PATRÍCIA APARECIDA PIMENTA PEREIRA

EFEITO DOS ADITIVOS NAS PROPRIEDADES REOLÓGICAS E SENSORIAIS DE GOIABADAS FUNCIONAIS SEM ADIÇÃO DE AÇÚCAR

LAVRAS – MG

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Tese apresentada à Universidade Federal de Lavras, como parte das exigências do Programa de Pós-Graduação em Ciência dos Alimentos, para obtenção do título de Doutor.

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APROVADA em 20 de agosto de 2012.

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LAVRAS-MG

2012

Aos meus pais José e Zilda, ao meu irmão Rafael e
ao Ulisses, que me encorajaram do início ao
término do trabalho

DEDICO

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RESUMO GERAL

O desenvolvimento de produtos sem adição de açúcar semelhante aos tradicionais requer o conhecimento dos mecanismos de ação e interação dos aditivos utilizados. Diante disso, o objetivo deste trabalho foi avaliar o efeito dos aditivos nas propriedades reológicas e sensoriais de goiabadas funcionais sem adição de açúcar. Foram utilizados os seguintes aditivos: sucralose, esteviosídeo, taumatina, maltitol, polidextrose, frutooligossacarídeo, goma xantana, goma carragena, goma locusta, pectina de baixo teor de metoxilação, cloreto de cálcio e cloreto de potássio. As adições de polidextrose e de frutooligossacarídeo nas concentrações utilizadas tornam-os doces funcionais. As etapas de desenvolvimento foram as seguintes: (1) avaliação do efeito dos diferentes aditivos (sucralose, esteviosídeo, taumatina, maltitol, polidextrose, frutooligossacarídeo, goma xantana, goma carragena, goma locusta, pectina de baixo teor de metoxilação) nas propriedades reológicas da goiabada funcional sem adição de açúcar e identificação dos aditivos que influenciam essas propriedades com a finalidade de selecionar os ingredientes que provocam efeitos desejáveis; (2) avaliação da influência dos mesmos aditivos utilizados na etapa 1 sobre os atributos sensoriais das goiabadas funcionais sem adição de açúcar e sua aceitabilidade, determinando os níveis das variáveis independentes para obter produto com doçura e consistência ideal, correlacionando os atributos sensoriais com os parâmetros do perfil de textura; (3) avaliação do efeito da adição de cloreto de cálcio e cloreto de potássio no comportamento reológico de goiabadas funcionais sem a adição de açúcar, bem como a correlação dos parâmetros reológico; (4) avaliação do efeito dos agentes gelificantes (goma carragena, goma locusta e pectina de baixo teor de metoxilação) e suas concentrações nas características sensoriais e de textura de goiabadas funcionais sem adição de açúcar. Dos edulcorantes estudados, a utilização da sucralose foi favorável para a aceitabilidade das goiabadas, sendo que a utilização de níveis elevados de frutooligossacarídeo e de agentes gelificantes promoveu uma aceitabilidade tendendo ao ideal e que quanto maior a concentração dos agentes gelificantes no produto maior foi a influência destes nas propriedades reológicas e maior foi a sua aceitabilidade. Os resultados indicaram que a utilização de cloreto de cálcio ($CaCl_2$) promoveu melhorias nas características reológicas das goiabadas e que a utilização de altas concentrações de pectina de baixo teor de metoxilação (1,16% a 2,84%) não influenciou as características sensoriais e de textura, podendo utilizar a pectina em uma concentração de 2,0% na elaboração das goiabadas. A utilização das gomas locusta e carragena em concentrações variando de 0,16% a 0,41% indicou maior aceitabilidade do produto.

Palavras-chave: Edulcorantes. Agentes de corpo. Agentes gelificantes. Propriedades reológicas. Atributos sensoriais

GENERAL ABSTRACT

The development of sugar-free products similar to a traditional requires knowledge of the mechanisms of action and interaction of the additives used. Therefore, the objective of this study was to evaluate the effect of additives on rheological and sensory properties of functional sugar-free guava preserves. The following additives were used: sucralose, stevioside, thaumatin, maltitol, polydextrose, fructooligosaccharides, xanthan gum, carrageenan gum, locust bean gum, pectin of low level of methoxyl, calcium chloride and potassium chloride. Addition of fructooligosaccharides and polydextrose in, in the concentration used, make them functional. The development steps were as follows: (1) evaluating the effect of various additives (sucralose, stevioside, thaumatin, maltitol, polydextrose, fructooligosaccharides, xanthan gum, carrageenan gum, locust bean gum, pectin of low level of methoxyl) in the rheological properties of functional sugar-free guava preserves and identifying additives which influence these properties in order to select the ingredients that cause desirable effects; (2) evaluate the influence of these additives used in step 1 on the sensory attributes of functional sugar-free guava preserves and its acceptability by determining the levels of the independent variables to obtain product with ideal consistency and sweetness, correlating sensory attributes and parameters of texture profile, (3) evaluate the effect of addition of calcium chloride and potassium chloride on the rheological behavior of functional sugar-free guava preserves, as well the correlation of the rheological parameters, (4) evaluating the effect of gelling agents (carrageenan gum, locust bean gum and pectin of low level of methoxyl) and their concentrations in the sensory characteristics and texture of functional sugar-free guava preserves. With the sweeteners studied the use of sucralose was favorable for the acceptability of guava preserve, and the use of high levels of fructooligosaccharides and gelling agents has promoted an acceptability tending to the ideal and that the greater concentration of gelling agents in the product greater was the influence these in the rheological properties and greater was their acceptability. The results show that the use of calcium chloride ($CaCl_2$) promoted improvements in the rheological characteristics of guava preserve (more hardness) and that the use of high pectin concentrations of low level of methoxyl (1.16% to 2.84%) did not influence the sensory characteristics and texture, may use the pectin at a concentration of 2.0% in the preparation of guava preserve. The use of locust bean gum and carrageenan gum in concentrations ranging from 0.16% to 0.41% indicated greater acceptability of the product.

Keywords: Sweeteners. Body agents. Gelling agents. Rheological properties. Sensory attributes.

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PRIMEIRA PARTE

1 INTRODUÇÃO

Nas últimas décadas ocorreu uma mudança no hábito do brasileiro, verificando-se diminuição no consumo de produtos industrializados de frutas tipo doces de massa, doces moles e geleias (LICODIEDOFF, 2008). Entre os motivos, tem-se a preocupação com o consumo de produtos calóricos e falta de hábito dos consumidores infanto-juvenil em consumir produtos elaborados com frutas, aliado a uma preferência pelo consumo de balas e confeitos, além do aumento alarmante no diabetes, obesidade e outras enfermidades (HRACEK; GLIEMMO; CAMPOS, 2010). Com isso, houve um aumento da procura de produtos sem a adição de açúcar (ACOSTA; VÍQUEZ; CUBERO, 2008; MESQUITA et al., 2012) e produtos que tragam algum benefício para a saúde do consumidor tais como produtos adicionados de compostos bioativos e de prebióticos (FOSTER et al., 2011).

O grande desafio na elaboração de doces de frutas sem adição de açúcar é produzi-los com características físicas e sensoriais similares aos tradicionais, uma vez que, o açúcar tem papel fundamental nesses parâmetros. Por isso, o desenvolvimento de produtos sem a adição de açúcar exige a inclusão de muitos aditivos para fornecer todas as funções do açúcar, entre esses aditivos destacam-se: edulcorantes, agentes de corpo e agentes gelificantes (HRACEK; GLIEMMO; CAMPOS, 2010).

Diante disso, o objetivo geral deste estudo foi: determinar uma formulação para goiabada funcional sem adição de açúcar, selecionando os agentes gelificantes, de corpo e edulcorantes e respectivas concentrações a serem empregados na formulação da goiabada considerando a aceitação

sensorial e influência na textura. Identificar o efeito de agentes gelificantes e edulcorantes nas propriedades reológicas e sensoriais do produto, identificando qual a função e interação entre os ingredientes de modo a selecionar agentes gelificantes e edulcorantes mais adequados para a elaboração da goiabada funcional sem adição de açúcar. Sendo que os objetivos específicos deste estudo foram: avaliar o efeito da composição nas propriedades reológicas de um sistema complexo (goiabada funcional sem adição de açúcar) e identificar os ingredientes que influenciam essas propriedades selecionando aqueles que provocam melhores efeitos (artigo 1); avaliar a influência dos ingredientes da formulação de goiabadas funcionais sem adição de açúcar sobre os atributos sensoriais e a aceitabilidade desta, determinando os níveis das variáveis independentes para obter produto com doçura e consistência ideal e o estudo da correlação entre esses atributos e os parâmetros do perfil de textura (artigo 2); avaliar o efeito da adição de sais no comportamento reológico de goiabadas funcionais sem a adição de açúcar bem como correlacionar seus parâmetros reológicos (artigo 3); avaliar o efeito dos tipos de agentes gelificantes e suas concentrações nas características sensoriais e de textura de goiabadas funcionais sem adição de açúcar (artigo 4).

2 REFERENCIAL TEÓRICO

2.1 Goiaba

A goiaba é um fruto tropical que pertence ao gênero *Psidium* da família *Myrtaceae*, sendo amplamente distribuída pelas regiões tropicais e subtropicais do mundo. Acredita-se que o seu local de origem seja a América Tropical (LEMOS et al., 1995).

O Brasil é um dos maiores produtores mundiais de goiaba, junto com outros países como México, Paquistão e Índia (SATO; SANJINEZ-ARGANDOÑA; CUNHA, 2004). O Estado de São Paulo é o maior produtor de goiaba no país, com 6 mil hectares plantados, o que totaliza cerca de 70% da produção nacional (LEITE et al., 2006).

No mundo há mais de 400 cultivares de goiaba, apesar de apenas algumas poucas dezenas serem, de fato, plantadas em escala comercial. As principais cultivares de polpa vermelha são 'Paluma', 'Pedro Sato', 'Sassaoka' e 'Rica' e, entre as de polpa branca, as de maior importância econômica são 'Kumagai' e 'Ogawa' (POMMER; MURAKAMI; WATLINGTON, 2006).

A época normal de produção da goiaba encontra-se entre janeiro e março, mas por meio de práticas culturais como a poda e a irrigação, é possível realizar a colheita durante o ano todo. O índice de maturidade ideal para a colheita é reconhecido pelos produtores observando o tamanho, a consistência e a cor do fruto (SIDDIQUI; SHARMA; GUPTA, 1991).

Destaca-se não só pelo seu aroma e sabor agradáveis, como também pela sua rica composição nutricional, que apresenta teores elevados de vitamina C, minerais, fibras, β-caroteno e licopeno (QUEIROZ et al., 2007; SOARES et al., 2007).

No mercado brasileiro, as goiabas de polpa vermelha correspondem a 75% da preferência do consumidor, sendo a cor condição indispensável ao aproveitamento industrial na fabricação de goiabadas, fruta em calda, geleias, base para bebidas, sucos, sorvetes e polpa concentrada (FERREIRA et al., 2002).

A goiaba é classificada como um fruto rico em pectina e de acidez média (JACKIX, 1988; KROLOW, 2005). Tais características têm grande importância prática para a obtenção de doce de frutas, uma vez que, a pectina é o principal componente para a formação do gel e a presença de ácido é atribuída à menor dissociação das carbonilas livres nas moléculas de pectina, o que diminui a repulsão intermolecular e favorece a formação de ligações cruzadas, essenciais para a geleificação (LÖFGREN; HERMANSSON, 2007; LUTZ et al., 2009; MENEZES et al., 2009).

2.2 Doces de frutas

Ao fazer um estudo para caracterizar a agroindústria de frutas no Estado de Minas gerais, Ferraz, Silva e Vilela (2002) fizeram o mapeamento da agroindústria no Estado mostrando a localização das cidades que possuem agroindústria de frutas, bem como o número de indústrias e seus produtos, classificados como suco/polpa e doces em geral, categoria a qual engloba doces em pasta, frutas cristalizadas, desidratadas e em calda (Figura 1).

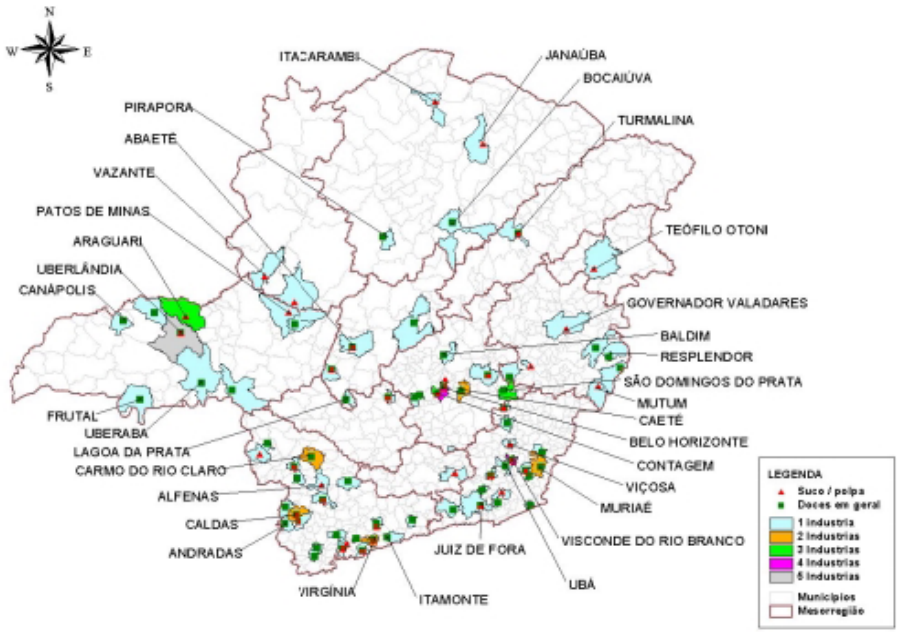


Figura 1 Mapeamento da agroindústria de frutas em Minas Gerais
Fonte: Ferraz, Silva e Vilela (2002)

Esse mapeamento mostra a forte presença da agroindústria de doces no Estado de Minas Gerais. Portanto, esse ramo de atividade pode ser muito promissor, uma vez que haja investimentos para melhorar a qualidade de tais produtos, pois a produção de doces ainda é muito baseada em métodos tradicionais e muitas vezes, rudimentares. De acordo com esses autores o segmento de produção de doces em pasta, frutas em calda e cristalizadas é caracterizado pelas micro e pequenas empresas, sendo a grande maioria com CI (capacidade instalada) menor que 500 kg.

2.2.1 Doces de frutas tradicionais

As Normas Técnicas Especiais Relativas a Alimentos e Bebidas, anexas ao decreto nº 272 de 22 de setembro de 2005, estabelecem: "doce em pasta é o produto oriundo de frutas, inteira(s), ou em parte(s) e ou semente(s), obtidas por secagem e/ou desidratação e/ou laminação e/ou fermentação, e/ou concentração, e/ou congelamento, e/ou outros processos tecnológicos considerados seguros para a produção de alimentos. Devem ser designadas por denominações consagradas pelo uso, seguidas de expressões relativa(s) ao(s) ingredientes que caracteriza(m) o produto. A designação pode ser seguida de expressões relativas ao processo de obtenção e/ou forma de apresentação e/ou característica específica" (BRASIL, 2005).

O doce em pasta pode ser simples (uma espécie) ou misto (mais de uma espécie) quanto ao vegetal empregado. Quanto à consistência pode ser cremoso (pasta homogênea e mole) ou em massa, de consistência homogênea que possibilita o corte. A designação do doce em massa é dada pelo nome da fruta acrescida do sufixo "ada" quando for elaborado com uma única espécie de fruta (BRASIL, 2005).

Segundo Policarpo et al. (2003) a produção de doces em massa, assunto amplamente estudado por Jackix (1988) implica no aquecimento da polpa e demais ingredientes por um determinado tempo, até alcançar uma concentração superior a 70º Brix, podendo afetar entre outras características, a cor, a textura e o sabor do produto final.

O tipo de açúcar apresenta importância na elaboração de doces convencionais. Na prática, geralmente, adiciona-se sacarose que é parcialmente hidrolisada durante o processo de cocção. A baixa inversão da sacarose poderá provocar cristalização, enquanto que a alta inversão poderá resultar numa

granulação de dextrose (glucose) no gel (GAVA, 1998). Dervisi, Lamb e Zabetakis (2001) em estudos com geleias de morango observaram que a diminuição da inversão da sacarose (hidrólise) provocou aumento na firmeza das geleias, pois, de acordo com esses autores, essa diminuição da hidrólise favorece a obtenção de géis mais rígidos.

2.2.2 Doces de frutas sem adição de açúcar

A demanda por alimentos nutritivos e seguros está crescendo mundialmente, e a ingestão de alimentos balanceados é a maneira correta de evitar ou mesmo corrigir problemas de saúde, como: obesidade, diabetes, desnutrição, cardiopatias, entre outros que têm origem, em grande parte, nos erros alimentares (GUTKOSKI et al., 2007).

Em cada cinco produtos lançados no mercado, pelo menos um oferece algum tipo de benefício para a saúde, desde a redução calórica até o enriquecimento com ingredientes que auxiliam na prevenção de enfermidades. Os produtos sem adição de açúcares (e consequentemente com redução de calorias) têm tido maior penetração no mercado, principalmente pela grande oferta de substitutos de açúcar que surgiram nos últimos anos (NACHTIGALL; ZAMBIAZI, 2006).

Na elaboração de doces de frutas a retirada do açúcar provoca a perda do "corpo" do produto, uma vez que, o açúcar, juntamente com a pectina, é a responsável pela formação do gel (FISZMAN, 1989; GARNIER; AXELOS; THIBAULT, 1994; LÖFGREN; HERMANSSON, 2007), fazendo com que a doçura e a textura diminuam e aumente a atividade de água do produto final (SANDROU; ARVANITOYANNIS, 2000). Por isso faz-se necessário a

utilização de vários aditivos tais como: edulcorantes, agentes de corpo, conservantes e agentes gelificantes (HRACEK; GLIEMMO; CAMPOS, 2010).

2.3 Aditivos utilizados em produtos sem adição de açúcar

Existem, no mercado, inúmeros aditivos que podem ser utilizados na elaboração de produtos sem adição de açúcar tais como sucralose, esteviosídeo e taumatina (edulcorantes); maltitol, polidextrose e frutooligossacarídeo (agentes de corpo); gomas xantana, locusta e carragena e pectina de baixo teor de metoxilação (agentes gelificantes).

2.3.1 Edulcorantes

Os edulcorantes são moléculas químicas que têm um sabor doce e uma potência muito superior à doçura da sacarose. A substituição de sacarose por edulcorantes permite produzir alimentos de baixo valor calórico com doçura semelhante aos produtos convencionais. Os edulcorantes mais utilizados são: sucralose, esteviosídeo, taumatina entre outros (SIEFARTH et al., 2011; SOUZA; BACCAN; CADORE, 2011).

As substâncias edulcorantes são consideradas não calóricas pelo fato de não serem metabolizadas pelo organismo ou por serem utilizadas em quantidades tão pequenas, que o aporte calórico torna-se insignificante. Devido a essas características são consideradas indispensáveis aos regimes dietéticos, caracterizado pelo diabetes, ou a dietas de perda ou manutenção do peso corporal (VERMUNT; SCHAAFSMA; KARDINAAL, 2003). A maioria dos edulcorantes de alta intensidade foi descoberta acidentalmente e a partir dessas descobertas foram sintetizados derivados e análogos dessas substâncias. Estudos

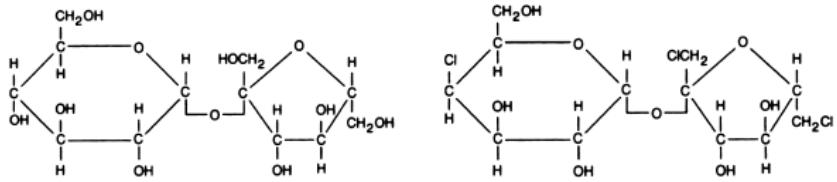
sobre a relação entre estrutura química e gosto doce têm sido conduzidos, objetivando a obtenção de substâncias com características ótimas em relação à intensidade e perfil sensorial (GRENBY, 1991).

Normas brasileiras que determinam os limites máximos dos aditivos alimentares são elaboradas com base em referências internacionais como o *Codex Alimentarius* (Norma Geral para Aditivos Alimentares - GSFA), a União Europeia e, ainda, Alimentos e Medicamentos dos EUA (*FDA*). Resolução RDC 3/2001, prevê o uso de adoçantes em alimentos, estabelecendo seus limites expressos em g/100 g ou g/100 mL do produto pronto para consumo. De acordo com essa legislação, a utilização de edulcorantes para alimentos é justificada apenas quando tenha havido redução de açúcar parcial ou total. Assim, a RDC 03/2001 aprova o uso de adoçantes em alimentos e bebidas para dietas controladas de açúcar, dietas com restrição de ingestão de açúcar, para controle de peso e informação nutricional complementar (BRASIL, 2001).

