto ácido, ou seja, não apresentaram percepção sensorial imediata, demonstrando perfil sensorial mais prolongado. Foi observado também que o parâmetro (Imax) apresentou valores próximos de 3 a 6 em todos os sistemas modelos (goma guar, gelana e xantana) avaliados.

Os sistemas modelos controle se caracterizam com maior intensidade e, logo, indicando maior perfil temporal para o atributo viscosidade. Por outro lado, as amostras

homogeneizadas se destacam por apresentarem perfis temporais para o gosto ácido mais intenso e duradouro, enquanto que a viscosidade aparece com menor intensidade.

O perfil temporal do gosto doce foi similar em cada um dos sistemas modelos (guar, gelana e xantana) analisados. Ou seja, o processo de homogeneização e as diferentes viscosidades não alteraram a percepção sensorial do gosto doce nas amostras.

Os ácidos orgânicos avaliados não interferiram na percepção sensorial da viscosidade e nem nos parâmetros reológicos analisados. As alterações observadas no perfil temporal, com consequente mudança na percepção da viscosidade ocorrem devido ao processamento em homogeneizador de alta pressão. Portanto, através do processo de homogeneização, as propriedades reológicas foram modificadas, e diferentes viscosidades nos sistemas modelos foram obtidas, conferindo, assim, diferentes percepções sensoriais para os atributos e parâmetros avaliados.

Os sistemas modelos apresentaram comportamento reológico descrito pelo modelo de Ostwald-de-Waele. O índice de consistência (*k*) reduziu, enquanto o índice de comportamento (*n*) aumentou com a elevação da pressão de homogeneização. Ou seja, amostras com menor viscosidade apresentaram menor índice de consistência (*k*) e maior índice de comportamento (*n*). De modo geral, foi observado que as amostras apresentaram o índice de comportamento (*n*) com valores inferiores a 1, indicando que os sistemas modelos apresentaram características de fluidos pseudoplásticos. Quanto menor o valor do índice de comportamento (*n*) maior a pseudoplasticidade do sistema modelo, portanto, as amostras de goma xantana apresentaram maior comportamento pseudoplástico, seguidas das de goma gelana e de goma guar. Através das curvas de escoamento é possível observar as diferenças entre as amostras controle e as submetidas à pressão de homogeneização. Os sistemas modelos de goma gelana e goma guar apresentaram valores próximos de viscosidade aparente a $50s^{-1}$, apesar de diferentes concentrações de espessantes. Dessa forma, como observado para a viscosidade aparente, o índice de consistência (*k*) e o índice de comportamento (*n*) estão relacionados às variações de concentrações da goma e as pressões utilizadas.

As propriedades sensoriais e reológicas dos sistemas modelos foram modificadas, apresentando diferentes características sensoriais e reológicas. Portanto, esses resultados demonstram que através do perfil sensorial tempo-intensidade, do comportamento reológico e da viscosidade aparente pode-se indicar novas perspectivas na percepção da viscosidade e na intensidade dos gostos para sistemas modelos, ou até mesmo otimizar um processo existente. Assim sendo, observou-se que a modificação das características reológicas são importantes para modificar propriedades físicas e sensoriais dos alimentos.

5. Conclusões Gerais

O desenvolvimento de sistemas modelos utilizando tecnologia não térmica é inovador, assim, o homogeneizador de alta pressão apresenta-se como um potencial viável para a indústria. Essa pesquisa é importante para o setor de néctares de frutas que tem o interesse em adotar novas tecnologias para conservação e diferentes características sensoriais para o gosto ácido, doce e viscosidade.

As amostras apresentaram comportamento pseudoplástico e características temporais específicas para os atributos gosto ácido, doce e viscosidade. Independente da concentração de espessante e pressão de homogeneização utilizadas, os sistemas modelos com ácido tartárico apresentaram maior intensidade para o gosto ácido, seguidos das amostras com ácido málico e ácido cítrico. Desse modo, por exemplo, os sistemas modelos com ácido tartárico têm potencial de aplicação para produtos de néctares com características de gosto ácido mais intenso e prolongado, enquanto que os sistemas modelos de goma xantana se destacam pela maior viscosidade.

Os sistemas modelos apresentaram diferentes viscosidades, sendo as amostras processadas menos viscosas. Ou seja, as amostras menos viscosas apresentaram menor perfil temporal para o atributo viscosidade, menor viscosidade aparente, menor índice de consistência (k) e maior índice de comportamento (n). Por sua vez, os sistemas modelos com maior viscosidade apresentaram resultados opostos nos respectivos parâmetros citados.

Vale salientar a importância dos espessantes e acidulantes para a formulação de néctares de frutas, pois são capazes de exercer várias funções para melhorar a qualidade do produto final. No entanto, quando aplicados em formulações e submetidos às diferentes condições de processamento, modificam as propriedades reológicas e sensoriais. Portanto, pesquisas sobre o comportamento reológico de sistemas modelos para desenvolvimento de bebidas tipo néctares devem ser estimuladas, assim como, avaliação das condições de processo e determinação do perfil tempo-intensidade.

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APÊNDICE

TERMO DE CONSENTIMENTO LIVRE E ESCLARECIDO

Título do Projeto: Caracterização sensorial e físico-química de soluções de espessantes e acidulantes com sacarose.

- Pesquisadora responsável: Gerlândia da Silva Pereira
- Coordenadora da pesquisa: Profª. Drª. Helena Maria André Bolini
- Número do CAAE: 52934315.7.0000.5404

Você está sendo convidado (a) para participar, como voluntário (a), do projeto de pesquisa: "Caracterização sensorial e físico-química de soluções de espessantes e acidulantes com sacarose". Esse estudo será realizado pelas pesquisadoras supracitadas da Faculdade de Engenharia de Alimentos da Universidade Estadual de Campinas (FEA/UNICAMP). O Termo de Consentimento Livre e Esclarecido visa a assegurar seus direitos como participante e é elaborado em duas vias: uma que deverá ficar com você e outra com o pesquisador. Por favor, leia com atenção, aproveitando para esclarecer suas dúvidas. Se houver perguntas antes ou mesmo depois de assiná-lo, você poderá esclarecê-las com o pesquisador.

O estudo do perfil sensorial de diferentes soluções de espessantes, acidificadas com ácidos orgânicos, pode consistir numa ferramenta útil para o desenvolvimento de novos produtos com características sensoriais específicas. Portanto, o objetivo da pesquisa é avaliar diferentes soluções de espessantes acidificadas com ácidos orgânicos e adicionadas de sacarose, determinando o perfil sensorial descritivo, tempo-intensidade e caracterização físico-química.

Nos testes sensoriais, serão avaliados os parâmetros de sabor e textura. O período de participação no projeto de pesquisa será de aproximadamente 8 meses, quando será necessário comparecer ao laboratório de Ciência Sensorial do Departamento de Alimentos e Nutrição (FEA/UNICAMP) para realizar os testes sensoriais.

Sua participação como voluntário auxiliará no levantamento de características sensoriais importantes a serem consideradas durante o desenvolvimento de novos produtos. É muito improvável qualquer desconforto ou risco para você, participante dessa pesquisa, pois todos os espessantes e acidulantes estudados têm uso permitido no Brasil.

Os pesquisadores irão tratar a sua identidade com sigilo. A participação no estudo não acarretará custos para você e não será disponível nenhuma compensação financeira adicional.

Para esclarecimento sobre qualquer dúvida a respeito da pesquisa, procure os pesquisadores responsáveis pelo estudo, cujos dados de contato encontram-se abaixo:

- Gerlândia da Silva Pereira, e-mail: gerlandiasp14@gmail.com.
- Profª. Drª. Helena Maria André Bolini, e-mail: hellini@fea.unicamp.br.

Faculdade de Engenharia de Alimentos, Universidade Estadual de Campinas (FEA/UNICAMP), Departamento de Alimentos e Nutrição. Rua Monteiro Lobato, 80, CEP 13083-862, Campinas-SP, (19)3521-4084.

Além disso, qualquer denúncia e/ou reclamação referentes aos aspectos éticos dessa pesquisa podem ser esclarecidos no Comitê de Ética em Pesquisa da UNICAMP, Rua Tessália Vieira de Camargo, 126; CEP 13083-887; Campinas-SP. Telefone: (19)3521-8936; e-mail: cep@fcm.unicamp.br.

Você é livre para decidir se aceita ou não participar como voluntário dessa pesquisa, retirar seu consentimento ou interromper a sua participação a qualquer momento, sem que isso lhe cause qualquer prejuízo.

CONSENTIMENTO DA PARTICIPAÇÃO DA PESSOA COMO SUJEITO

Declaro, por meio deste termo, que concordo em participar do projeto de pesquisa intitulado “Caracterização sensorial e físico-química de soluções de espessantes e acidulantes com sacarose”. Afirmo que aceito participar por vontade própria, sem receber qualquer incentivo financeiro e com a finalidade exclusiva de colaborar com a pesquisa. Fui informado (a) dos objetivos acadêmicos do estudo e de que posso me retirar da pesquisa a qualquer momento sem sofrer qualquer prejuízo.

Campinas, ____ de _____ de _____.

Assinatura do (a) participante: _____

Assinatura da pesquisadora: _____

ANEXOS

ANEXO A – APROVAÇÃO COMITÊ DE ÉTICA

COMITÊ DE ÉTICA EM PESQUISA DA UNICAMP - CAMPUS CAMPINAS

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Caracterização sensorial e físico - química de soluções de espessantes e acidulantes com sacarose

Pesquisador: Gerlândia da Silva Pereira

Área Temática:

Versão: 2

CAAE: 52934315.7.0000.5404

Instituição Proponente: Faculdade de Engenharia de Alimentos

Patrocinador Principal: MINISTERIO DA CIENCIA, TECNOLOGIA E INOVACAO

ANEXO B – CADASTRO SISGEN

Ministério do Meio Ambiente
CONSELHO DE GESTÃO DO PATRIMÔNIO GENÉTICO

SISTEMA NACIONAL DE GESTÃO DO PATRIMÔNIO GENÉTICO E DO CONHECIMENTO TRADICIONAL ASSOCIADO

Comprovante de Cadastro de Acesso

Cadastro nº A728243

A atividade de acesso ao Conhecimento Tradicional Associado, nos termos abaixo resumida, foi cadastrada no SisGen, em atendimento ao previsto na Lei nº 13.123/2015 e seus regulamentos.

Número do cadastro:	**A728243**
Usuário:	**Gerlandia da Silva Pereira**