2.3.1.1 Sucralose

A sucralose (1,6-dicloro-1,6-dideoxi β-D-frutofuranosil, 4cloro-4-deoxi-α-D- galactopiranosídeo) caracteriza-se pelo sabor semelhante ao da sacarose e a ausência de residual desagradável, possuindo poder edulcorante cerca de 600 vezes o da sacarose. É obtida por processo industrial relativamente simples, mediante cloração seletiva da sacarose (Figura 2). Uma das vantagens marcantes da sucralose reside em sua notável estabilidade, tanto em altas temperaturas quanto em amplas faixas de pH (HANGER; LOTZ; LEPENIOTIS, 1996; WIET; MILLER, 1997; ZHAO; TEPPER, 2007), além disso numerosos estudos toxicológicos confirmam sua segurança (GOLDSMITH, 2000; GRICE;

GOLDSMITH, 2000; ROBERTS et al., 2000; SIMS et al., 2000; WOOD; JOHN; HAWKINS, 2000).



Sacarose Sucralose

Figura 2 Estruturas químicas da sacarose e da sucralose
Fonte: Grice e Goldsmith (2000)

De acordo com a RDC 18 de 24 de março de 2008, que dispõe sobre o Regulamento Técnico que autoriza o uso de aditivos edulcorantes em alimentos, com seus respectivos limites máximos a sucralose deve ser adicionada em alimentos somente até o limite máximo de 0,04 g/100 g (BRASIL, 2008b).

2.3.1.2 Esteviosídeo

O esteviosídeo, um edulcorante natural, é um glicosideo diterpenoide, compreendendo uma aglicona (esteviol) e três moléculas de glicose (CHATSUDTHIPONG; MUANPRASAT, 2009). É extraído das folhas de *Stevia rebaudiana*. Possui poder dulçor 150 a 300 vezes maior que a sacarose, mas apresenta forte sabor amargo residual. Tem grande aplicação na indústria alimentícia devido a sua estabilidade frente ao calor e a uma ampla faixa de pH (MIOTTO; MACHADO, 2004; TOSKULKAO; SUTHEERAWATTANANON, 1994). Além disso, é seguro para diabéticos e para fenilcetonúricos, pois não é absorvido pelo trato gastrintestinal e não possui nenhum aminoácido aromático (GRENBY, 1991). Na Figura 3 encontra-se a estrutura química do esteviosídeo.

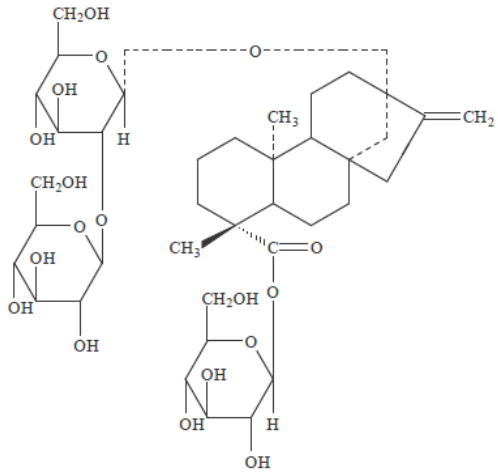


Figura 3 Estrutura química do esteviosídeo
Fonte: Alvarez e Kusumoto (1997)

O limite máximo permitido pela legislação brasileira a ser adicionado em alimentos é de 0,06 g/100g (BRASIL, 2008b).

2.3.1.3 Taumatina

A taumatina é uma proteína intensamente doce (300 a 3000 vezes mais doce que a sacarose), produzida por um planta originária da África ocidental chamada *Thaumatococcus daniellii* (MENU-BOUAOUICHE et al., 2003) e utilizada como edulcorantes em chicletes, produtos lácteos e na indústria farmacêutica (DANIELL et al., 2000). Há pelo menos cinco formas de taumatina que ocorrem naturalmente, mas são as taumatinas I e II as formas predominantes, nas quais possuem o mesmo sabor doce (DANIELL et al., 2000;

VAN DER WEL; LOEVE, 1972). É considerada a substância mais doce do planeta (DANIELL et al., 2000).

A taumatina é composta por 207 resíduos de aminoácidos e oito pontes de dissulfetos (IDE; MASUDA; KITABATAKE, 2007) sendo que segundo Coiffard, Coiffard e Roeck-Holtzhauer (1997) é instável a altas temperaturas e a baixos pH, com taxa de degradação constante de 0,14 d^{-1} a 90 °C em pH 4,0 .

Segundo Suami et al. (1997) a taumatina não é mutagênica nem carcinogênica e de acordo com a RDC 18 de 24 de março de 2008, que dispõe sobre o Regulamento Técnico que autoriza o uso de aditivos edulcorantes em alimentos, com seus respectivos limites máximos não existe limitação para a adição desse edulcorante em alimentos e bebidas (BRASIL, 2008b).

2.3.1.4 Interação entre os edulcorantes

Há muitas vantagens na utilização de combinações de edulcorantes em alimentos e bebidas tais como, melhores estabilidade e aceitação do edulcorante, uma vez que, as combinações de adoçantes muitas vezes reduz características indesejáveis, como sabor amargo além disso, a utilização de edulcorantes em combinação pode reduzir o consumo de edulcorantes, fazendo com que a quantidade de cada edulcorante utilizado permaneça abaixo da ingestão diária recomendada (IDR) e dentro dos limites legais (CÂNDIDO; CAMPOS, 1996; GAVA, 1986; LIM; SETSER; KIM, 1989; LINDLEY, 1991; PORTMANN; KILCAST, 1998).

Em estudos com doce de frutos do cerrado mistos sem adição de açúcar e com prebióticos Souza (2012) verificou que a doçura desejada pelos consumidores é conseguida com uma mistura de sucralose e acessultame-k na proporção de 3:1 e uma concentração de 0,047%. Já Menezes (2011) concluiu

que a combinação ideal de edulcorantes em goiabadas sem adição de açúcar e com prebióticos é 0,0225% de sucralose e 0,1185% de taumatina.

2.3.2 Agentes gelificantes

Os agentes gelificantes são polissacarídeos que apresentam a propriedade de reter moléculas de água, formando soluções coloidais e controlando desse modo a atividade de água de um sistema, além, de conferir ao alimento uma textura mais firme, ou seja, atuam no alimento para que o mesmo adquira a consistência de um gel (MOREIRA; CHENLO; TORRES, 2011).

Estudos indicam que os agentes gelificantes (hidrocoloides) têm papel importante na saúde humana (GLICKSMAN, 1991). Muitos trabalhos experimentais têm demonstrado que hidrocoloides, tais como carragena e goma guar, funcionam fisiologicamente como fibra solúvel, sendo bastante eficazes na redução do nível de colesterol sanguíneo (BRENNAN et al., 1996).

Dentre os mais conhecidos na elaboração de produtos alimentícios, podem ser mencionados a goma guar, goma carragena, goma gelana, pectina e a carboximetilcelulose. A goma xantana e a goma locusta são consideradas agentes gelificantes, pois sob certas condições formam géis (MOREIRA; CHENLO; TORRES, 2011; WILLIAMS, 2007).

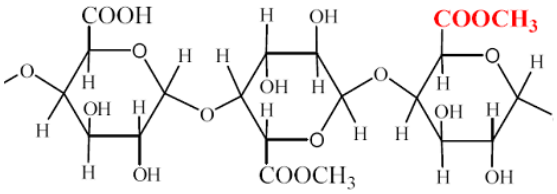
2.3.2.1 Pectina

A pectina é um ingrediente funcional de alto valor, amplamente utilizado como um agente gelificante e estabilizante na indústria de alimentos na produção de doces e geleias, sucos e produtos de confeitaria. É um abundante

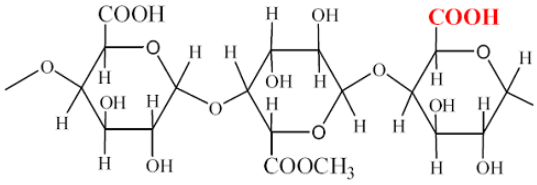
componente das paredes das células de todas as plantas terrestres (WILLATS; KNOX; MIKKELSEN, 2006).

Essa macromolécula é um polissacarídeo, constituída por domínios lineares e ramificados cujos detalhes das estruturas podem ser muito heterogêneos, mesmo dentro de uma única parede celular (NGOUÉMAZONG et al., 2012a; VORAGEN et al., 2009).

O parâmetro-chave que define as propriedades gelificantes é a grau de metoxilação (GM) (BRANDÃO; ANDRADE, 1999; CARDOSO; COIMBRA; SILVA, 2003; FISZMAN, 1989; GARNIER; AXELOS; THIBAULT, 1994; NGOUÉMAZONG et al., 2012b). Geralmente, as pectinas de alto grau de metoxilação (Figura 4a) (ATMs), com mais de 50% de grupos metoxilicos na forma esterificada, podem formar géis em condições ácidas, na presença de altos níveis de sólidos solúveis, tais como açúcares, enquanto as pectinas de baixo grau de metoxilação (BTMs) (Figura 4b), com menos de 50% de grupos metoxilicos na forma esterificada, estão associados com gelificação por cátions divalentes, geralmente cálcio (FRAEYE et al., 2010; NGOUÉMAZONG et al., 2012b; VIDECOQ et al., 2011).



(a)



(b)

Figura 4 Estruturas das pectinas de (a) alto teor de metoxilação, (b) baixo teor de metoxilação
Fonte: Licodiedoff (2008)

As BTMs são amplamente utilizadas na elaboração de produtos de baixo valor calórico (NGOUÉMAZONG et al., 2012b). A estrutura tridimensional do gel da BTM envolve sequências de dois ácidos galacturônicos dispostos paralelamente, formando a ponte íons Ca^{+2} e carboxilas livres, entrelaçando-as, estando suplementadas por pontes de hidrogênio. Zonas de junção secundárias podem surgir das pontes de hidrogênio com moléculas de água e açúcar (FISZMAN, 1989). No entanto, altas concentrações do íon cálcio podem causar formação de forças repulsivas nas zonas de junção ou ligações excessivas entre as moléculas, causando contração e gerando a sinérese, que consiste na expulsão espontânea da fase aquosa da rede do gel (FRAEYE et al., 2010; GRANT et al., 1973; WILLATS et al., 2001). De acordo com a literatura, as propriedades do gel dependem não somente do grau, mas também do padrão da metoxilação (FRAEYE et al., 2009; FRAEYE et al., 2010; POWELL et al., 1982).

Fraeye et al. (2010) estudaram a influência dos parâmetros intrínsecos e extrínsecos na textura de géis de pectina-cálcio. Esses autores observaram que a quanto menor o grau de metoxilação (GM) maior o módulo da elasticidade do gel, indicando que as pectinas com mais baixo GM geram géis mais rígidos.

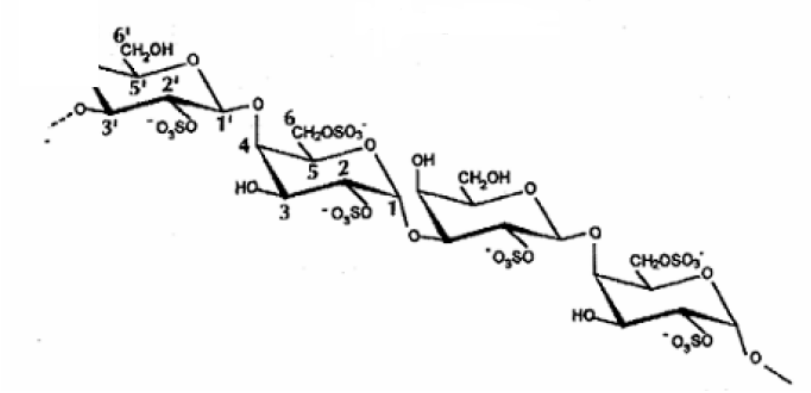
Segundo Ngouémazong et al. (2012b), os efeitos reológicos da demetoxilação nas características dos géis de pectina ainda são poucos explorados. Esses autores fizeram estudos objetivando investigar o efeito do grau e do padrão da demetoxilação sobre desenvolvimento da rede de géis da pectina-Ca^{2+}, obtendo resultados semelhantes aos encontrados por Fraeye et al. (2010).

Essas mudanças na estrutura da pectina também pode influenciar as interações da pectina com a água (EINHORN-STOLL; HATAKEYAMA; HATAKEYAMA, 2012). Molecularmente, um gel aquoso apresenta três elementos: (a) zonas de junção onde as moléculas poliméricas estão juntas, (b) segmentos de interjunção dos polímeros que são relativamente móveis, e (c) água presa na rede polimérica. Uma zona de junção pode envolver ligações covalentes, eletrostáticas, de hidrogênio ou interações hidrofóbicas (THAKUR; SINGH; HANDA, 1997).

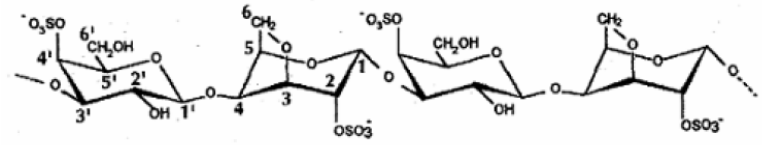
2.3.2.2 Goma Carragena

A goma carragena é um biopolímero de galactose solúvel em água, sendo classificada em três principais frações: λ-, ι- e κ-carragena (Figura 5). Possui aplicação em diversas indústrias, principalmente na farmacêutica e alimentícia. As carragenas apresentam moléculas muito flexíveis, sendo que a altas concentrações podem formar uma estrutura mais ordenada na forma de duplas hélices, a qual pode levar à formação de géis, por isso são utilizadas

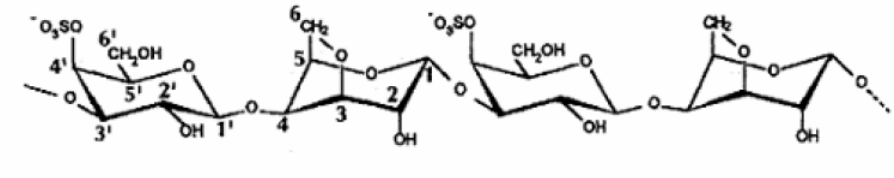
como agentes gelificantes e estabilizantes (DUNSTAN et al., 2001; SPAGNUOLO et al., 2005).



(a)



(b)



(c)

Figura 5 Ilustração das estruturas moleculares de (a) κ-carragena, (b) ι-carragena e (c) λ-carragena

Fonte: De Ruiter e Rudolph (1997)

As λ-carragenas não formam géis. Já as frações ι- e κ-carragena formam géis termorreversíveis pelo resfriamento na presença de íons cálcio ou potássio (HUANG et al., 2007).

A presença de três substituições ésteres sulfúricos (-O-$SO3^-$) (Figura 5c), responsáveis pela forte eletronegatividade dos dímeros da λ-carragena, provocam a repulsão das cadeias umas em relação às outras. Por outro lado, a disposição no espaço em forma de "zig-zag" não permite a formação de uma estrutura em hélice. As cadeias de λ-carragena permanecem dispersas na água, qualquer que seja o cátion que entre na sua constituição. Essa é a razão porque a λ-carragena não forma gel em solução aquosa, mas eleva, em contrapartida, a sua viscosidade. Esta carragena é solúvel a frio ou a baixas temperaturas (15 - 20º C) (McCANDLESS; RICHTER, 1971; PEREZ et al., 1992).

A iota carragena (ι-carragena) é vizinha da κ-carragena, mas possui um éster sulfúrico suplementar substituído, situado no carbono 2 da 3,6 anidro-α-D-galactopiranose (Figura 5b). A presença de dois ésteres sulfatados reduz o caráter hidrofóbico da ι-carragena: o gel produzido por essa carragena é mais brando e não tem sinérese (HUANG et al., 2007).

A cadeia de κ-carragena (Figura 5a) dispõe-se, no espaço, numa sucessão de hélices. Cada cadeia aproxima-se da vizinha para proteger os grupos hidrofóbicos das moléculas de água. Em consequência, a κ-carragena gelifica a solução onde se encontra (McCANDLESS; RICHTER, 1971; PEREZ et al., 1992).

Em solução aquosa, a κ-carragena tem uma conformação aleatória em espiral em altas temperaturas, mas após o resfriamento, abaixo de uma temperatura crítica, ocorre uma transição da conformação para hélice. A temperatura de transição aumenta com a concentração crescente de sal e é fortemente dependente do tipo de íons empregado. O sódio não é muito eficaz e

induz a gelificação à temperatura apenas acima de 0,2 M, ao passo que o potássio é muito eficaz com 0,01 M provoca a gelificação à temperatura ambiente. Quando géis são aquecidos derretem, e as cadeias de κ-carragena voltam para a conformação de espiral, mas isso ocorre a uma temperatura significativamente mais elevada (NONO et al., 2011).

De acordo com Montero e Pérez-Mateos (2002), a adição de sais (em concentrações e tipo) exerce uma considerável influência na gelatinização das carragenas, sendo que, dependendo da quantidade de sais adicionado, pode-se ter a formação de géis tão fortes que promovem a sinérese.

Na literatura há um grande número de estudos avaliando os efeitos desses sais nas características de géis modelos (CHEN; LIAO; DUNSTAN, 2002; IGLAUER et al., 2011; MONTERO; PÉREZ-MATEOS, 2002). Nono et al. (2011) estudaram a formação de aglomerados e separação de fases em misturas de κ-carragena sódica e caseinato de sódio. Esses autores observaram que em todas as concentrações de caseinato de sódio houve aglomerações. Eles concluiram que provavelmente isso ocorre devido às fortes ligações que ocorrem entre a κ-carragena sódica e o caseinato de sódio. Os autores também observaram que os aglomerados aumentam linearmente com o aumento da concentração de κ-carragena sódica, mas esse aumento não é dependente do aumento da concentração de caseinato de sódio. Já Chen, Liao e Dunstan (2002) utilizaram outro tipo de sal (cloreto de potássio) para avaliar a força do gel nas propriedades reológicas de géis de κ-carragena, observaram que utilizando pequenas quantidades de cloreto de potássio a força do gel de κ-carragena aumenta, havendo, portanto, forte efeito sinérgico entre eles.

Comercialmente, a carragena apresenta-se como uma mistura dos três tipos fazendo com que se tenha sinergismo entre as propriedades de cada carragena (LARA, 1993).

2.3.2.3 Goma Locusta

A goma locusta (LBG) é um polissacarídeo solúvel em água extraído do endosperma de sementes de alfarrobeira (Figura 6). É frequentemente usado nas indústrias de alimentos, farmacêuticas e de cosméticos. Atua como espessante, estabilizante de emulsões, inibidor de sinérese e apresenta estabilidade na faixa de pH de 3,5 a 11,0 (ARDA; KARA; PEKCAN, 2009). Consiste de uma rede linear de resíduos de manose que é substituído não regularmente por resíduos de galactose (WU et al., 2012). Não tem poder gelificante, mas possui fortes interações sinérgicas com outros componentes como goma carragena, goma xantana entre outros (CHRONAKIS; BORGSTRÖM; PICULELL, 1999; CHEN et al., 2001; MANDALA; SAVVAS; KOSTAROPOULOS, 2004; RAMÍREZ et al., 2002), formando géis, principalmente na presença de sais formados por íons Na^{+} e Ca^{2+} (CHRONAKIS; BORGSTRÖM; PICULELL, 1999; HIGIRO et al., 2007; VEGA; DALGLEISH; GOFF, 2005).

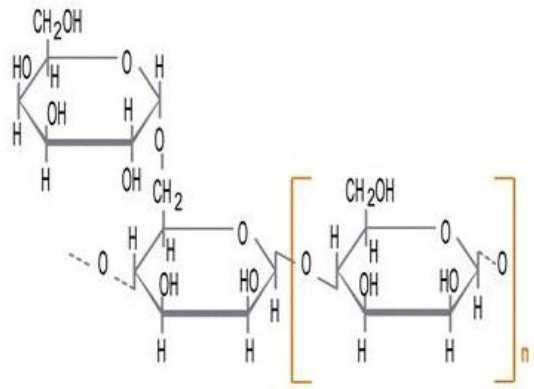


Figura 6 Estrutura molecular da goma locusta
Fonte: Chronakis, Borgström e Piculell (1999)

Efeitos sinérgicos da LBG com diferentes razões de manose/galactose com a κ-carragena foram estudadas Wu et al. (2012). Esses autores observaram que esses efeitos são mais fortes com altas razões de manose/galactose.

2.3.2.4 Goma Xantana

A goma xantana é um polissacarídeo extracelular produzido por *Xanthomonas campestris*. Ele é constituído por uma cadeia principal de unidades de D-glucose unidas entre si por ligações β-1,4, com resíduos alternados de D-manose e ácido D-glicurônico, na proporção molar de 2:1, formando a cadeia lateral, possuindo, ainda, grupos acetil e pirúvico (JANSSON; KENNE; LINDBERG, 1975). Por causa dessa estrutura, a molécula de goma xantana possui comportamento reológico pseudoplástico e é solúvel em água fria (SANDERSON, 1982). A estrutura molecular está representada na Figura 7.

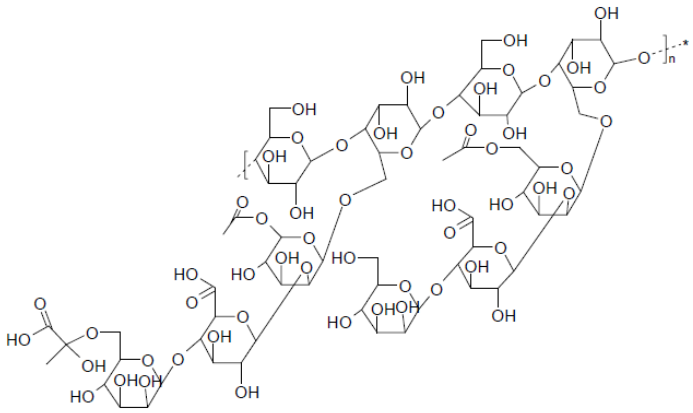


Figura 7 Estrutura molecular da goma xantana

Fonte: Sanderson (1982)

Esse biopolímero tem sido bastante utilizado em alimentos, no Brasil e no mundo, tendo sido aprovado pelo *FDA* (*Food and Drug Administration*) em 1969. Devido à grande aplicabilidade industrial da goma xantana, pesquisas vêm sendo desenvolvidas para otimizar as condições de crescimento celular, de produção, de recuperação, e de purificação desse exopolissacarídeo. Possui várias propriedades em solução, tais como aumento da viscosidade na presença de sais, que estabilizam a conformação em bastão; alta pseudoplasticidade; viscosidade elevada em repouso e baixa sob cisalhamento, como resultado de interações moleculares fracas em concentrações baixas; viscosidade estável em altas temperaturas e em ampla faixa de pH; efeito sinérgico com as gomas guar e locusta provocando aumento na viscosidade (BRANDÃO; ESPERIDIÃO; DRUZIAN, 2010; RAMÍREZ et al., 2002).

A goma xantana dispersa-se rapidamente em água fria ou quente, obtendo-se assim alta viscosidade (IGOE, 1982). Devido à rápida hidratação em diferentes meios, especialmente à temperatura ambiente, a goma xantana é muito efetiva em misturas instantâneas, onde sua função é espessar, sustentar e dar corpo aos produtos (IMESON, 1997). Ainda, é estável ao ataque enzimático e, praticamente, não é degradada pelo tratamento térmico como a esterilização (SANDERSON, 1982).

2.3.2.5 Interação entre os agentes gelificantes

Há inúmeros trabalhos que mostram as vantagens de se utilizar os agentes gelificantes misturados (ARDA; KARA; PEKCAN, 2009; DUNSTAN et al., 2001; MANDALA; SAVVAS; KOSTAROPOULOS, 2004; RAMÍREZ et al., 2002). Segundo Fernandes e Figueiredo (1995), o efeito sinérgico das

interações entre esses agentes gelificantes possui grandes aplicações tecnológicas. De acordo com Lau, Tang e Paulson (2001), a mistura de dois polímeros diferentes pode melhorar significativamente as propriedades do gel, tais como força do gel, temperatura de fusão e estabilidade à temperatura ambiente.

Arda, Kara e Pekcan (2009) concluíram que a utilização de misturas de goma carragena e goma LBG fazem com que aumentem a força do gel e a capacidade de ligação com a água, bem como modifica a textura do gel tornando-o mais elástico e resistente, mas esses efeitos ocorrem até uma determinada concentração de LBG, a partir daí há diminuição desses efeitos. Segundo esses mesmos autores esse fenômeno é chamado de pico sinérgico e que esse pico é visto com razões de 8/1 de carragena/LBG.

As interações sinérgicas observadas entre a goma locusta (LBG) e a goma xantana são principalmente governadas pelas regiões livres de galactose da cadeia de manose (FERNANDES; FIGUEIREDO, 1995). Segundo Sandolo et al. (2010) a gelificação ocorre através da ligação direta entre esses dois polímeros e não por incompatibilidade termodinâmica. Esses mesmos autores dizem que quando misturadas, dão uma rede cuja força depende da temperatura de preparação e da relação de peso entre os dois componentes.

No desenvolvimento de goiabada sem adição de açúcar e com prebiótico Menezes (2011) estudou várias concentrações de mistura de goma xantana e goma locusta (relação 1:1) e várias concentrações de pectina de baixo teor de metoxilação (0 a 1,008%) e percebeu que com o aumento da concentração dessa mistura de gomas xantana/locusta houve um aumento na dureza instrumental e na aceitabilidade do produto.

Em estudos relacionado a pectina e a carragena em sobremesas lácteas Arltoft, Madsen e Ipsen (2008) viram que a utilização desses dois agentes

gelificantes aumentam a força do gel da sobremesa láctea aumentando a sua aceitação.

2.3.3 Agentes de corpo

Agentes de corpo são compostos com propriedades de dar estrutura ao alimento, que no caso de doces e geleias sem adição de açúcar devem apresentar características similares as da sacarose: reposição de sólidos, estabilidade em diferentes condições de pH e temperatura, ausência de sabor residual e contribuir com a coloração (CAMPOS, 2000; VISSOTO; GOMES; BATISTA, 2005). Existem várias substâncias consideradas agentes de corpo, mas as mais utilizadas são o matitol, polidextrose, frutooligossacarídeo e inulina.

2.3.3.1 Maltitol

O maltitol é produzido pela hidrogenação catalítica da maltose, por isso não ocorre na natureza. Seu poder edulcorante é de aproximadamente 80 a 90% o da sacarose e seu valor calórico é de 4 $kcal.g^{-1}$. A habilidade de conferir "corpo", a solubilidade, o calor de dissolução, os efeitos de atividade de água e de depressão do ponto de congelamento são semelhantes aos da sacarose. Tem boa estabilidade térmica, química e enzimática e não deixa sensação refrescante ou sabor residual (GOMES et al., 2007). Na Figura 8 encontra-se a estrutura molecular do maltitol.

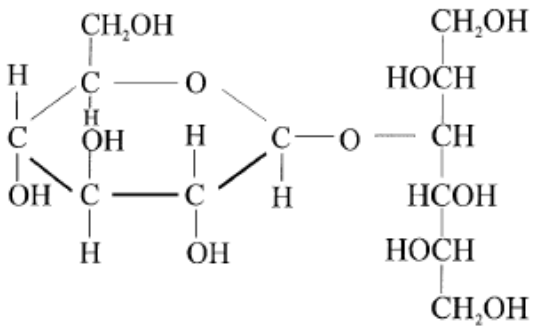


Figura 8 Estrutura molecular do maltitol
Fonte: Faivre et al. (1999)

Portmann e Kilcast (1996) caracterizando diferentes edulcorantes verificaram que é viável a utilização do maltitol na indústria de alimentos tanto como edulcorante como agente de corpo, uma vez que, esse aditivo promoveu efeitos sinérgicos entre os outros edulcorantes utilizados (ciclamato e acessulfame-K), reduzindo significativamente o sabor residual do acessulfame-K e do ciclamato e melhorou a textura dos produtos avaliados sem a adição de açúcar.

Além de conferir corpo ao produto Asvarujanon, Ishizuka e Hara (2005) verificaram que o maltitol promove um aumento na absorção de cálcio em ratos.

2.3.3.2 Polidextrose

A polidextrose (Figura 9) é um polissacarídeo sintetizado pela polimerização randômica da glicose e pode ser considerado como alimento funcional, pois é parcialmente fermentado no intestino grosso, mas não é digerido nem absorvido no intestino delgado e, em sua maior parte, é excretado

pelas fezes. Além disso, esse polímero é extremamente estável dentro de uma ampla faixa de pH, temperatura, condições de processamento e estocagem (PAUCAR-MENACHO et al., 2008). É solúvel em água e seu valor calórico é de 1 $kcal.g^{-1}$. Atua em alimentos melhorando a textura, funcionando com espessante e estabilizante, além de umectante (MONTENEGRO et al., 2008).

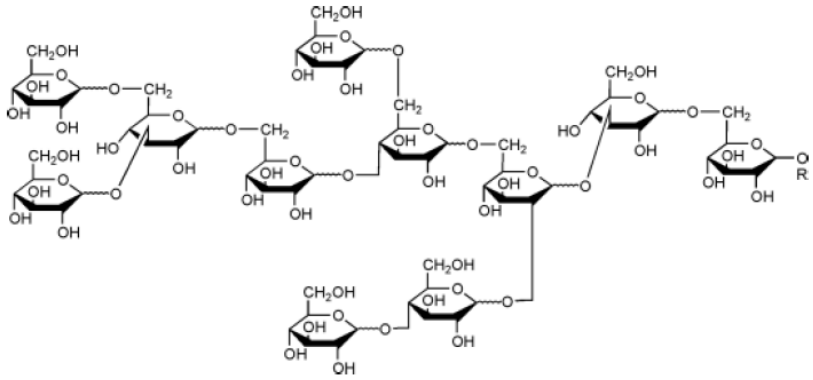


Figura 9 Estrutura molecular da polidextrose
Fonte: Craig et al. (1998)

Jie et al. (2000) demonstraram que os efeitos fisiológicos da polidextrose são concordantes com os causados pelas fibras dietárias. Segundo esses autores, em estudo com humanos, a polidextrose favoreceu a função intestinal e melhorou a facilidade de defecação. Além disso, inibiu a absorção excessiva de glicose no intestino delgado e a fermentação no intestino grosso produziu ácidos graxos de cadeia curta favorecendo a redução do pH do intestino. Portanto, a ingestão diária de 4-12 g de polidextrose melhora a função fisiológica sem produzir efeitos adversos.

Flood, Auerbach e Craig (2004) encontraram em estudo realizado em humanos, que a polidextrose não provoca diarreia em adultos com uma ingestão abaixo de 50 g por dia, mas, possivelmente, a ingestão de doses superiores pode conduzir a esse sintoma.

De acordo com a Agência Nacional de Vigilância Sanitária (BRASIL, 2008a), um produto que contenha polidextrose só poderá alegar propriedade funcional se a porção do produto pronto para consumo fornecer no mínimo 3g de polidextrose, se o alimento for sólido, ou 1,5 g, se o alimento for líquido, e que a recomendação diária de consumo do produto que contenha polidextrose não deva resultar na ingestão dessa fibra acima de 90 g ou cuja porção única de consumo resulte em ingestão de polidextrose superior a 50 g.

2.3.3.3 Frutooligossacarídeo (FOS)

Os Frutooligossacarídeos (FOS) consistem de moléculas de sacarose, nas quais uma ou duas outras unidades de frutose são adicionadas por ligações β-(2-1) à molécula de frutose da sacarose. É considerado fibra solúvel, por isso pode ser utilizado como agente de corpo, além disso, possui propriedades prebióticas, uma vez que, existe um consenso de que os FOS modificam o habitat intestinal, causando aumento no bolo fecal, normalização da frequência fecal e efeito prebiótico (aumenta o número de bactérias e/ou atividade do número de bifidobactérias e bactérias ácido-láticas, no intestino humano). Em vista disso, os frutooligossacarídeos têm sido utilizados numa ampla variedade de alimentos como iogurte, leite, queijo, leite de soja, confeitos, cereais em barra e cereais infantis (THAMER; PENNA, 2005).

Os frutooligossacarídeos (FOS) são constituintes naturais de muitos alimentos, sendo amplamente distribuídos nos produtos de origem vegetal. Esses oligossacarídeos possuem frutose como unidade monomérica principal em diferentes graus de polimerização, podendo ser produzidos industrialmente, por meio de extração, fermentação microbiana ou pela ação de enzimas. A nomenclatura FOS é dada apenas a oligômeros de frutose, que são compostos de

1-kestose (GF2), nistose (GF3) e frutofuranosil nistose (GF4) (Figura 10), em que as unidades de frutosil (F) são ligadas na posição β- 2,1 da sacarose, o que os distingue de outros oligômeros (YUN, 1996).

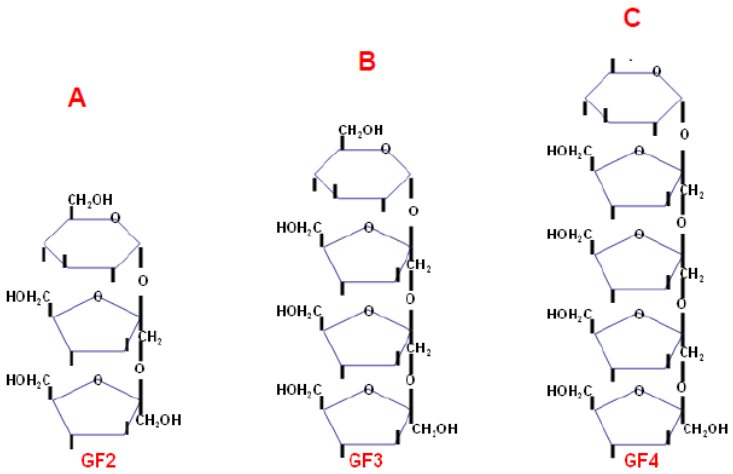


Figura 10 Estrutura química dos principais frutooligossacarídeos: 1-kestose (A), nistose (B) e frutofuranosil nistose (C)
Fonte: Passos e Park (2003)

Gomes et al. (2007) utilizaram 10% de FOS em formulação de chocolate *diet* em sacarose e observaram que as características reológicas foram semelhantes àquelas formulação com sacarose.

Assim como a polidextrose, de acordo com a Agência Nacional de Vigilância Sanitária (BRASIL, 2008a), um produto que contenha FOS só poderá alegar propriedade funcional se a porção do produto pronto para consumo fornecer no mínimo 3 g de FOS, se o alimento for sólido, ou 1,5 g, se o alimento for líquido. Ainda estabelece que o uso de FOS não deva ultrapassar 30 g na recomendação diária do produto pronto para consumo.

2.4 Alimentos funcionais

Devido aos inúmeros estudos científicos sobre a relação entre alimentação e saúde surgiu um novo segmento alimentar de rápida expansão nos últimos anos: o mercado dos alimentos funcionais. Há um interesse por parte da população em manter ou melhorar o estado de saúde por meio do consumo de alimentos tradicionais incorporados de ingredientes bioativos (RENHE et al., 2008).

São considerados alimentos funcionais aqueles que, além de fornecerem a nutrição básica, promovem a saúde. Esses alimentos possuem potencial para promover a saúde por meio de mecanismos não previstos por meio da nutrição convencional, devendo ser salientado que esse efeito restringe-se à promoção da saúde (LAJOLO, 2001; MORAES; COLLA, 2006; SAAD, 2006).

Dentre os papéis potencialmente benéficos desses alimentos funcionais, como suplemento dietético, são citados: a manutenção da microbiota intestinal, ativação do sistema imune, atividade anticarcinogênica, síntese de vitaminas do complexo B, melhora na digestão da lactose por indivíduos lactase não persistentes, modulação do colesterol sanguíneo e a melhora da biodisponibilidade de alguns minerais, entre eles o cálcio (LAJOLO, 2001; MACHADO et al., 2001).

Os vários fatores que têm contribuído para o desenvolvimento dos alimentos funcionais são inúmeros, sendo um deles o aumento da consciência dos consumidores, que desejando melhorar a qualidade de suas vidas, optam por hábitos saudáveis (MORAES; COLLA, 2006).

A Secretaria de Vigilância Sanitária do ministério da Saúde define que “alimento funcional é todo alimento ou ingrediente que, além das funções nutricionais básicas, quando consumidos na dieta usual, produz efeitos

metabólicos e/ou fisiológicos e/ou efeitos benéficos à saúde, devendo ser seguro para o consumo sem supervisão médica" (BRASIL, 2008a).

Dentre os principais campos de atuação dos alimentos funcionais destacam-se a fisiologia do trato digestivo (funções associadas à flora bacteriana, imunidade, biodisponibilidade de micronutrientes, modulação da proliferação epitelial), o sistema antioxidante (defesa contra o estress oxidativo através de determinadas vitaminas, com efeito protetor contra a aterosclerose, alguns tipos de câncer e o envelhecimento), e o metabolismo de macronutrientes (redução de efeitos patológicos decorrentes da resistência à insulina, prevenindo doença cardiovascular por reduzir a glicemia e colesterolemia) (HASLER, 2001; OHAMA; IKEDA; MORIYAMA, 2006).

Existe uma grande variedade de produtos considerados alimentos funcionais, dentre eles os frutooligossacarídeos e a polidextrose, os quais modificam a composição da microbiota colônica de tal forma que as bactérias com potencial de promoção de saúde e não patogênicas, especialmente *Lactobacilos* e *Bifidobactérias* se tornam a maioria predominante (HUEBNER; WEHLING; HUTKINS, 2007; SILVA; NÖRNBERG, 2003).

2.5 Reologia de alimentos semissólidos

Todo material sob uma força externa apresenta uma resposta entre as duas extremidades do comportamento ideal: um sólido elástico e um líquido viscoso. O primeiro é descrito pela lei de Hooke, enquanto que um líquido viscoso ideal obedece à lei de Newton (GUILLET, 2010; GUNASEKARAN; AK, 2000; RYCHLEWSKI, 1984). No entanto, a maior parte dos alimentos comporta-se como um material viscoelástico, ou seja, dependendo da tensão aplicada e da escala de tempo, um corpo sólido pode apresentar propriedades da

fase líquida e um material líquido pode mostrar propriedades de um corpo sólido. O comportamento viscoelástico de alimentos vem sendo largamente estudado em reômetros que cisalham a amostra (força tangencial), enquanto que parâmetros reológicos em tração ou compressão (força normal) vêm sendo cada vez mais utilizados na caracterização da textura de produtos alimentícios. Além disso, é possível a caracterização do produto a baixas ou altas deformações independentemente do tipo de força aplicada (ISHIHARA et al., 2011; KARAMAN et al., 2011; KUMAGAI et al., 2009; LU; ABBOTT, 1996). Por isso que a reologia é de extrema importância na indústria de alimentos, principalmente no desenvolvimento de produtos em que há substituição total ou parcial do açúcar em suas formulações (ACOSTA; VÍQUEZ; CUBERO, 2008; HRACEK; GLIEMMO; CAMPOS, 2010), exigindo um profundo entendimento da funcionalidade de ingredientes no desenvolvimento dos produtos, estudos de controle de qualidade, de vida de prateleira e determinação da textura do alimento (STEFFE, 1996).

Existem várias análises utilizadas para determinar as propriedades reológicas dos alimentos semissólidos, mas a análise de perfil de textura (TPA), teste de relaxação de tensão e teste de compressão uniaxial são as mais utilizadas.

2.5.1 Análise de perfil de textura (TPA)

As características de textura da superfície do alimento são um dos primeiros parâmetros de qualidade avaliados pelos consumidores, sendo fundamental para a aceitação do produto, mesmo antes de o mesmo ser levado à boca. A textura é composta por um conjunto de atributos sensoriais de elevada relevância, uma vez que essas influenciam ou determinam a aceitação/rejeição

do alimento (FUNAMI et al., 2012; KOTWALIWALE; BAKANE; VERMA, 2007; MOJET; KÖSTER, 2005; TANIWAKI; HANADA; SAKURAI, 2006).

A análise do perfil de textura (TPA) é um método objetivo de avaliar as propriedades sensoriais. O teste consiste em comprimir uniaxialmente o alimento (amostra em estudo) duas vezes num movimento recíproco, imitando a ação da mandíbula. Assim, durante o teste é realizada uma primeira compressão seguida por um relaxamento e uma segunda compressão. Desse teste obtém-se um gráfico: força *versus* tempo (Figura 11), do qual se calculam os parâmetros de textura (BOURNE, 2002; HERRERO et al., 2007; HONIKEL, 1998; LAU; TANG; PAULSON, 2000) estabelecidos por Friedman, Whitney e Szczesniak (1963), modificados por Bourne (1968) e apresentados por Van Vliet (1991) conforme descrito abaixo:

- Dureza (*Hardness*) (F2): força necessária para atingir uma dada deformação;
- Fracturabilidade (*Fracturability*) (F3): força com que o material fratura;
- Coesividade (*Cohesiveness*) ($A_{4:6}/A_{1:3}$): é a extensão até a qual o material pode ser distendido antes de romper irreversivelmente;
- Elasticidade (*Springiness*) ($t_{4:5}/t_{1:2}$): velocidade com que o material deformado volta a sua condição original após ser retirada a força deformante;
- Adesividade (*Adhesiviness*) ($A_{3:4}$): quantidade de força para simular o trabalho necessário para sobrepor as forças de atração entre a superfície do alimento e a superfície em contato com este;
- Gomosidade (*Gumminess*) (dureza x coesividade): energia requerida para se desintegrar um alimento semissólido ao ponto de ser engolido;
- Mastigabilidade (*Chewiness*) (elasticidade x gomosidade): energia requerida para mastigar um alimento sólido até o ponto de ser engolido;

- Resiliência (*Resilience*) ($A_{2:3}/A_{1:2}$): medida de quanto a amostra recupera de sua deformação tanto em termos de velocidade quanto em termos de suas forças derivadas.

  A resiliência é um parâmetro que não pertence à análise de perfil de textura convencional, mas vem sendo aplicado ao TPA por desenvolver um olhar mais próximo da recuperação elástica da amostra, sendo que quanto maior o comportamento elástico (propriedade de um material sólido) maior a resiliência (EXTRALAB, 2010).

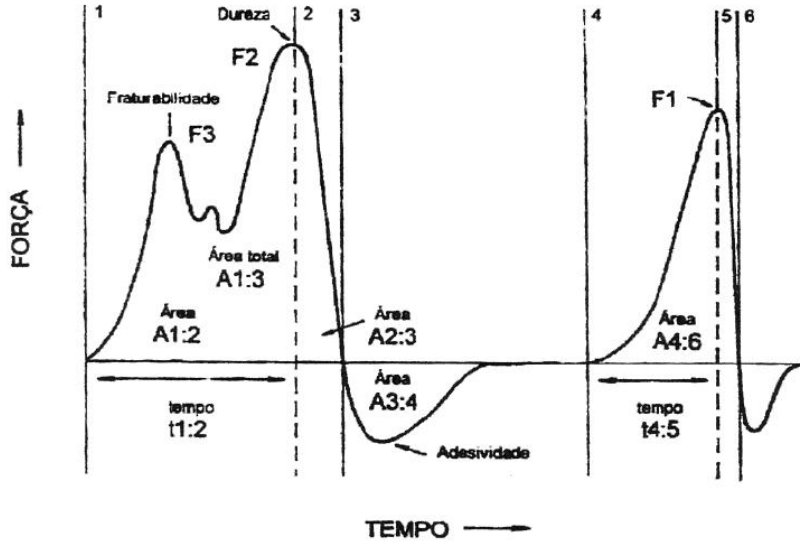


Figura 11 Típico gráfico força *versus* tempo da análise do perfil de textura
Fonte: Marques (2001)

A análise de perfil de textura simula a mastigação, exigindo assim, grandes deformações (de 20% a 50%) (HUANG et al., 2007). Essas deformações fazem com que as amostras entrem em colapso, não sendo adequado para o cálculo de alguns parâmetros, como a adesividade (PONS;

FISZMAN, 1996), uma vez que, esse parâmetro é uma característica de superfície (ADHIKARI et al., 2001; BESBES et al., 2009; HUANG et al., 2007).

Em 2006, Kealy utilizou o perfil de textura para estudar as características reológicas de vários alimentos semissólidos e observou alta correlação entre os parâmetros de dureza, coesividade e adesividade com o sabor dos alimentos, sendo que o aumento desses parâmetros do TPA diminui o sabor dos alimentos.

2.5.2 Teste de relaxação de tensão

As teorias clássicas, que descrevem o comportamento reológico de materiais idealmente elásticos não são suficientes para explicar o comportamento de muitos materiais conhecidos, entre eles os alimentos sólidos e semissólidos. A grande maioria desses alimentos apresenta características de sólidos elásticos e líquidos viscosos, podendo assim ser classificados como viscoelásticos. O comportamento desses materiais pode ser explicado por teorias de elasticidade. Sendo assim, o entendimento de conceitos básicos, como força, deformação, tensão e deformação relativa são importantes para descrever o seu comportamento reológico (RAO, 1992).

O teste de relaxação baseia-se na aplicação de uma deformação instantânea a um corpo e na manutenção dessa deformação durante todo período do teste (RAO, 1992). A forma como o corpo reage à tensão imposta é então monitorada em função do tempo como demonstrado na Figura 12 (WARD; SWEENEY, 2004). No caso de alimentos, é importante que o tempo e a deformação aplicada sejam suficientemente pequenos (na ordem de minutos, por exemplo, 10 min ou menos e de 2% a 10% de deformação), para minimizar a

ocorrência de alterações físicas no material (CUNHA, 2002; PELEG, 1987), principalmente devido à troca de umidade com o ambiente.

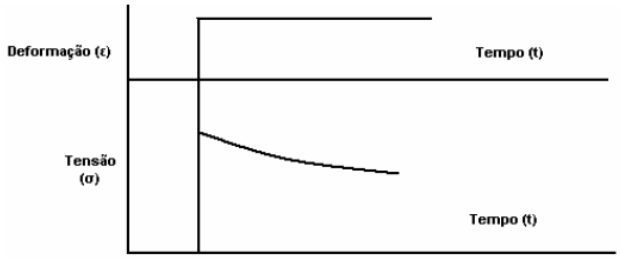


Figura 12 Curva de relaxação de tensão de polímeros semissólidos
Fonte: Ward e Sweeney (2004)

Existem vários modelos matemáticos para descrever os materiais viscoelásticos, mas os modelos de Maxwell e de Peleg são os mais utilizados para descrever o comportamento de géis em sistemas alimenticios (ANDRÉS; ZARITZKY; CALIFANO, 2008; BELLIDO; HATCHER, 2009; KAMPF; NISSINOVITCH, 1997; KHAZAEI; MOHAMMADI, 2009; MORALES et al., 2007).

2.5.2.1 Modelo de Maxwell

No modelo de Maxwell intervêm dois elementos simples que, combinados de formas distintas, representam diferentes comportamentos. Esses dois elementos são o elemento elástico ideal, que pode ser representado como uma mola e cujo comportamento é definido pela constante elástica *E,* e o elemento viscoso ideal que é representado por meio de um amortecedor e cujo comportamento é definido pela sua viscosidade η (CAMPUS et al., 2010). É

classificado por Navarro (1997) como a mais simples analogia mecânica para representar a viscoelasticidade, obtida pela associação de uma mola de módulo E em série com um amortecedor que contém um fluido de viscosidade η, representado na Figura 13. Esse modelo é comumente utilizado para predição do comportamento de relaxação da tensão, pelo fato da associação em série resultar em tensões iguais nos 2 elementos, mola e amortecedor, enquanto a deformação, ou alongamento total, é a soma das deformações nos dois elementos.

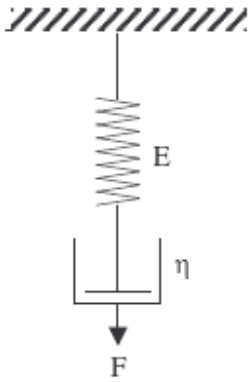


Figura 13 Representação esquemática do Modelo de Maxwell
Fonte: Costell, Fiszman e Durán (1997)

No modelo de Maxwell com uma deformação constante (ε_0), a tensão ($\sigma(t)$) após um tempo t (NOBILE et al., 2007) é dado por:

$$\sigma(t) = \varepsilon_0 \left(E.\exp(-\frac{t}{\lambda}) + E_e \right) \quad (1)$$

onde E é o módulo de elasticidade do material, E_e é o módulo de elasticidade de equilíbrio e λ é o tempo de relaxação, dado por η/E. Alguns alimentos viscoelásticos não seguem o modelo simplificado de Maxwell, necessitando de

modelos mais complexos para descrever seu comportamento. Um exemplo desse caso é o modelo de Maxwell generalizado (Figura 14), que consiste em um número infinito de modelos de Maxwell mais uma mola em paralelo.

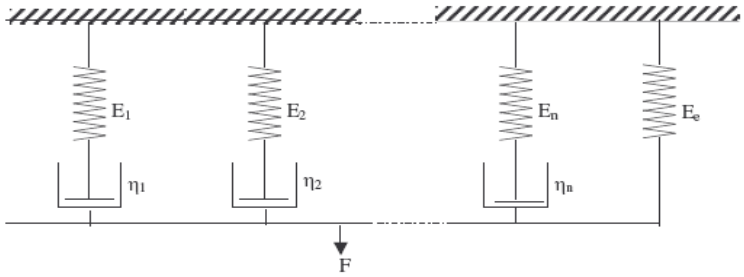


Figura 14 Representação esquemática do Modelo de Maxwell Generalizado
Fonte: Costell, Fiszman e Durán (1997)

As curvas de relaxação (tensão *versus* tempo) podem ser ajustadas por meio da equação 2, que fornece parâmetros viscoelásticos do modelo generalizado de Maxwell.

$$\sigma(t) = \varepsilon_0 \left(E_1 \exp(-\frac{t}{\lambda_1}) + E_2 \exp(-\frac{t}{\lambda_2}) + ... + E_e \right) \quad (2)$$

onde E_1, E_2... são os módulos de elasticidade do corpo elástico ideal e λ_1 e λ_2 ... são os tempos de relaxação.

A viscosidade do elemento i pode ser calculada conforme a equação 3:

$$\eta_i = E_i \lambda_i \quad (3)$$

As propriedades de elasticidade (E_e, E_1, E_2...) quantificam a rigidez do material (PELEG, 1987; RODRÍGUEZ-SANDOVAL; FERNÁNDEZ-

QUINTERO; CUVELIER, 2009). Já em relação ao tempo de relaxação (λ), Bhattacharya (2010), Campus et al. (2010) e Nobile et al. (2007) afirmam que maiores valores dessa propriedade indicam materiais mais elásticos e consequentemente mais firmes. Quanto maior os valores da viscosidade (η) maior o comportamento sólido do material (PELEG, 1987; RODRÍGUEZ-SANDOVAL; FERNÁNDEZ-QUINTERO; CUVELIER, 2009).

Rensis, Petenate e Viotto (2009) utilizaram o teste de relaxação para caracterizar reologicamente queijos tipo prato com teor reduzido de gordura após 30 e 60 dias de armazenamento refrigerado. O modelo de Maxwell generalizado utilizado neste estudo foi capaz de descrever satisfatoriamente o comportamento viscoelástico dos queijos, já que as curvas experimentais obtidas apresentaram boa correlação

Em um estudo sobre o comportamento reológico de sementes de trigo e canola, Bargale, Irudayaraj e Marquis (1995) concluíram que o módulo de elasticidade e a máxima tensão de contato foram fortemente influenciados pela umidade das sementes, sendo que esses valores variaram inversamente com o teor de umidade. O modelo de Maxwell com três termos de decaimento exponencial foi o que melhor descreveu o processo de relaxação de tensão.

2.5.2.2 Modelo de Peleg

No modelo de Peleg os dados da relaxação de tensão podem ser interpretados por dados da tensão normalizada *versus* o tempo (Figura 15), conforme a equação 4 (PELEG; NORMAND, 1983):

$$\frac{\sigma_0 t}{\sigma_0 - \sigma(t)} = k_1 + k_2 t \tag{4}$$

onde $\sigma(t)$ é a tensão no tempo t durante o teste, σ_0 é a tensão inicial, e k_1 e k_2 são constantes. O inverso de k_1 representa a taxa de decaimento inicial enquanto que k_2 é o valor hipotético da força assintótica normalizada que permanece sem relaxar (RODRÍGUEZ-SANDOVAL; FERNÁNDEZ-QUINTERO; CUVELIER, 2009; TANG; TUNG; ZENG, 1998).

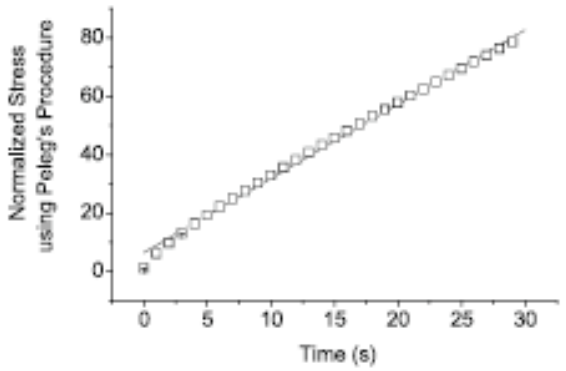


Figura 15 Representação esquemática da tensão normalizada *versus* tempo do modelo de Peleg
Fonte: Bellido e Hatcher (2009)

De acordo com Bhattacharya, Narasimha e Bhattacharya (2006), Rodríguez-Sandoval, Fernández-Quintero e Cuvelier (2009), Sozer e Dalgic (2007), Sozer, Kaya e Dalgic (2008) e Tang, Tung e Zeng (1998), a utilização do modelo de Peleg para descrever os dados de relaxação é um meio simples de descrever e comparar a relaxação de tensão com dados da literatura sobre reologia, uma vez que utiliza somente dois parâmetros: a taxa de decaimento inicial ($1/k_1$) e a tensão normalizada (k_2). O parâmetro k_1 é uma medida da facilidade com que o material se deforma, ou seja, valores mais altos k_1 sugerem um material mais duro, o qual dissipa menos energia, exigindo assim mais força

para ser comprimido (GUO; CASTELL-PEREZ; MOREIRA, 1999; RODRÍGUEZ-SANDOVAL; FERNÁNDEZ-QUINTERO; CUVELIER, 2009). Já o parâmetro k_2 representa o grau de relaxamento do material (BELLIDO; HATCHER, 2009; GUO; CASTELL-PEREZ; MOREIRA, 1999; RODRÍGUEZ-SANDOVAL; FERNÁNDEZ-QUINTERO; CUVELIER, 2009) e segundo Peleg (1980) $1/k_2$ representa as condições de equilíbrio do material, ou seja, a porção do material que permaneceu sem relaxar no estado de equilíbrio.

Bellido e Hatcher (2009) utilizaram o modelo de Peleg para apresentar os dados de relaxação de tensão de macarrão asiático e observaram que o procedimento foi eficaz com o entendimento das propriedades viscoelásticas do material.

2.5.3 Teste de compressão uniaxial

Em ensaios de compressão uniaxial utiliza uma deformação suficientemente elevada para levar à ruptura do material, com isso, é possível a caracterização do produto a baixas ou altas deformações independentemente do tipo de força aplicada (ISHIHARA et al., 2011; KARAMAN et al., 2011; KUMAGAI et al., 2009; LU; ABBOTT, 1996). No ponto de ruptura pode-se determinar as propriedades que fornecem informações sobre as características do material e correlacioná-las com a textura do produto. A tensão de ruptura (σ_{rup}) e a deformação de ruptura (deformação de Hencky – ε_{rup}), definidas respectivamente pelas Equações 5 e 6, devem ser utilizadas em ensaios de ruptura com altos valores de deformação, porque consideram as modificações que o material passa durante o experimento (BAYARRI; DURÁN; COSTELL,

2003; BAYARRI et al., 2007; COSTELL; PEYROLÓN; DURÁN, 2000; SATO; SANJINEZ-ARGANDOÑA; CUNHA, 2004):

$$\sigma = F\left(\frac{h_0 - \Delta h}{A_0 h_0}\right) \tag{5}$$

$$\varepsilon = \ln\left(\frac{h_0}{h_0 - \Delta h}\right) \tag{6}$$

Onde F é a força aplicada, h_0 a altura inicial, A_0 a área inicial da amostra e Δh a variação na altura da amostra durante a compressão.

A tensão de ruptura (σ_{rup}) é definida como a tensão necessária para romper a matriz alimentícia (CUNHA, 2002) e de acordo com Marudova e Jilov (2003) maiores tensões de ruptura pressupõe um comportamento mais rígido. Já a deformação de ruptura (deformação de Hencky – ε_{rup}) indica o quão quebradiça é a textura do alimento, isto é, até que ponto o produto pode ser deformado sem se romper (CUNHA, 2002). Materiais com altas tensão e deformação de ruptura são rígidos e fortes, enquanto que materiais com elevada tensão de ruptura, mas com baixo valor de deformação de ruptura são rígidos e quebradiços.

O módulo de elasticidade (E) e o trabalho na ruptura (W_{rup}) são calculados por meio dos gráficos σ (tensão) – ε (deformação) (Figura 16) sendo que W_{rup} é dado pela área sob a curva até o ponto de ruptura e o módulo de elasticidade, pelo coeficiente angular da parte linear inicial da curva (THYBO; NIELSEN; MARTENS, 1999).

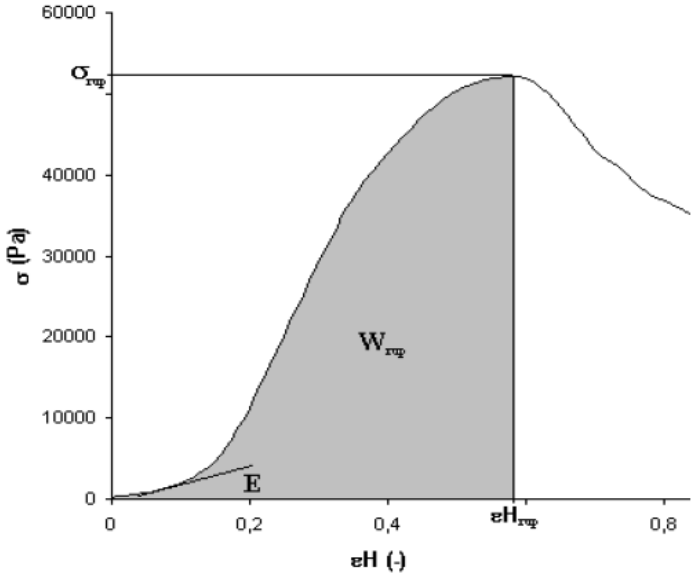


Figura 16 Representação esquemática da curva tensão *versus* deformação
Fonte: Sato, Sanjinez-Argandoña e Cunha (2004)

Géis com elevados valores de módulo de elasticidade (E) são mais rígidos (FRAEYE et al., 2010). O trabalho na ruptura é a propriedade que indica da energia necessária para induzir ruptura do gel (ROOPA; BHATTACHARYA, 2009).

Bayarri, Durán e Costell (2003) utilizaram o teste de compressão uniaxial para avaliar a relação das propriedades físicas de dois tipos de hidrocoloides e dois tipos de edulcorantes em sistemas modelo. Bayarri, Durán e Costell (2007) para avaliar a influência da textura na percepção da doçura de géis também utilizou o teste de compressão uniaxial. Por meio desse teste, esses autores conseguiram correlacionar as propriedades de textura com a percepção de doçura.

2.6 Avaliação sensorial de alimentos

A análise sensorial foi definida, em 1975, pela Divisão de Avaliação Sensorial do *Institute of Food Technologists*, da seguinte forma: "avaliação sensorial é uma disciplina científica usada para evocar, medir, analisar e interpretar reações à características de alimentos e materiais percebidas pelo sentidos da visão, olfato, paladar, tato e audição" (STONE; SIDEL, 1993).

Para que a substituição da sacarose seja aplicada com êxito é necessário, além da segurança absoluta de seus substitutos, que eles apresentem características sensoriais agradáveis, com comportamento semelhante ao da sacarose, principalmente a doçura. A única forma de se avaliar a aceitação de um produto como esse é a aplicação da análise sensorial (BATTOCHIO, 2007).

2.6.1 Teste de aceitação

A análise de aceitação possibilita a obtenção de informações importantes, refletindo o grau que os consumidores gostam ou não de um determinado produto. É muito utilizado para comparar produtos concorrentes, desenvolvimento de novos produtos e melhoria da qualidade (MATSUURA; CARDOSO; RIBEIRO, 2002; MORAES, 1988; TEIXEIRA; MEINERT; BARBETTA, 1987; UMBELINO, 2005).

As análises devem ser realizadas por uma equipe de no mínimo 25 provadores, que seja representativa do público-alvo, em um laboratório de análise sensorial (STONE; SIDEL, 1993).

Entre os métodos sensoriais existentes para medir a aceitação e preferência de um grupo de provadores, o que utiliza escala hedônica de nove pontos é o mais aplicado, devido à sua simplicidade, confiabilidade e validade

de seus resultados (MORAES, 1988; STONE; SIDEL, 1993; TEIXEIRA; MEINERT; BARBETTA, 1987).

2.6.2 Escala do ideal (*just-about-right*)

Dentre os métodos sensoriais existentes para se medir a quantidade ideal de um determinado componente a ser adicionada para provocar a melhor aceitação e preferência de um grupo de julgadores, a escala do ideal é o método afetivo mais aplicado, tanto devido à confiabilidade e validade de seus resultados como à simplicidade em ser utilizada pela equipe. Nessa análise, a equipe de provadores avalia as amostras e registra suas respostas em escala específica, mostrando o quão ideal se encontram em relação ao atributo que se deseja avaliar (por exemplo, doçura, textura, etc.), conforme o método de Vickers (1988) (CARDOSO, 2007; EPLER; CHAMBERS IV; KEMP, 1998; GACULA JÚNIOR et al., 2007).

Os dados obtidos são então submetidos à análise estatística através de histograma de distribuição das respostas sensoriais (em porcentagem) em função da concentração do componente que está variando e também por regressão linear simples entre os valores hedônicos e a concentração do componente que está variando. Com a aplicação da análise de aceitação com a escala do ideal é possível transformar dados subjetivos em objetivos, e obter informações importantes sobre a concentração adequada de um composto a ser adicionado em um alimento ou bebida, ou mesmo o tempo de cocção para se obter a textura ideal de um determinado produto (CARDOSO, 2007; CARDOSO; BATTOCHIO; CARDELLO, 2004; GACULA JÚNIOR et al., 2007).

3 CONSIDERAÇÕES FINAIS

A partir do estudo dos efeitos dos aditivos utilizados na elaboração da goiabada funcional sem adição de açúcar observou-se que em baixas concentrações (0 a 1,008%), a pectina de baixo teor de metoxilação afeta alguns parâmetros do perfil de textura e alguns parâmetros do teste de relaxação, sendo que o aumento da concentração aumenta a aceitabilidade das goiabadas. Já em altas concentrações (1,16% a 2,84%) a pectina de baixo teor de metoxilação não influencia nas características sensoriais e de textura, podendo utilizar a pectina em uma concentração de 2,0% na elaboração das goiabadas.

Em relação aos outros aditivos estudados, observou-se que maiores aceitabilidades são conseguidas utilizando níveis elevados de FOS e de agentes gelificantes (em contrações entre 0 e 0,20%), mas altas concentrações de gomas locusta e carragena essa aceitabilidade diminui, concluindo, assim, que a utilização das duas gomas variando de 0,16% a 0,41% provoca boa aceitação das goiabadas. A goma xantana não causou efeitos na aceitabilidade das goiabadas, mas provocou efeitos negativos na dureza e na gomosidade, por isso não foi empregada no estudo em altas concentrações de gomas.

Observou-se também que a utilização de maltitol, nos níveis estudados, não afeta nenhum parâmetro em estudo, sendo assim, inviável sua utilização nas goiabadas funcionais sem adição de açúcar.

Correlações positivas foram observadas entre os parâmetros do perfil de textura e os atributos sensoriais, indicando que os consumidores preferem uma goiabada mais firme e que é viável utilização de cloreto de cálcio para promover melhorias nas características de textura.

Este estudo demonstra o grande desafio de produzir goiabadas sem adição de açúcar com características semelhantes às tradicionais e a influência de cada aditivo nas propriedades reológicas e sensoriais desse produto.

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SEGUNDA PARTE- ARTIGOS

ARTIGO 1: FUNCTIONAL SUGAR-FREE GUAVA PRESERVE: INFLUENCE OF ADDITIVES ON RHEOLOGICAL PROPERTIES

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ABSTRACT

The development of sugar-free products requires the inclusion of many additives, including sweeteners, bulking agents, gelling agents and preservatives, to provide all of the characteristics of sugar products. Thus, the present study aimed to evaluate the effect of different additives (fructooligosaccharide, thaumatin, sucralose, stevioside, maltitol, xanthan gum, locust bean gum, carrageenan, low methoxyl pectin and polydextrose) on the rheological properties of functional sugar-free guava preserves. The Plackett & Burman design was used with 19 tests, including 3 trials at the midpoint, and the analyzed responses were texture profile analysis (TPA) parameters and stress relaxation test properties (using the generalized Maxwell model and Peleg model). The results were analyzed by analysis of the effects and principal component analysis. The low methoxyl pectin positively affected some parameters of the texture profile (hardness and gumminess) and relaxation test properties (η_2 and k_1). The other ingredients (except maltitol, which did not affect any rheological parameters) affected only the texture profile parameters. The hardness was positively affected by sucralose, locust bean gum and carrageenan, and it was negatively affected by fructooligosaccharide and xanthan gum. The cohesiveness was positively affected by stevioside and xanthan gum, and it was negatively affected by fructooligosaccharide, thaumatin, locust bean gum and polydextrose. The adhesiveness was positively affected by locust bean gum and negatively affected by carrageenan. The gumminess was positively affected by fructooligosaccharide, sucralose and locust bean gum, and it was negatively affected by carrageenan and xanthan gum. The results also indicated that higher concentrations of gelling agents in the product resulted in greater influences on the rheological properties observed by principal component analysis

Keywords: processing, gelling agents, sweeteners, bulking agents

1. INTRODUCTION

Humans are predisposed to like sweet foods, and this trend along with a sedentary lifestyle has led to an alarming increase in diabetes and obesity (Hracek et al., 2010). As a consequence, the demand for sugar-free products has increased (Acosta et al., 2008).

However, there are technological problems for the replacement of sugar in food systems because sugar has several important roles, such as developing the sweetness, viscosity and desired texture as well as lowering the water activity (Sandrou & Arvanitoyannis, 2000).

Guava preserves are appreciated in Brazil and result from the processing of the edible parts of healthy guavas combined with sugar; the preserves can be made with or without the addition of water, gelling agents and pH adjusters as well as other ingredients and additives used for proper consistency (Brazil, 1978). In producing this product, sugar has an important role because gelation of high methoxyl pectin only occurs in the presence of a co-solute (usually sugar in high concentrations) (Thakur et al., 1997).

For these above-mentioned reasons, the development of sugar-free products requires the inclusion of many additives, including sweeteners, bulking agents, preservatives and gelling agents, to provide all of the functions of sugar (Hracek et al., 2010).

Gelling agents include xanthan gum, carrageenan, locust bean gum (LBG) and low methoxyl pectin (LMP). Xanthan gum has a high industrial interest mainly in the food, pharmaceutical and petrochemical industries due to its physicochemical properties, particularly its high viscosity in aqueous solutions at low concentrations (0.05 to 1.0%), branched structure, high molecular weight, stability in a wide range of temperatures, pH and shear-

thinning behavior (Ramírez et al., 2002). Locust bean gum is often used in the food, pharmaceutical and cosmetic industries acting as a thickener, emulsion stabilizer and syneresis inhibitor as well as providing stability in the pH range of 3.5 to 11.0 (Arda et al., 2009). Carrageenan is a biopolymer galactose that is soluble in water and is classified into the following three main fractions: λ-, ι- and κ-carrageenan. Moreover, carrageenan molecules are flexible but can form a more ordered structure in the form of double helices at high concentrations, which may lead to gel formation; therefore, they are used as gelling agents and stabilizers (Dunstan et al., 2001; Spagnuolo et al., 2005). Low methoxyl pectin (LMP) forms a gel in the presence of divalent metal ions (typically calcium) without the requirement of sugars (Ngouémazong et al., 2012).

Guava has calcium contents ranging between 2.7 and 2.78 mg/100 g (dry basis) according to Morgano et al. (1999). Moreover, the gelling ability of carrageenan is enhanced in the presence of 0.5 g calcium/100 g (water basis) (Montero and Pérez-Mateos, 2002), and gelation of low methoxyl pectin is achieved with 0.4 g of calcium/100 g (water basis) (Beaulieu et al., 2001).

There are numerous studies that have demonstrated the advantages of using a combination of these gelling agents (Arda et al., 2009, Dunstan et al., 2001; Mandala et al., 2004, Ramirez et al., 2002). According to Arda et al. (2009), using a mixture of carrageenan and LBG causes an increase in the gel strength and binding ability with water. Moreover, this mixture changes the texture of the gel, making it more elastic and durable, but these effects occur only until a certain concentration of LBG is reached; beyond that concentration, these effects decrease. According to those authors, this phenomenon is called a synergistic peak, and this peak is observed with a carrageenan/LBG ratio of 8:1. The observed synergistic interactions between the locust bean gum (LBG) and xanthan gum are mainly governed by regions free of galactose and mannose

chains (Fernandes and Figueiredo, 1995). According to Sandolo et al. (2010), gelation occurs through a direct connection between these two polymers rather than a thermodynamic incompatibility, and this results in a net force that depends on the preparation temperature and the weight ratio between the two components.

The sweeteners used in sugar-free products should have the following characteristics: no aftertaste; low calories; ability to promote the functional properties of sucrose, including chemical stability; sweetening power equal to or higher than that of sucrose; soluble; non-toxic; not carcinogenic; and affordable (Hanger et al., 1996). Sucralose, stevioside and thaumatin are among the many sweeteners found on the market. Sucralose is characterized by a taste similar to sucrose and a lack of unpleasant aftertaste. Moreover, sucralose has a sweetness approximately 600 times that of sucrose, and it is stable at high temperatures and in a wide range of pH values (Rahn and Yaylayan, 2010). Stevioside is already a natural sweetener that has a sweetness power 150-300 times greater than sucrose, but it has a strong residual bitter taste. Stevioside has a wide application in the food industry due to its stability at high temperatures and a wide pH range (Catharino and Santos, 2011). Thaumatin is a protein that is intensely sweet (300 to 3000 times sweeter than sucrose) (Menu-Bouaouiche et al., 2003) and is used as a sweetener in chewing gum, dairy products and the pharmaceutical industry (Daniell et al., 2000).

In the preparation of sugar-free preserves, it is necessary to use bulking agents, such as maltitol, polydextrose and fructooligosaccharide (FOS). The ability of maltitol to provide "body" is similar to sucrose because it possesses good thermal stability, chemical stability and enzymatic stability. In addition, maltitol leaves no aftertaste or cooling sensation, and it has a sweetness power that is approximately 80-90% that of sucrose and their calorific value is 4 $kcalg^{-1}$

(Ronda et al., 2005). Polydextrose improves food texture and functions as a thickener and stabilizer. In addition to moist, it is extremely stable over a wide range of pH values, temperatures, processing conditions and storage conditions (Montenegro et al., 2002). Moreover, polydextrose is considered a functional food because it is fermented in the large intestine but is not digested or absorbed in the small intestine, and it is mostly excreted in feces (Paucar-Menacho et al., 2008). Fructooligosaccharide (FOS), in addition to being an agent that provides body, it is also considered a functional ingredient because it is not digested or absorbed in the small intestine and results in modification of the intestinal habitat, thereby causing an increase in stool. The normalization of stool frequency exerts a prebiotic effect (increases the number of bacteria and/or activity of the number of bifidobacteria and lactic acid bacteria in the human intestine) (Cherbut, 2002; Nyman, 2002; Roberfroid, 2007, Rodríguez-Cabezas et al., 2010; Rodrigues et al., 2011).

There have been numerous studies evaluating the effect of these components in model systems (Dunstan et al., 2001, Ramirez et al., 2002; Mandala et al., 2004; Spagnuolo et al., 2005; Arda et al., 2009; Rodrigues et al., 2011; Ngouémazong et al., 2012), but studies in complex systems, such as in fruit preserves, are scarce.

Thus, the aim of this study was to evaluate the effect of additives on the rheological properties of a complex system (functional sugar-free guava preserves) and to identify the ingredients that influence these properties.

2. MATERIALS AND METHODS

2.1 Processing of guava preserves

Ripe guavas (Pedro Sato cultivar) were used from a local market. The guavas were processed in the Pilot Plant Processing Plant Products in the Department of Food Science at the Federal University of Lavras/MG.

The fruits were washed in running water, sanitized in a 200 mg L^{-1} sodium hypochlorite solution for 15 min, selected, pulped in an electrical depulper (sieve of 6.0 mm diameter), packed in polyethylene bags of low density and frozen at -18 °C according to the methodology proposed by Menezes et al. (2009).

The following ingredients were used: fructooligosaccharide (Beneo P95), thaumatin, sucralose, stevioside, maltitol (Nutramax Catanduva, Brazil), xanthan gum, LBG, carrageenan, low methoxy pectin (LMP) (Danisco Sao Paulo, Brazil), polydextrose (Litesse Sao Paulo, Brazil), citric acid monohydrate (Nuclear São Paulo, Brazil) and potassium sorbate (Vetec Sao Paulo, Brazil).

The different formulations of guava preserves were processed in open stainless steel pots according to the methodology proposed by Menezes (2011). The mixture of pulp and polydextrose was heated to 45 °Brix, and it was then added to the gum and LMP previously homogenized under high stirring in water at 80 °C. The mixture was cooked to achieve a soluble solids content of 50 °Brix. The FOS (fructooligosaccharide) was diluted 1:1 in water at room temperature and was then added to the mixture in this step. The process of cooking continued until a total soluble solids content of 65 °Brix was obtained. Citric acid (0.2%) and potassium sorbate (0.05%) were added at the end of the cooking process (diluted 1:1 in water at room temperature) to prevent

degradation at the high temperature. The sweeteners were added in this step. The guava preserves were placed in polypropylene containers with the filling performed at a high temperature (85 °C). The containers were then closed, cooled to room temperature and stored in a BOD at 20 °C for later analysis.

No calcium was added in the preparation of the functional sugar-free guava preserves because guava fruit is rich in calcium.

2.2 Rheological properties

2.2.1 Texture profile analysis

The texture profile analyses (TPA) were performed in texturometer (Stable Micro Systems Model TA-XT2i; Goldaming, England) under the following conditions: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, post-test speed of 1.0 mm/s, distance of 20.0 mm and compression with a cylindrical probe of a 6.0 mm aluminum (Szczesniak, 1963a; Szczesniak, 1963b). The following parameters were analyzed: hardness, adhesiveness, cohesiveness and gumminess. The test was performed in triplicate. The analyses were conducted in the pots containing the guava preserves, which contained approximately 5.0 cm of preserves.

2.2.2 Stress relaxation test

There are several mathematical models that can explain the behavior of viscoelastic food products, but the Maxwell and Peleg models are used most frequently to describe the behavior of gels and alimentary systems (Kampf and

Nissinovitch, 1997; Morales et al., 2007; Andrés et al., 2008; Bellido and Hatcher, 2009; Khazaei & Mohammadi, 2009).

The Maxwell model involves two simple elements combined in a series to represent different behaviors. These two elements are the ideal elastic element, which can be represented as a spring and has a behavior defined by an elastic constant (E), and the ideal viscous element, which is represented by a dashpot and has a behavior defined by its viscosity (η) (Campus et al., 2010).

In the Maxwell model with a constant strain (ε_0), σ describes the tension applied from σ_0 for σ(t) after a time t (Nobile et al., 2007) as follows:

$$\sigma(t) = \varepsilon_0 \left(E.\exp(-\frac{t}{\lambda}) + E_e \right) \quad (1)$$

where E is the elastic modulus of the material; E_e is the equilibrium elastic modulus; and λ is the relaxation time given by η/E. Some foods do not follow the Maxwell simplified viscoelastic model. Therefore, the description of their behavior requires more complex models. An example of this case is the generalized Maxwell model, which consists of an infinite number of Maxwell models in parallel over a spring.

The stress relaxation curves (stress versus time) can be adjusted by equation 2, which provides the viscoelastic parameters of the generalized Maxwell model as follows:

$$\sigma(t) = \varepsilon_0 \left(E_1 \exp(-\frac{t}{\lambda_1}) + E_2 \exp(-\frac{t}{\lambda_2}) + ... + E_e \right) \quad (2)$$

where E_1, E_2 ... are the elastic moduli of the ideal elastic body; E_e is the equilibrium elastic modulus; and λ_1, λ_2 … are the relaxation times.

The viscosity of element i can be calculated according to equation 3 as follows:

$$\eta_i = E_i \lambda_i \tag{3}$$

In the Peleg model, stress relaxation data can be interpreted in accordance with the stress normalized according to equation 4 (Peleg & Normand, 1983) as follows:

$$\frac{\sigma_0 t}{\sigma_0 - \sigma(t)} = k_1 + k_2 t \tag{4}$$

where $\sigma(t)$ is the stress at any time during the test; σ_0 is initial relaxation stress; and k_1 and k_2 are constants. The reciprocal k_1 represents the initial decay rate, and reciprocal k_2 is the hypothetical value of the asymptotic normalized force that remains without relaxing (Rodríguez-Sandoval et al., 2009; Tang et al, 1998).

The stress relaxation test was performed in a texturometer (Stable Micro Systems Model TA-XT2i). The samples were cut into cylindrical shapes (2.0 cm in height and 2.0 cm in diameter) and compressed to 5.0% of their original height with a speed of 1.0 mm/s. The deformation was kept constant for 10.0 minutes, which allowed the stress to reach equilibrium. During that time, the relaxation of tension was measured at a rate of 1.0 measure per second. A cylindrical probe with a diameter of 7.0 cm, which had been lubricated to eliminate the influence of friction between the sample and probe, was used.

Three measurements were performed for each treatment. The nonlinear regression program R (2011) was used to determine the constants of the Maxwell model.

Determination of the Peleg model constants was also performed using the nonlinear regression program R (2011).

2.3 Experimental design and statistical analysis

Plackett & Burman design was designed to determine the effect of 10 variables. Each variable was examined in two levels as follows: -1 to +1; and the lower level to the higher level. Three repetitions were performed at the central point resulting in 16 trials with three additional tests at the central point totaling 19 tests (Table 1). This design is effective to evaluate the effect of a large number of variables using a small number of tests (Rao and Divakar, 2001; Djekrif-Dakhmouche et al., 2006; Li et al., 2009; Zhou et al., 2011). The independent variables were as follows: fructooligosaccharide (FOS), thaumatin, sucralose, stevioside, maltitol, xanthan gum, locust bean gum (LBG), carrageenan, low methoxyl pectin (LMP) and polydextrose. Table 2 shows the actual values of the independent variables.

Table 1. Levels of independent variables in the Plackett & Burman design 16.

Tests	FOS	Th	Su	Es	Ma	GXA	GLBG	CA	LMP	PO
1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	-1	1	1	-1	1	-1	-1
3	1	1	-1	1	1	-1	1	-1	1	-1
4	1	1	-1	-1	1	-1	-1	-1	-1	1
5	1	-1	1	1	-1	1	1	-1	-1	1
6	1	-1	1	-1	-1	1	-1	-1	1	-1
7	1	-1	-1	1	-1	-1	1	1	-1	-1
8	1	-1	-1	-1	-1	-1	-1	1	1	1
9	-1	1	1	1	-1	-1	-1	1	1	1
10	-1	1	1	-1	-1	-1	1	1	-1	-1
11	-1	1	-1	1	-1	1	-1	-1	1	-1
12	-1	1	-1	-1	-1	1	1	-1	-1	1
13	-1	-1	1	1	1	-1	-1	-1	-1	1
14	-1	-1	1	-1	1	-1	1	-1	1	-1
15	-1	-1	-1	1	1	1	-1	1	-1	-1
16	-1	-1	-1	-1	1	1	1	1	1	1
17	0	0	0	0	0	0	0	0	0	0
18	0	0	0	0	0	0	0	0	0	0
19	0	0	0	0	0	0	0	0	0	0

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; GLBG: LBG gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose

Table 2. Levels of real independent variables in the Plackett & Burman design 16.

Variables	Variable coded		
	-1	**0**	**1**
FOS (%)*	4.93	12.18	19.43
Thaumatin (%)*	0	0.0125	0.025
Sucralose (%)*	0	0.003	0.006
Stevioside (%)*	0	0.0045	0.009
Maltitol (%)*	0	0.025	0.05
Xanthan gum (%)*	0	0.1008	0.2016
Locust bean gum (%)*	0	0.1008	0.2016
Carrageenan (%)*	0	0.1008	0.2016
Low methoxyl pectin (%)*	0	0.504	1.008
Polydextrose (%)*	20.00	30.08	40.16

* relative to the amount of pulp added

The rheological properties of the functional sugar-free guava preserves were analyzed using Statistica software 8.0 and principal component analysis in SensoMaker software version 1.0.

3. RESULTS AND DISCUSSION

3.1 Texture profile analysis

Texture profile analysis (TPA) is a simple and rapid analytical technique that has been used extensively in the food industry. TPA is also used to some extent in the pharmaceutical industry to characterize semi-solid formulations (Thrimawithana et al., 2010). The mechanical properties, including hardness, cohesiveness, gumminess and adhesiveness, of the functional sugar-free guava preserves were derived from the force vs. time plots (Jones et al., 1996; Jones et al., 1997; Rahman & Al-Farsi, 2005; Ahmed & Ramaswamy, 2006; Kealy, 2006).

Table 3 shows the estimated effects of the texture profile analysis (TPA) of functional sugar-free guava preserves.

Table 3. Estimated effects of the texture profile analysis (TPA) of functional sugar-free guava preserves.

Factor	Hardness (N)	Adhesiveness (N.s)	Cohesiveness	Gumminess (N)
Mean	0.30*	108.18*	0.46*	0.13*
FOS	-0.14*	57.23*	-0.11**	-0.07*
Th	0.01	-7.36	-0.08*	0.01
Su	0.11*	-15.69	0.05	0.04*
St	0.00	-23.54	0.10*	0.01
Ma	-0.07	23.28	-0.02	-0.02
GXA	-0.18**	54.53*	0.12**	-0.07**
LBG	0.22**	-26.21	-0.07*	0.07*
CA	0.13*	-69.72*	0.05	0.07*
LMP	0.15*	-44.32	0.00	0.06*
PO	-0.05	37.52	-0.10*	-0.03

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; LBG: locust bean gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; *p<0.05; **p<0.01

FOS caused negative effects on the hardness, cohesiveness and gumminess parameters, and it caused a positive effect on adhesiveness (adhesiveness is a negative magnitude, so its absolute value will be used in the present study to better understand the effects of independent variables on this parameter). This effect of FOS on hardness was not expected because FOS is soluble fiber contribute to the "body" of the product making it firmer. This negative effect with respect to hardness may have been due to the addition of

water to solubilize FOS because greater amounts of FOS in the product result in greater amounts of water required to solubilize FOS and, consequently, a longer amount of time required to cook the guava preserves to obtain the same °Brix. When heated to 80 °C for a long time in an acid medium, Rasttal (2010) reported that FOS are hydrolyzed to monosaccharides and this degradation of FOS changes the product's stability in the aqueous phase making the product less firm (Glibowski & Wasko, 2008). With regard to adhesiveness, increased FOS in the guava preserves made the preserves more adhesive. According El Nagar et al. (2002), the enhancement of the adhesiveness by increasing the concentration of FOS is due to FOS making the product more viscous because more water is bound to promote the formation of a stable gel. Cohesiveness is a rheological parameter that is correlated with the property that characterizes the ability to swallow food, especially if the food is solid (Lucas et al., 2002; Ishihara et al., 2011), and cohesiveness is calculated by the ratio between the area under the curve of strength versus time in the second compression cycle and that in the first compression cycle (Bourne, 1982; Gujral et al., 2002). Lower cohesiveness values (observed for higher values of FOS) result in more disintegrated material in the first compression cycle (Extralab 2010), which means that FOS makes the product more easily to disintegrate. The gumminess (force required to chew a semi-solid food) (Oliveira et al., 2009) was negatively affected by FOS because increasing the amount of FOS caused the guava preserve to become softer and, consequently, less gummy.

For high intensity sweeteners, the concentration of stevioside and thaumatin in the product caused the cohesiveness to decrease and increase, respectively. Increased sucralose increased the hardness and gumminess of the preserves. According to the literature (Bayarri et al., 2004; Bayarri et al., 2006; Bayarri et al., 2007), this result was not expected as high intensity sweeteners do

not affect texture parameters because they are added in small quantities. This result may have been due to the chemical interactions between the sweeteners and gel networks, which may have altered the parameters of the texture profile.

Xanthan gum negatively affected the hardness and gumminess, and it positively affected the cohesiveness and adhesiveness. In studies on surimi gels, Ramírez et al. (2002) reported negative effects of xanthan gum on the mechanical properties (shear stress and shear strain) of the gels. These effects occurred because the interaction between xanthan gum, an anionic starch not associated with proteins in surimi (also anionic in nature), and surimi results in repulsion. Menezes (2011) reported the opposite result obtained for cohesiveness when evaluating the effect of a mixture of xanthan gum and locust bean gum (1:1) (addition of 0 to 0.4032%) in sugar-free guava preserves with prebiotics. According to Jeanes (1974), the properties of molecular interactions and weak gelatinization are manifested in solutions with high concentrations of xanthan gum, thereby confirming the results obtained in the present study, which demonstrated that increased xanthan gum concentration diminished the hardness and gumminess of the guava preserve.

The hardness and gumminess were positively affected by LBG, carrageenan and low methoxyl pectin (LMP). Locust bean gum (LBG) alone does not form gel (Fernandes et al., 1991), but when LBG is associated with other polysaccharides, such as carrageenan and pectin, it can form gels (Azero and Andrade, 2006; Hernandez et al., 2001; Bourbon et al., 2010). Spagnuolo et al. (2005) reported that the carrageenan molecule is flexible and may form a more ordered structure in the form of a double helices at high concentrations, which may lead to gel formation, and they reported that the gelation process is highly influenced by the following factors: type and concentration of salts in solution; cooling and heating rates; concentration of the hydrocolloid; and

presence of other biopolymers. Modifications of these factors greatly affect the gelling and rheological properties of gels (Baeza et al., 2002).

Peçanha et al. (2006) studied the microbiological, physicochemical and sensorial characteristics of guava preserves produced in the northern state of Rio de Janeiro, and they found that there is a segment of the market that prefers guava preserves with high hardness and gumminess. A similar result was obtained by Menezes (2009) when optimizing and evaluating the effects of potassium sorbate and packaging on guava preserves during storage.

Cohesiveness was negatively affected by both LBG and polydextrose indicating that increased concentrations of these at the levels studied caused a decrease in the values of cohesiveness, i.e., they caused the guava preserve to easily disintegrate. Menezes (2011) used low methoxyl pectin (0 to 1.008%), mixtures of xanthan gum and locust bean gum (1:1) (0 to 0.4032%) and polydextrose (20.0 at 40.16%) in sugar-free guava preserves with prebiotics (FOS) and observed the same behavior. The author attributed this fact to the contribution of polydextrose to the soluble solids of the product, which is a determining factor for the end of cooking, thus resulting in a weaker gel.

The adhesiveness was negatively affected by carrageenan. Increasing the concentration of carrageenan in functional sugar-free guava preserves made them less adhesive (in module) and, therefore, less sticky.

Maltitol did not influence any of the parameters studied. This result was not expected because maltitol is considered a bulking agent (Ronda et al., 2005), which would be expected to cause changes in the texture profile parameters. The lack of influence of maltitol on the parameters may have been due to the low concentration of maltitol used in the present study (0 to 0.05%).

3.2 Stress relaxation test

When a stress relaxation test is performed, different behaviors can be observed. Ideal elastic materials do not relax, and ideal viscous materials instantaneously show a relaxation over time. Viscoelastic solids gradually relax and reach an equilibrium stress greater than zero. For viscous fluids, however, the residual stress vanishes to zero. To evaluate the relaxation curve, the applied stress is separated into two components as follows: a relaxing stress component and a non-relaxing stress component. The relaxing component represents the viscous property, and the non-relaxing component represents the elastic property (Wu et al., 2011).

Generalized Maxwell model

Table 4 shows the estimated effect on the stress relaxation test using the generalized Maxwell model of functional sugar-free guava preserves.

The values obtained for the generalized Maxwell model with two elements and a spring in parallel were used for the analysis of this model (Equation 2). This model was chosen because it presented a better fit (higher R^2) than the Maxwell model (Equation 1). Furthermore, there was no a considerable improvement when the generalized Maxwell model of three elements and a spring in parallel was tested.

Table 4. Estimated effect on the stress relaxation test using the generalized Maxwell model of functional sugar-free guava preserves.

Factor	E_e (N/m^2)	E_1 (N/m^2)	λ_1 (s)	η_1 (N/m^2.s)	E_2 (N/m^2)	λ_2 (s)	η_2 (N/m^2.s)
Mean	2.55*	2.17*	5.67*	21.25*	1.71*	119.29*	354.63*
FOS	-1.11	-0.94	0.05	-8.94	-0.75	-1.96	-162.48
Th	0.77	0.68	-0.16	5.75	0.48	-3.07	89.93
Su	0.92	1.05	2.09	9.00	0.86	53.61	183.08
St	-0.23	-0.50	-0.39	-5.87	-0.39	-0.69	-77.84
Ma	0.01	-0.32	0.37	-2.04	-0.24	4.68	-33.60
GXA	-2.01	-1.73	-2.63	-17.20	-1.37	-55.90	-296.15
LBG	2.31	1.93	4.83	18.62	1.41	106.05	295.40
CA	1.46	1.41	2.78	14.44	1.18	47.68	232.01
LMP	2.48	1.63	5.55	17.98	1.45	110.64	326.79*
PO	0.46	0.22	2.56	2.82	0.32	50.06	64.24

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; LBG: locust bean gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; *$p<0.05$

Only the viscous component (η_2) was affected by an independent variable (LMP) (Table 4), which positively affected the response variable indicating that increases in the low methoxyl pectin concentration caused functional sugar-free guava preserves to behave more solid. According Macku et al. (2009), increasing the pectin concentration makes the product more rigid and, therefore, more viscous.

Peleg model

The estimated effects on the stress relaxation test using the Peleg model of functional sugar-free guava preserves is presented in Table 5.

Table 5. Estimated effects on the stress relaxation test using the Peleg model of functional sugar-free guava preserves.

Factor	k_1 (s)	k_2
Mean	28.86*	0.88*
FOS	-2.02	-0.01
Th	-1.92	-0.02
Su	14.47	0.36
St	0.54	0.05
Ma	1.60	0.03
GXA	-13.71	-0.37
LBG	23.27	0.76
CA	12.05	0.35
LMP	29.81*	0.82
PO	14.50	0.41

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; LBG: locust bean gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; *$p<0.05$

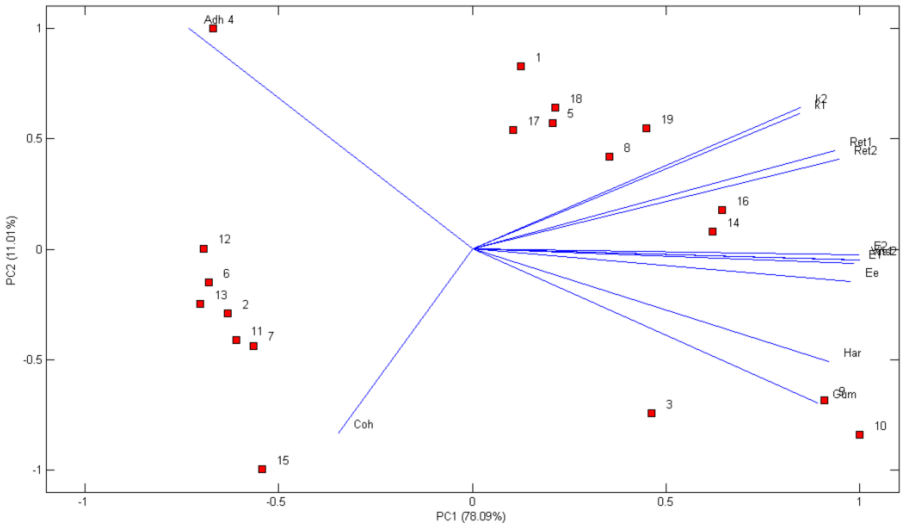
According to Tang et al. (1998), Bhattacharya (2010), Sozer & Dalgic (2007), Sozer et al. (2008) and Rodríguez-Sandoval et al. (2009), the application of Peleg's model to describe the relaxation data is a simple way to describe and compare the stress relaxation using rheology data reported in the literature because this model uses only two properties as follows: initial decay rate ($1/k_1$) and normalized stress ($1/k_2$) that would have remained unrelaxed until equilibrium. The k_1 property is a measure of the ease with which the material deforms, i.e., higher values of k_1 suggest a harder material, which dissipates less energy, thus requiring more force to be compressed (Guo et al., 1999; Rodríguez-Sandoval et al., 2009). The k_1 was positively affected by increasing

the low methoxyl pectin concentration (LMP) in the functional sugar-free guava preserves, thus resulting in a harder product. Similar behavior was observed by Oakenfull, (1987), Fiszman, (1989), Garnier et al. (1994), Ngouémazong et al. (2012) where the increase of the pectin has more rigid under study product.

The k_2 property represents the degree of relaxation of the material (Guo et al., 1999; Bellido & Hatcher, 2009; Rodríguez-Sandoval et al., 2009). According to Peleg (1980), $1/k_2$ represents the equilibrium conditions of the material, i.e., the portion of the material that remains without relaxing at equilibrium. The k_2 property was not influenced by any variable studied, which indicated that the effects of adding the independent variables to the guava preserve study did not influence the degree of relaxation of the material at a 5% significance level.

3.3 Principal components analysis

To better understand the differentiation of the tests, a study of the multivariate data was conducted. Principal components analysis was used to evaluate the results when considering the weight of all the measurements obtained experimentally. Figure 1 shows the results of the separation tests and rheological properties.



Har: hardness; Adh: adhesiveness; Coh: cohesiveness; Gum: gumminess; Ret1: relaxation time 1; Ret2: relaxation time 2; vis1: viscosity 1; vis2: viscosity 2; E_e: equilibrium elastic moduli; E_1: elastic moduli of the elastic body ideal 1; E_2: elastic moduli of the elastic body ideal 2; $1/k_1$: initial decay rate; $1/k_2$: hypothetical value of the asymptotic normalized force

Figure 1. Principal components analysis obtained for the functional sugar-free guava preserves.

The first axis (principal component 1) explained 78.09% of the variation occurring between the tests. Together, principal components 1 and 2 explained 89.10% of the variation occurring between the tests.

In this type of analysis, vectors of small size indicate that the parameters of the trials differ slightly (Cardello & Faria, 1998). Only the cohesiveness vector had reduced size compared to the other vectors indicating that the parameter vectors that have equivalent sizes are those that have similar importance to explain the variations between experiments. According to Muñoz et al. (1992), vectors with steps further from zero correspond to variations with a greater influence on the value of the major component, and vectors closer to zero correspond to a variable with little influence on the major component. Thus, all

of the attributes generated for the functional sugar-free guava preserves corresponded to variations with great influence, except for cohesiveness (close to zero).

Most of the tests were influenced by the rheological properties. Table 1 and Figure 1 show the major influence of the rheological properties of at least two types of gelling agents at levels of 0 to +1, thereby suggesting that the use of gelling agents in functional sugar-free guava preserves changes the rheological behavior of the preserves (trials 1, 3, 5, 8, 9, 10, 14, 16, 17, 18, 19).

4 CONCLUSION

The low methoxyl pectin affected several parameters of the texture profile (positive effect on hardness and gumminess) and several relaxation test propertis (positive effect on η_2 and k_1). The other ingredients (except maltitol, which did not affect any rheological parameter) affected only the texture profile parameters. Due negatives effects of the xanthan gum on hardness and gumminess and no significant effects for maltitol concludes that it is impractical to use these in developing the product. Furthermore, this study demonstrated that higher concentrations of low methoxyl pectin, locust bean gum and carrageenan in the product results in greater influences on the rheological properties.

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ARTIGO 2: THE EFFECT OF COMPOSITION ON THE SENSORY CHARACTERISTICS OF FUNCTIONAL SUGAR-FREE GUAVA PRESERVES

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ABSTRACT

The development of products without the addition of sugar requires the inclusion of many additives to provide all the functions of the sugar. These additives include sweeteners, bulking agents, preservatives and gelling agents that can alter the sensory properties and texture of the food. Therefore, the objective of this study was to evaluate the influence of the ingredients on the sensory attributes of functional sugar-free guava preserves and its acceptability by verifying which of these independent variables influence the sweetness and consistency and the ideal levels of these properties. Moreover, the objective was to correlate the sensory attributes with the parameters of texture profile and study these correlations. We used the Plackett & Burman design with 16 tests over three trials at the midpoint. The independent variables were the concentrations of fructooligosaccharide, thaumatin, sucralose, stevioside, maltitol, xanthan gum, locust bean gum, carrageenan, low methoxyl pectin and polydextrose. An acceptance test was conducted using the ideal just-about-right scale, and an internal preference map was generated for the Three-way and Pearson correlations. The results indicated that sucralose had more influence on the flavor, sweetness and overall likability. Since low methoxyl pectin increased consistency. These results suggest that the higher concentration of sucralose and the greater amount of pectin increased the acceptability of the functional sugar-free guava preserves. Further acceptability is achieved by using high levels of FOS and gelling agents, and such formulations tended to be rated as ideal. Positive correlations were observed between the parameters of the texture profile analysis (hardness, adhesiveness and gumminess) and the sensory attributes (ideal consistency and consistency), indicating that consumers prefer firmer guava preserves.

Keywords: Acceptance testing, just-about-right scale, PARAFAC, correlation

1. INTRODUCTION

The food industry has mainly relied on incremental innovation for its new product launches but is becoming increasingly aware that breakthrough, "new to the world" innovations are needed to remain competitive. The modification of food structure to generate novel flavor and texture sensations in products that provide consumers with unique eating experiences is increasing the importance of understanding the relationships among food structure, mastication, and sensory perception (Foster et al., 2011). Moreover, due to an alarming increase in diabetes, obesity and other diseases (Hracek et al., 2010), there is an increased demand for sugar-free products (Acosta et al., 2008; Mesquita et al., 2012) and products that have some benefit to consumer health such as added bioactive compounds and prebiotics products (Foster et al., 2011).

However, there are technological problems for the replacement of sugar in food systems because sugar has several important functions, such as the development of sweetness, contribution to the viscosity and desired texture, and lowering the water activity of other (Sandrou & Arvanitoyannis, 2000). Therefore, the development of sugar-free products requires the inclusion of many additives, including sweeteners, bulking agents, preservatives and gelling agents, to provide all the functions of sugar (Hracek et al., 2010).

Among the gelling agents included are xanthan gum, carrageenan, locust bean gum (LBG) and low methoxyl pectin (LMP). These agents act as thickeners, stabilizers and inhibitors of syneresis (Dunstan et al., 2001; Spagnuolo et al., 2005; Arda et al., 2009; Ngouémazong et al., 2012), and the combined use of these agents have several advantages due to synergistic effects (Dunstan et al., 2001; Ramírez et al., 2002; Mandala et al., 2004; Arda et al., 2009).

The sweeteners used in sugar-free products should have the following characteristics: no aftertaste, promote the functional properties of sucrose, chemically stable, low calorie, have sweetening power equal to or higher than that of sucrose, soluble, non-toxic or non-carcinogenic and affordable (Hanger et al., 1996). Among the many sweeteners found on the market are sucralose, stevioside, and thaumatin. Sucralose is characterized by a taste similar to sucrose and the lack of an unpleasant aftertaste, with a sweetness approximately 600 times that of sucrose and high stability at high temperatures and in a wide range of pH (Rahn & Yaylayan, 2010). Stevioside is a natural sweetener that has a sweetness 150-300 times greater than that of sucrose but has a strong bitter residual taste. Stevioside has wide applications in the food industry due to its stability against heat and in a wide pH range (Catharino & Santos, 2011). Thaumatin is a protein that is intensely sweet, 300 to 3000 times sweeter than sucrose (Menu-Bouaouiche et al., 2003) and is used as a sweetener in chewing gum, dairy products and in the pharmaceutical industry (Daniell et al., 2000).

In the preparation of sugar-free products, it is necessary also to use bulking agents such as maltitol, polydextrose and fructooligosaccharide (FOS). The ability of maltitol in giving "body" is similar to that of sucrose, and it possesses good thermal, chemical and enzymatic stability, leaves no aftertaste or cooling sensation and has a sweetness that is approximately 80-90% that of sucrose (Ronda et al., 2005). Polydextrose is active in improving the texture of foods and functions as a thickener and stabilizer, in addition to wetting. It is extremely stable over a wide range of pH, temperature, processing and storage conditions (Montenegro et al., 2008). Furthermore, it is deemed functional because it is partially fermented in the large intestine but is not digested nor absorbed in the small intestine and because it is mostly excreted via the feces (Paucar-Menacho et al., 2008). Fructooligosaccharide (FOS), in addition to

being an agent that provides body, is also considered as a functional ingredient because it is not digested or absorbed in the small intestine but modifies the intestinal habitat, thereby causing increase in stool and normalization of stool frequency via a prebiotic effect (increasing the number and/or activity of bifidobacteria and lactic acid bacteria in the human gut) (Cherbut, 2002; Nyman, 2002; Lobo et al., 2006; Roberfroid, 2007; Rodríguez-Cábezas et al., 2010; Rodriguez et al., 2011).

Fruit preserves, such as guava preserves, are very appreciated in Brazil, but their traditional consumption with sugar added is decreasing due to the factors previously reported (Menezes, 2011). Therefore, it is necessary for studies to rescue the historical importance of this type of product by coupling it with new market trends, and it is extremely important to understand the interactions between the ingredients used in the preparation of sugar-free fruit preserves as well as the influence of these ingredients in the sensory perception of the consumer because food properties such as structure, composition, appearance, size, and shape influence the masticatory function (Togashi et al., 2000; Lenfant et al., 2009), and the way food is broken down intraorally influences the perception of that food. These perceptions vary between individuals for many reasons, including age, physiological state, and eating occasion (Braxton et al., 1996; Szczesniak, 2002) and influence the textural characteristics of food (Morkore & Einen, 2003).

Therefore, the objective of this study was to evaluate the influence of the formulation ingredients (the independent variables) on the sensory attributes of functional sugar-free guava preserves and their acceptability by determining the levels of independent product for the right consistency and sweetness. Moreover, the objective was to correlate the sensory attributes with the parameters of the texture profile and study these correlations.

2. MATERIAL AND METHODS

2.1. Material

The ingredients used were as follows: fructooligosaccharides (FOS) (Beneo P95), thaumatin (Nutramax), sucralose (Nutramax), stevioside (Nutramax), maltitol (Nutramax), xanthan gum (Danisco), locust bean gum (LBG) (Danisco), carrageenan (Danisco), low methoxyl pectin (LMP) (Danisco), polydextrose (Litesse), citric acid monohydrate (Nuclear) and potassium sorbate (Vetec).

2.2. Preparation of guava preserves

We used the ripe guava cultivar Pedro Sato from the local market. These fruits were processed in the Pilot Plant Processing Plant Products, Department of Food Science, Federal University of Lavras/MG.

The fruits were washed in running water, sanitized in a solution of sodium hypochlorite at 200 mg L^{-1} for 15 minutes, selected, pulped in an electrical depulper (with a sieve 6.0 mm in diameter), packed in polyethylene bags of low density and frozen at -18 °C, according to the methodology proposed by Menezes et al. (2009).

The different formulations of guava preserves were processed in open stainless steel pots, according to the methodology proposed by Menezes (2011). The mixture of pulp and polydextrose was heated to 45 °Brix, and it was then added to the gum and LMP previously homogenized under high stirring in water at 80 °C. The mixture was cooked to achieve a soluble solids content of 50 °Brix. The FOS (fructooligosaccharide) was diluted 1:1 in water at room

temperature and was then added to the mixture in this step. The process of cooking continued until a total soluble solids content of 65 °Brix was obtained. Citric acid (0.2%) and potassium sorbate (0.05%) were added at the end of the cooking process (diluted 1:1 in water at room temperature) to prevent degradation at the high temperature. The sweeteners were added in this step. The guava preserves were placed in polypropylene containers with the filling performed at a high temperature (85 °C). The containers were then closed, cooled to room temperature and stored in a BOD at 20 °C for later analysis.

No calcium was added in the preparation of the functional sugar-free guava preserves because guava fruit is rich in calcium with calcium contents ranging between 2.7 and 2.78 mg/100 g (dry basis) according to Morgano et al. (1999). Moreover, the gelling ability of carrageenan is enhanced in the presence of 0.5 g calcium/100 g (water basis) (Montero & Pérez-Mateos, 2002), and gelation of low methoxyl pectin is achieved with 0.4 g of calcium/100 g (water basis) (Beaulieu et al., 2001). Furthermore, salt was not added to the preserves.

2.3 Affective test

The acceptance test in relation to flavor, consistency, sweetness and overall liking was conducted in a laboratory with 60 consumers evaluating the guava using the 9-point hedonic scale, where 1 = extremely dislike and 9 = extremely like (Stone & Sidel, 1985).

The just-about-right scale was also used with 60 consumers, according to Lawless & Heymann (1998), to evaluate the sweetness and consistency attributes. A nine-point mixed structured scale was used, in which (+4) represented a sweetness or consistency much stronger than the ideal, (0)

represented the ideal and (–4) represented a sweetness or consistency less strong than the ideal.

The approximately 10.0 g samples (Acosta et al., 2008) were served in 50 mL disposable cups of at room temperature, following the order of presentation proposed by Wakeling and MacFie (1995). These were coded with three digit numbers taken from a table of random numbers. The test was performed in individual booths under white light.

2.4 Texture Profile Analysis

The texture profile analyses (TPA) were performed under the following conditions: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, post-test speed of 1.0 mm/s, a distance of 20.0 mm and compression with a 6.0 mm aluminum cylindrical probe and trigger force of 5.0 g using a texturometer (Stable Micro Systems Model TA-XT2i, Goldaming, England) (Szczesniak, 1963a; Szczesniak, 1963b). The parameters analyzed were hardness, adhesiveness, cohesiveness and gumminess. The test was performed in triplicate. The analyses were conducted in the pots containing a 5.0 cm height of the guava preserves, in triplicate.

2.5 Experimental design and statistical analysis

The Plackett & Burman design was designed to determine the effect of 10 variables. Each variable was examined at two levels, -1 to +1 (the lower level to the higher level), and three repetitions were performed at the central point, resulting in 16 trials with three tests central for a total of 19 tests (Table 1). This design is effective in evaluating the effect of a large number of variables using a

small number of tests (Rao & Divakar, 2001; Djekrif-Dakhmouche et al., 2006; Li et al., 2009; Zhou et al., 2011). The independent variables were FOS, thaumatin, sucralose, stevioside, maltitol, xanthan gum, LBG gum, carrageenan, LMP pectin and polydextrose. Table 2 shows the actual values of the independent variables.

Table 1. The levels of the independent variables in the 16 trial Plackett & Burman design.

Trials	**FOS**	**Th**	**Su**	**St**	**Ma**	**GXA**	**GLBG**	**CA**	**LMP**	**PO**
1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	-1	1	1	-1	1	-1	-1
3	1	1	-1	1	1	-1	1	-1	1	-1
4	1	1	-1	-1	1	-1	-1	-1	-1	1
5	1	-1	1	1	-1	1	1	-1	-1	1
6	1	-1	1	-1	-1	1	-1	-1	1	-1
7	1	-1	-1	1	-1	-1	1	1	-1	-1
8	1	-1	-1	-1	-1	-1	-1	1	1	1
9	-1	1	1	1	-1	-1	-1	1	1	1
10	-1	1	1	-1	-1	-1	1	1	-1	-1
11	-1	1	-1	1	-1	1	-1	-1	1	-1
12	-1	1	-1	-1	-1	1	1	-1	-1	1
13	-1	-1	1	1	1	-1	-1	-1	-1	1

(...continue...)

"Table 3 Cont."

14	-1	-1	1	-1	1	-1	1	-1	1	-1
15	-1	-1	-1	1	1	1	-1	1	-1	-1
16	-1	-1	-1	-1	1	1	1	1	1	1
17	0	0	0	0	0	0	0	0	0	0
18	0	0	0	0	0	0	0	0	0	0
19	0	0	0	0	0	0	0	0	0	0

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; GLBG: LBG gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose

Table 2. The levels of the real independent variables in the 16 trial Plackett & Burman design.

Variables	**Variable coded**		
	-1	**0**	**1**
FOS (%)*	4.93	12.18	19.43
Thaumatin (%)*	0	0.0125	0.025
Sucralose (%)*	0	0.003	0.006
Stevioside (%)*	0	0.0045	0.009
Maltitol (%)*	0	0.025	0.05
Xanthan gum (%)*	0	0.1008	0.2016
Locust bean gum (%)*	0	0.1008	0.2016
Carrageenan (%)*	0	0.1008	0.2016
Low methoxyl pectin (%)*	0	0.504	1.008
Polydextrose (%)*	20.00	30.08	40.16

* relative to the amount of pulp added

To evaluate the influence of the independent variables on the sensory perception, an analysis of the effects was performed using the software Statistica 8.0. A mean test was also performed, employing the Scott-Knott test at 5% probability using SAS for Windows software, version 5.0, to evaluate the acceptability of the functional sugar-free guava preserves and a histogram analysis was performed to determine the optimum sweetness and the consistency of these guava pastes. Furthermore, to correlate the sensory parameters, an internal preference mapping was performed by PARAFAC SensoMaker software version 1.0, and to correlate the parameters of the texture profile analysis with the sensory attributes, the Pearson correlation in SAS for Windows, version 5.0 was used.

3. RESULTS AND DISCUSSION

3.1 Comparison between the samples on their acceptance parameters and the sensory evaluations of the effects of variables

Tables 3 and 4 show the analysis of variance and the mean results of the sensory attributes, respectively, obtained by evaluating the 60 consumers.

Table 3. A summary of the analysis of variance for the sensory analysis of the functional sugar-free guava preserves.

Source of Variation	Degree of freedom	Mean Square			
		Flavor	Consistency	Sweetness	Overall liking
Tests	16	86.11*	79.05*	98.15*	71.02*
Residue	2	3.57	3.36	3.53	3.05
General Average		5.09	5.47	4.85	5.09
CV (%)		37.12	33.51	38.74	34.15

* significant at 5% probability; CV: Coefficient of variation. Test 17 is the mean of trials 17, 18 and 19.

Table 4. The sensory characteristics of the functional sugar-free guava preserves.

Tests	Flavor	Consistency	Sweetness	Overall liking
1	6.62 a	4.98 c	6.40 a	5.98 a
2	6.45 a	5.32 b	6.37 a	6.10 a
3	5.30 b	6.77 a	4.97 c	5.45 b
4	3.40 d	2.62 e	3.18 e	3.00 e
5	6.67 a	6.57 a	6.52 a	6.60 a
6	6.15 a	5.72 b	5.90 b	5.87 a
7	3.75 d	4.07 d	3.45 e	3.78 d
8	5.15 c	6.22 a	4.05 d	4.78 c
9	5.68 b	6.83 a	5.75 b	5.98 a
10	6.08 a	6.60 a	5.87 b	6.18 a
11	3.10 d	5.67 b	3.52 e	4.12 c
12	3.45 d	4.68 c	3.12 e	3.60 d
13	5.85 b	4.07 d	5.75 b	5.20 b
14	5.15 b	6.23 a	4.85 c	5.53 b
15	4.27 c	4.82 c	4.07 d	4.57 c
16	3.20 d	5.93 b	3.10 e	3.87 d
17	6.06 a	5.94 b	5.85 b	5.98 a

Means followed by same letter in columns do not differ statistically among themselves by Scott-Knott test at 5% probability. Test 17 is the mean of trials 17, 18 and 19

There were differences among the analysis of variance regarding all the attributes (Table 3). The flavor attribute ranged from 3.10 (test 11) and 6.67 (test 5), and tests 1, 2, 5, 6, 10 and 17 obtained average values significantly higher than the other tests. Most of these tests had maximum levels of FOS and sucralose, which may have led to the highest acceptance. Renuka et al. (2009)

observed that there is no significant difference between juices made with sucrose and juices with 50% of the sucrose replaced by FOS. Gonzalez et al. (2011) prepared yogurt with skim and whole milks and added prebiotics (14 g kg^{-1}) and/or symbiotics (10 g kg^{-1}) and found higher notes for acceptance for the formulation with whole milk when FOS was added. In a beverage systems model, Zhao & Tepper (2007) observed higher acceptance for those beverages that were prepared with sucralose compared to those made with other sweeteners. Lower scores for the flavor attribute were observed in tests 4, 7, 11, 12 and 16 (Table 4). Most of these tests use thaumatin in combination with another sweetener without sucralose and use various types of gelling agents at high concentrations. According to Coiffard et al. (1997), thaumatin is unstable at low pH and high temperatures and has a degradation rate constant of 0.14 d^{-1} at 90 °C at pH 4.0 (close to the pH of guava preserve). This degradation could be responsible for the low acceptance related to the presence of thaumatin. As thaumatin comprises approximately 207 amino acid residues and eight disulfide bridges (Ide et al., 2007), when degraded it can release these compounds in the system so that it loses its sweetening property. The low flavor acceptance in these tests also may have been due to the high concentrations of the gelling agents because according to Wilson & Brown (1997), Costell et al., (2000) and Bayarri et al (2007), the higher concentration of these agents alters the perception of flavor of the food, and according Bayarri et al. (2004) and Bayarri et al. (2006), the concentration of gelling agents modifies the mechanical properties of the gels, influencing the perception of flavor.

The most widely accepted tests for the attribute of consistency were 3, 5, 8, 9, 10 and 14, and trial 4 was less than acceptable. The greater acceptability occurred in the presence of two gums or at least one gum and pectin (Table 4). The lower acceptability of test 4 was due to the lack of gelling agents in the

system, which led to a lower consistency. This result is in agreement with Oliveira et al. (2009), who studied the use of banana peel in the development of banana preserve mass and found that consumers prefer a sweeter product with more consistency.

Tests 1, 2 and 5 had higher notes for the attribute of sweetness, and tests 4, 7, 11, 12, and 16 had the lowest notes for that attribute. It is observed that the most accepted tests have high levels of FOS and at least two types of sweeteners at higher levels, showing that consumers prefer a product with greater sweetness. Katapodis et al. (2003) and Mabel et al. (2008) argue that the FOS is considered a low-intensity sweetener and may interact synergistically with high intensity sweeteners, thereby promoting greater sweetness. In mango, pineapple and orange juices, Renuka et al. (2009) found that the replacement of sucrose by FOS (in quantities of 3.79 g/100 mL, 3.45 g/100 mL and 3.62 g/100 ml in mango, pineapple and orange juices, respectively) does not affect the acceptability of the products. For tests with lower notes of sweetness, it was observed that FOS was at the highest level (+1) with only one other sweetener or there was an absence of FOS and the presence of two sweeteners at the highest level (+1). These lower notes can be attributed to a low sweetening power caused by the presence of a sweetener in the absence of FOS or the presence FOS in the presence of two sweeteners, which were not able to give the sweetness desired by consumers in the concentrations used. In studies with a functional mix of sugar-free cerrado fruit preserves, Souza (2012) found that the sweetness desired by consumers occurs at concentrations of FOS and sweeteners of 13.18% and 0.047%, respectively (sucralose:acessultame-k ratio of 3:1). Regarding the overall likability, it is observed that the test with the lowest acceptability (4) lacks gelling agents (gums and pectin) in its formulation, thus obtaining an average score of 3.0 (moderately dislike). Tests 1, 2, 5, 6, 9, 10 and

17 were the most widely accepted for this sensory attribute, and these tests all have the presence of at least two types of gelling agents at levels 0 to +1.

Table 5 shows the estimates of the effects of different ingredients in relation to the sensory parameters of functional sugar-free guava preserves.

Table 5. The estimates of the effects of different ingredients in relation to the sensory parameters of functional sugar-free guava preserves.

Factor	Flavor	Consistency	Sweetness	Overall liking
Mean	5.19*	5.52*	4.95*	5.19*
FOS	0.61	-0.32	0.64	0.31
Th	0.11	-0.02	0.18	0.03
Su	2.08*	0.69	2.24*	1.79*
St	0.38	0.06	0.49	0.34
Ma	0.10	-0.70	0.07	-0.15
GXA	0.14	0.04	0.15	0.10
LBG	0.07	0.57	-0.03	0.17
CA	0.17	0.31	0.12	0.24
LMP	0.10	1.20*	-0.01	0.32
PO	-0.15	-0.41	-0.17	-0.32

FOS: fructooligosaccharide; Th: thaumatin; Su: sucralose; St: stevioside; Ma: maltitol; GXA: xanthan gum; LBG: locust bean gum; CA: carrageenan; LMP: low methoxyl pectin; PO: polydextrose; *p≤0,05

It is observed that only the attributes of the independent variable sucralose had a positive effect on flavor, sweetness and overall liking, indicating that the increase in the concentration of sucralose increases these attributes, thus obtaining a higher preference by consumers. According to Melo et al. (2009), the sweetener sucralose is preferred in the preparation of food products because the taste of sucrose approaches that of sugar and has no bitterness. Because the

consistency suffered, the positive effect of the low methoxyl pectin shows that to increase the consistency, the pectin content must be increased in the product. This shows that consumers prefer a firmer product.

3.2 Evaluation of the sweetness and consistency profiles

Figure 1 shows the histogram of the ideal of sweetness of the functional sugar-free guava preserves.

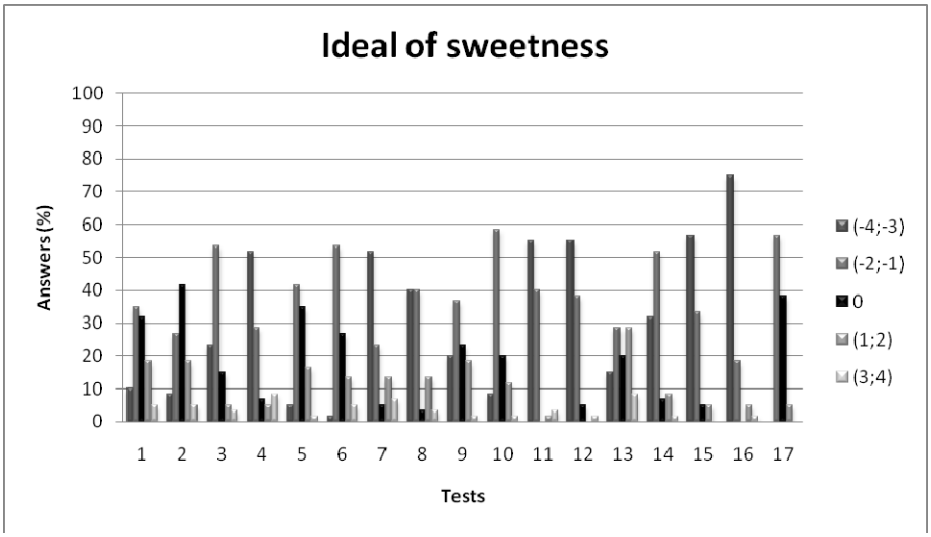

Figure 1 Histogram of the distribution of notes for the just-about-right scale of ideal sweetness of the functional sugar-free guava preserves. Test 17 is the mean of trials 17, 18 and 19.

Tests 2 and 17 received higher percentages of 0 notes (ideal) (approximately 40% of the notes), indicating an ideal sweetness (Figure 1). Test 2 had lower levels (-1) of stevioside (0.0%), locust bean gum (0.0%), low methoxyl pectin (0.0%) and polydextrose (20.0%), whereas the other variables

were at peak levels (+1). Test 17 had all the variables at the midpoint. Bayarri et al. (2003) studied the sweetness of gellan gum and carrageenan gum in gels and perceived a higher sweetness with lower levels of these gums because according to Gibson (1992), a lower gel strength more easily disintegrates in the mouth, releasing the sweetener sooner.

Test 16 (no sweeteners, lower concentration of FOS and higher concentrations of bulking agents maltitol and polydrextrose) received the majority of notes between -3 and -4 (much less sweet than the ideal and extremely less sweet than the ideal; Figure 1). The presence of FOS and a low-intensity sweetener (Mabel et al., 2008) did not produce the optimum sweetness.

Figure 2 shows the histogram of the ideal consistency of the functional sugar-free guava preserves.

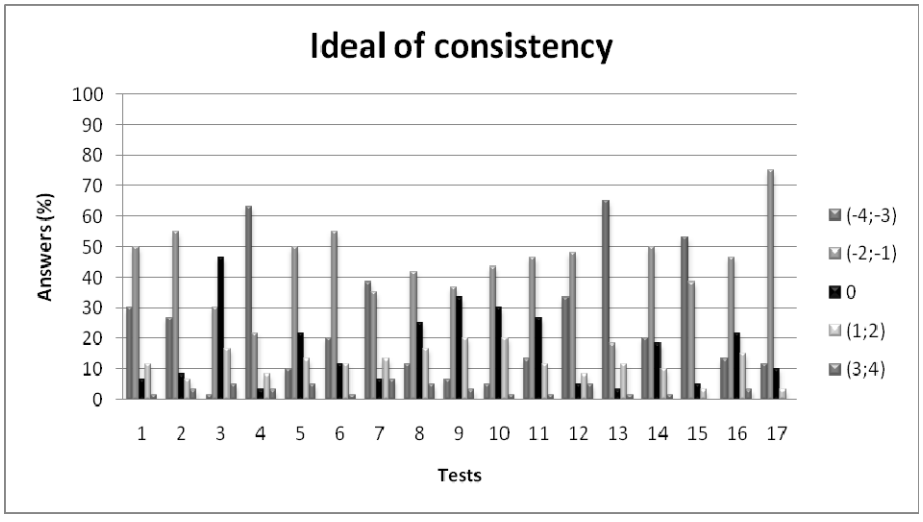

Figure 2 Histogram of the distribution of notes for the just-about-right scale of ideal consistency of the functional sugar-free guava preserves. Test 17 is the mean of trials 17, 18 and 19.

For test 3, approximately 45.0% of the notes for consistency were 0 (ideal) (Figure 2). This trial possessed lower levels (-1) of sucralose (0.0%), xanthan gum (0.0%), carrageenan (0.0%) and polydextrose (20.0%), whereas the other variables were at higher concentrations (level +1). Maltitol, polydextrose and FOS are bulking agents that are considered to affect the structure of food and improve food texture (Martinez-Cervera et al., 2012, Ribeiro et al., 2003, Roonda et al., 2005). Locust bean gum and low-methoxyl pectin are gelling agents that act as thickeners, gelling agents and stabilizers (Williams, 2007; Arda et al., 2009, Moreira et al., 2011). These results indicate that the right consistency of functional sugar-free guava preserves is achieved using such ingredients.

Tests 4 and 13 had higher frequencies of notes between -3 and -4 (much less consistent than ideal and extremely less consistent than the ideal; Figure 2). All studied gelling agents (xanthan gum, locust bean gum, carrageenan and low methoxyl pectin) are absent in these tests; therefore, the products were less firm.

3.3 Assessment of the acceptability of functional sugar-free guava preserves using PARAFAC

Figure 3 shows the three-way internal map, which is a representation of the distribution of consumers, the samples and the acceptance of the sensory attributes evaluated by consumers.

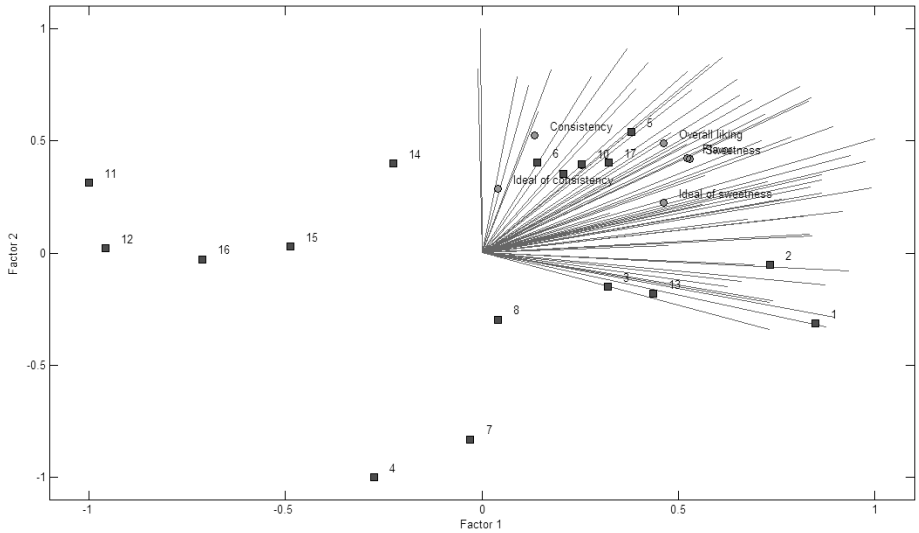

Figure 3. Three-way internal map (PARAFAC) for flavor, consistency, sweetness, overall liking, ideal of sweetness and ideal of consistency obtained for the functional sugar-free guava preserves. Test 17 is the mean of trials 17, 18 and 19.

It can be seen that tests 1, 2, 3, 5, 6, 9, 10, 13, 17 were the most preferred. The preference for tests 5, 6, 9, 10 and 17 is explained by the influence of studied sensory attributes, corroborating the results of the test medium.

3.4 Correlation between the sensory attributes and the texture profile parameters

To correlate the texture profile parameters with the sensory attributes of the different tests of the functional sugar-free guava preserves, Pearson's correlation was performed (Table 7). Table 6 shows the averages of the profile texture.

Table 6. The average of the TPA parameters of the different tests of the functional sugar-free guava preserves.

Tests	Hardness (N)	Adhesiveness (N.s)	Cohesiveness	Gumminess (N)
1	0.14	61.34	0.50	0.07
2	0.12	72.45	0.57	0.07
3	0.58	176.13	0.39	0.23
4	0.00	0.00	0.00	0.00
5	0.49	83.44	0.33	0.16
6	0.07	46.85	0.60	0.04
7	0.19	120.37	0.49	0.09
8	0.33	122.96	0.44	0.14
9	0.68	278.33	0.43	0.30
10	0.80	229.65	0.41	0.32
11	0.15	90.87	0.58	0.08
12	0.05	33.13	0.55	0.03
13	0.06	36.54	0.70	0.04
14	0.58	166.62	0.39	0.23
15	0.19	159.63	0.71	0.14
16	0.53	146.54	0.39	0.21
17	0.27	76.81	0.43	0.12

Test 17 is the mean of trials 17, 18 and 19.

It is observed that all values of the texture profile parameters are zero for test 4. This was due to not gel formation because there was an absence of all gelling agents in this test.

Table 7. Pearson's correlation coefficients between the sensory attributes and the texture profile parameters.

	Hardness	**Adhesiveness**	**Cohesiveness**	**Gumminess**
Flavor	0.23	0.09	0.15	0.20
Consistency	0.78**	0.67**	0.09	0.78**
Sweetness	0.22	0.1	0.18	0.20
Overall liking	0.43	0.31	0.20	0.42
Ideal of sweetness	0.02	-0.09	0.13	-0.01
Ideal of consistency	0.74**	0.60*	-0.13	0.69**

* $p<0.05$; ** $p<0.01$

The parameters of the texture profile hardness, adhesiveness and gumminess were positively correlated with the sensory attribute of consistency and with the ideal for the consistency, indicating that the increase in hardness, adhesiveness and gumminess in functional sugar-free guava preserves make the scores for the sensory attribute of consistency increase, tending toward the ideal. In studies on the use of banana peel in the preparation of banana preserves, Oliveira et al. (2009) found that consumers prefer more firm preserves.

4. CONCLUSION

The results indicated that sucralose showed higher positive influence on the flavor, sweetness and overall liking of guava preserves and that low methoxyl pectin positively influenced the consistency. These results suggest that when the concentration of sucralose and the amount of pectin added are higher, the acceptability of the functional sugar-free guava preserves is higher. The results also show that higher acceptabilities are achieved by using high levels of FOS and gelling agents, as these formulations tended to be rated as ideal. Positive correlations were observed between the texture profile parameters and the sensory attributes, indicating that consumers prefer a firmer guava preserve.

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ARTIGO 3: RHEOLOGICAL BEHAVIOR OF FUNCTIONAL SUGAR-FREE GUAVA PRESERVES: EFFECT OF THE ADDITION OF SALTS

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ABSTRACT

The addition of salts to carrageenan and locust bean gum gels functions to improve the characteristics of texture, thereby increasing gel strength. This effect is widely studied in gels of model systems but is studied to a lesser extent in complex systems, such as fruit preserves. The objective of this study was to evaluate the effect of adding salts on the rheological behavior of functional sugar-free guava preserves, as well as to correlate the rheological parameters. To this end, three types of texture properties were analyzed (texture profile, stress relaxation and uniaxial compression) in functional sugar-free guava preserves prepared with different concentrations of KCl and $CaCl_2$ salt. The analyses were performed with a texturometer (Stable Micro Systems, Model TA - XT2i), and the parameters were analyzed using a Scott-Knott test at 5% probability, principal components analysis and Pearson correlation. $CaCl_2$ was more effective for improving the characteristics of texture, especially gel strength (concentration near the F3: 0.33%), whereas KCl addition degraded gel strength. In the analysis of test relaxation, the Maxwell model parameters provided better discrimination between samples than the Peleg model parameters. Positive and negative correlations were observed, and the parameters of hardness, adhesiveness and elastic modulus ideal (E_1) were the most correlated with other rheological parameters.

Keywords: texture profile, stress relaxation, uniaxial compression, principal components analysis, Pearson correlation.

1. INTRODUCTION

All materials exhibit a response to an external force between the two extremities of ideal behavior: elastic solid and viscous liquid. An elastic solid is described by Hooke's law, and an ideal viscous liquid obeys the Newton's law (Rychlewski, 1984; Gunasekaran & Ak, 2000; Guillet, 2010). However, most food behaves as viscoelastic material; depending on the stress applied and the time scale, a solid body may have liquid phase properties and a liquid material can show solid body properties. The viscoelastic behavior of food has been widely studied in rheometers of sheared samples (tangential force), whereas the rheological parameters of tension or compression (normal force) are being increasingly used to characterize the texture of food products. Furthermore, it is possible to characterize the product to low or high deformations irrespective of the type force applied (Lu & Abbott, 1996; Kumagai et al., 2009; Ishihara et al., 2011; Karaman et al., 2011). Therefore, the rheology is extremely important for the food industry, mainly in the development of product formulations in which there is total or partial replacement of sugar (Acosta et al., 2008; Hracek et al., 2010), as is the case with preserves and jellies with low soluble solids content. An effective approach to the technological problems due to this substitution, such as the loss of sweet taste, desired viscous texture and increased water activity (Sandrou & Arvanitoyannis, 2000), requires a deep understanding of the functionality of ingredients in product development, quality control studies of shelf-life and determination of the texture of the food (Steffe, 1996).

In the preparation of jams and jellies with low soluble solids, low methoxyl pectin (LMP) is used, which forms gel in the presence of divalent metal ions (usually calcium) and does not necessarily require the presence of sugars (Ngouémazong et al., 2012). However, this type of pectin does not

contribute the same rheological characteristics of the conventional product. Therefore, it is necessary to use other gelling agents, such as carrageenan and locust bean gums (Williams, 2007; Moreira et al., 2011), as well as sweeteners and bulking agents.

According García-García & Totosaus (2008), the combination of carrageenan and locust bean gums are generally used in food production. Arda et al. (2009) demonstrated that synergistic peaks occur at the carrageenan/LBG ratio of 8/1 (maximum concentration of the mixture carrageenan/LBG gels in which introduces its best features) because locust bean gum modifies the texture characteristics of carrageenan gels, and these characteristics improve with the addition of salts, in particular KCl and $CaCl_2$ (García-García & Totosaus, 2008). The fractions ι- and κ- carrageenan form thermoreversible gels by cooling in the presence of calcium ions or potassium (Huang et al., 2007), whereas, according to Michel et al. (1997), the addition of small potassium ion concentrations results in a greater improvement in carrageenan gels than do calcium ions. Montero & Pérez-Mateos (2002) report that cations (Ca^{+2}>K^{+}>Na^{+}) bind to the double helices of carrageenans, neutralizing the sulfated groups and affecting the balance of the attractive and repulsive forces between the molecules, which increases gel rigidity. These authors also report that optimum gel strength will occur at certain levels of each cation and, depending on the amount of salt added, gel formation can become strong enough to promote syneresis.

The bulking agents must have characteristics similar to those of sucrose, which include replacement of solid stability at different pH and temperature conditions, no aftertaste, contribution to colour and interaction with starch and protein in a manner similar to that of sugar (Vissoto et al., 2005). Additionally, sweeteners should have no aftertaste and should be chemically stable with a low calorie sweetening power equal to or greater than that of sucrose, as well as

being soluble, non-toxic or non-carcinogenic and economically feasible (Hanger et al., 1996).

Among the agents commonly used, we highlight polydextrose and FOS (fructooligosaccharides). Polydextrose actively improves food texture and operates as a thickener, stabilizer and humectant (Martínez-Cervera et al., 2012; Ribeiro et al., 2003). Menezes (2011) used low methoxyl pectin content (0% to 1.008%), mixtures of xanthan gum, locust bean gum (1:1) (0% to 0.4032%) and polydextrose (20.0% to 40.16%) in guava preserves with no added sugar and with prebiotics (FOS) and found that concentrations above 20 g polidextrose/100 g pulp decreased the hardness of guava preserves. The author attributed this finding to the contribution of polydextrose to soluble solids of the product, which is a determining factor for the end of cooking process (when the preserves reached 60 °Brix). FOS, besides serving as a bulking agent, is also considered as a functional ingredient because it is not absorbed in the small intestine and exerts a prebiotic effect on the intestinal habitat, which causes an increase in stool and normalization of stool frequency (increases the number of bacteria and/or activity of the number of bifidobacteria and lactic acid bacteria in the human gut) (Cherbut, 2002; Nyman, 2002; Lobo et al., 2006; Roberfroid, 2007; Rodríguez-Cábezas et al., 2010; Rodrigues et al., 2011).

There are numerous sweeteners on the market, among which sucralose and thaumatin stand out. Sucralose is characterized by a lack of unpleasant aftertaste and a taste similar to but approximately 600 times sweeter than sucrose. Sucralose has the advantage of remarkable stability, both at high temperatures and over a wide pH range (Rahn e Yaylayan, 2010). Because thaumatin is an intensely sweet protein (300 to 3000 times sweeter than sucrose) produced by plants (Menu-Bouaouiche et al., 2003), it is used in chewing gum,

dairy products and pharmaceuticals (Daniell et al., 2000). According to Bayarri et al. (2004), high intensity sweeteners have no effect on product texture.

In the literature, there are a large number of studies that examine the effects of added salts on gel characteristics in model systems (Montero & Pérez-Mateos, 2002; Chen et al., 2002; Iglauer et al., 2011), but similar research on fruit preserves and jellies is scarce.

As such, the objective of this study was to evaluate the effect of adding salts on the rheological behavior of functional sugar-free guava preserves, as well as to correlate the rheological parameters.

2. MATERIAL AND METHODS

2.1. Material

We used ripe guava Pedro Sato cultivars from a local market. They were processed in the Pilot Plant Processing Plant Products, Department of Food Science, Federal University of Lavras/MG.

The ingredients used were as follows: fructooligosaccharides (Beneo P95), thaumatin (Nutramax), sucralose (Nutramax), gum LBG (Danisco), carrageenan (Danisco), low methoxyl pectin (LMP) (Danisco), polydextrose (Litesse), citric acid monohydrate (Nuclear) and potassium sorbate (Vetec).

According National Agency of Sanitary Vigilance, for a food to be considered functional, the portion of the product ready for consumption must provide at least 3 g of FOS and/or polydextrose if the food is solid or 1.5 g of FOS and/or polydextrose if the food is liquid (Brasil, 2008). For functional sugar-free guava preserves, which has a solid portion equivalent to 40 g (DRC 359; December 23, 2003 – Brasil, 2003), the minimum concentration of FOS and/or polydextrose required is 7.5%. Mesquita et al. (2012), in studies on the degradation of FOS in sugar-free guava jam with added prebiotics found that in processing the product was a loss of 36.0% FOS.

2.2. Preparation of guava preserves and experimental design

The different formulations of guava preserves were processed in open stainless steel pots, according to the methodology proposed by Menezes (2011). The mixture of pulp and polydextrose was heated to 45 °Brix, and then added gums (LBG + carrageenan), pectin LMP and salts previously homogenized

under high stirring in water at 80 °C, and remained cooking to achieve a soluble solids content of 50 °Brix. FOS (fructooligosaccharide) diluted 1:1 in water at room temperature was added in this step. The process of cooking continued until a total soluble solids of 65 °Brix was obtained. Citric acid, potassium sorbate and sweeteners were added at the end of the cooking process (diluted 1:1 in water at room temperature) to prevent degradation at the high temperature. Guava preserves were placed in packaging polypropylene transparent (volume: 402.0 cm^3, height: 50.50 mm, diameter: 100.70 mm), with the filling performed at a high temperature (85 °C) and were then closed, poured, and cooled to room temperature and stored in a chamber at 20 °C for later analysis.

For all guava preserves prepared, the following percentages of ingredients were established: 60.0% pulp guava, 2.0% pectin LMP (35% degree of methoxylation), 0.23% locust bean gum (0.44% (w/w) potassium, 0.1% (w/w) of calcium and 5.99 mg/kg of iron), 1.84% carrageenan (2.27% (w/w) potassium, 0.32% (w/w) calcium and 76.77 mg/kg of iron), 0.012% sucralose, 0.099% thaumatin, 17.71% FOS, 19.08% polydextrose, 0.2% citric acid and 0.05% potassium sorbate (all levels were determined in accordance with preliminary tests). The carrageenan used was composed of the mixture of kappa, iota and lambda carrageenan (food grade Danisco: Grindsted Carrageenan CL 350 H).

Was used a completely randomized design with three replications for this study.

Table 1 presents the percentages of salt added to the formulations of the functional sugar-free guava preserves.

Table 1 Percentages of salt added of the formulation of the functional sugar-free guava preserves.

Formulations	$CaCl_2$	KCl
F1	-	-
F2	0.165%	-
F3	0.33%	-
F4	0.66%	-
F5	-	0.165%
F6	-	0.33%
F7	-	0.66%

2.3. Texture Profile Analysis

The characteristics of food surface texture is one of the first quality parameters that consumers evaluate, and therefore becomes critical to product acceptance even before it is put in the mouth. Texture is composed of a set of sensory attributes that are highly important, considering that they determine or influence the acceptance/rejection of the food (Mojet & Köster, 2005; Taniwaki et al., 2006; Kotwaliwale et al., 2007; Funami et al., 2012).

Texture profile analysis (TPA) is a method to evaluate the sensory properties. The test consists of compressing the food (study sample) twice in a reciprocating motion to mimick the action of the mandible. Therefore, a first compression and relaxation followed by a second compression are performed during testing. This test yields a graph of force versus time, from which the texture parameters are calculated (Honikel, 1998; Lau et al., 2000; Bourne, 2002; Herrero et al., 2007).

The texture profile analyses (TPA) were performed in penetration mode under the following conditions: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, post-test speed of 1.0 mm/s, time interval between penetration cycles of 5.0 s, a distance of 20.0 mm and compression with a cylindrical probe of 6.0 mm diameter of the aluminum using the Stable Micro Systems Model TA-XT2i texturometer (Goldaming, England). The parameters analyzed were hardness, adhesiveness, cohesiveness and gumminess. The test was performed in triplicate. The analyses were conducted in the packaging containing the guava preserves (height: 50.50 mm, diameter: 100.70 mm).

2.4. Stress relaxation test

There are several mathematical models that can explain the behavior of viscoelastic food products, but the Maxwell and Peleg models are used most frequently to describe the behavior of gels and alimentary systems (Kampf & Nissinovitch, 1997; Morales et al., 2007; Andrés et al., 2008; Bellido & Hatcher, 2009; Khazaei & Mohammadi, 2009).

The Maxwell model involves two simple elements combined in a series to represent different behaviors. These two elements are the ideal elastic element, which can be represented as a spring and has a behavior defined by elastic constant E, and the ideal viscous element, which is represented by a dashpot and has a behavior defined by its viscosity η (Campus et al., 2010).

In the Maxwell model with a constant strain (ε_0), σ describes the tension applied from σ_0 for σ(t) after a time t (Nobile et al., 2007), given as follows:

$$\sigma(t) = \varepsilon_0 \left(E.\exp(-\frac{t}{\lambda}) + E_e \right) \quad (1)$$

where E is the elastic modulus of the material, E_e is the equilibrium elastic modulus and λ is the relaxation time given by η/E. Some foods do not follow the Maxwell simplified viscoelastic model, and therefore the description of their behavior requires more complex models. An example of this case is the generalized Maxwell model, which consists of an infinite number of Maxwell models in parallel over a spring.

The stress relaxation curves (stress versus time) can be adjusted by means of equation 2, which provides the viscoelastic parameters of the generalized Maxwell model.

$$\sigma(t) = \varepsilon_0 \left(E_1 \exp(-\frac{t}{\lambda_1}) + E_2 \exp(-\frac{t}{\lambda_2}) + ... + E_e \right) \quad (2)$$

where E_1, E_2 ... are the elastic modulus of the elastic body ideal, E_e is the equilibrium elastic modulus and λ_1, λ_2 … are the relaxation times.

The viscosity of element i can be calculated according to equation 3:

$$\eta_i = E_i \lambda_i \quad (3)$$

In the Peleg model, stress relaxation data can be interpreted in accordance with the stress normalized, according to equation 4 (Peleg & Normand, 1983):

$$\frac{\sigma_0 t}{\sigma_0 - \sigma(t)} = k_1 + k_2 t \quad (4)$$

where $\sigma(t)$ is the stress at any time during the test, σ_0 is initial relaxation stress, k_1 and k_2 are constants. The reciprocal k_1 represents the initial decay rate, whereas $1/k_2$ is the hypothetical value of the asymptotic normalized force that remains without relaxing (Rodríguez-Sandoval et al., 2009; Tang et al, 1998).

The stress relaxation test was performed in a texturometer (Stable Micro Systems Model TA-XT2i). The samples were cut into cylindrical shapes of 2.0 cm in height and 2.0 cm in diameter and compressed to 5.0% original height with a speed of 1.0 mm/s. The deformation was kept constant for 10.0 minutes, which allowed the stress to reach equilibrium. During that time, the relaxation of tension was measured at a rate of 1.0 per second. A 7.0 cm diameter probe cylinder, which had been lubricated to eliminate the influence of friction between the sample and the equipment, was used. Three measurements were performed for each formulation. The nonlinear regression program R (2011) was used.

Determination of the Peleg model constants was also performed using the nonlinear regression program R (2011).

2.5. Measurement of the uniaxial compression

Compression tests were performed in a texturometer (Stable Micro Systems Model TA-XT2i) using a 7.0 cm diameter probe cylinder. The samples were cut into cylindrical shapes of 2.0 cm in height and 2.0 cm in diameter and compressed to 80.0% original height with a speed of 1.0 mm/s.

From the force versus time/deformation curve, the following properties were calculated: true rupture stress (σ) and true rupture strain (ε) according to equations 5 and 6 (Hamann, 1983; Hernández et al., 1999; Bayarri et al., 2003; Bayarri et al., 2007):

$$\sigma = F\left(\frac{h_0 - \Delta h}{A_0 h_0}\right) \quad (5)$$

$$\varepsilon = \ln\left(\frac{h_0}{h_0 - \Delta h}\right) \quad (6)$$

where F is the rupture force, h_0 and A_0 are the initial height and cross-section area of the sample, respectively, and Δh is the change in height during compression.

From the stress versus strain curves, the true rupture stress (σ_{rup}), the true rupture strain (deformation Hencky - ε_{rup}) and work of rupture (W_{rup}) were obtained. The true rupture stress is the point at which gel fracture occurs (y axis) (maximum stress in the graphic tension versus deformation), and true rupture strain is the strain at the break of the sample (x axis). The modulus of elasticity (Young's modulus - E) was obtained from the slope of the linear part of the initial stress-strain curve using 2.0% deformation and the work of fracture (W_{rup}) was given by the area under the curve strength versus the distance from the rupture point.

2.6. Data analysis

To compare formulations with different levels of salts, the Scott-Knott test at 5% probability was used. A better understanding of the differentiation between the samples became the purpose of the principal component analysis. Furthermore, Pearson's correlation was used to correlate the rheological

properties. Data analysis was performed in software R (2011) (R Development Core Team) and SensoMaker software version 1.0.

3. RESULTS AND DISCUSSION

3.1. Texture Profile Analysis

Table 2 presents the mean texture profile analysis of functional sugar-free guava preserves with $CaCl_2$ or KCl. It was observed that all texture profile parameters were able to discriminate between samples, indicating a significant difference in all these parameters.

Table 2 Texture profile analysis of functional sugar-free guava preserves with $CaCl_2$ or KCl.

Formulations	Hardness (N)	Adhesiveness (N.s)	Cohesiveness	Gumminess (N)
F1 (no added salts)	2.87±0.4^{d}	-68.48±0.4^{b}	0.39±0.01^{b}	1.17±0.2^{b}
F2 (0.165% $CaCl_2$)	6.93±0.4^{a}	-116.05±0.3^{c}	0.39±0.01^{b}	2.78±0.1^{a}
F3 (0.33% $CaCl_2$)	6.23±0.2^{b}	-97.52±0.2^{c}	0.37±0.02^{c}	2.32±0.1^{b}
F4 (0.66% $CaCl_2$)	5.86±0.3^{b}	-80.30±0.3^{b}	0.37±0.02^{c}	2.14±0.4^{b}
F5 (0.165% KCl)	4.60±0.5^{c}	-67.43±0.2^{b}	0.36±0.01^{c}	1.64±0.1^{c}
F6 (0.33% KCl)	4.04±0.2^{c}	-42.59±0.2^{a}	0.35±0.01^{c}	1.63±0.2^{c}
F7 (0.66% KCl)	1.72±0.2^{e}	-49.52±0.1^{a}	0.45±0.01^{a}	0.85±0.2^{d}

Means followed by same letter in columns do not differ statistically among themselves by Scott-Knott test at 5% probability.

Regarding the hardness parameter, the formulation with the addition of 0.165% $CaCl_2$ (F2) had the highest value, and as the concentration of salt increased, hardness decreased. For KCl, the formulations with the salt had values of greater hardness than the formulation with 0% (F1), with the exception of F7. In the levels evaluated, it was observed that with an increasing KCl concentration, hardness decreased. In their studies with solutions of pectin in

various pHs and various concentrations of salts (sodium caseinate), Ambjerg-Pedersen & Jorgensen (1991) reported that the effect of cations depends on the concentration because the saturation of anionic groups at high concentrations destabilizes gel structure. Thrimawithana et al. (2010), in a study on iota and kappa carrageenan in the presence of calcium chloride and potassium chloride, reported that this decline in hardness may be due to the inability of cations to form connections with the oxygen group and anhydrous sulfates groups of the adjacent disaccharide units of carrageenan, which reduces intermolecular association. These authors suggest that at certain concentrations of cations, partial gelatinization of the carrageenan gel makes the product less hard. In studies on the use of banana peel in the preparation of banana preserves, Oliveira et al. (2009) found that consumers prefer a more firm preserve.

Regarding adhesiveness, increasing the concentration of both KCl and $CaCl_2$ decreased the value of this parameter (Table 2). Adhesiveness is a surface feature (Adhikari et al., 2001; Huang et al., 2007; Besbes et al., 2009), and according to Pons & Fiszman (1996), large deformation (as in the case of the TPA) is not recommended for the calculation of adhesiveness.

The cohesiveness of the rheological parameters is correlated with the properties of a food as it is swallowed, especially if it is in solid state (Lucas et al., 2002; Ishihara et al., 2011). This measurement is calculated from the ratio between the area under the curve for strength versus time in the second compression cycle and the curve's area in the first compression cycle (Bourne, 1982, Gujral et al., 2002), i.e., the lower the cohesiveness, the greater the disintegration of material in the first compression cycle (Extralab, 2010). In the present study, it was observed that increasing the concentration of $CaCl_2$ decreases the cohesiveness, i.e., the product disintegrates more easily because the concentration of salt can cause partial gelatinization (Thrimawithana et al.,

2010). Regarding formulations containing KCl, there were no differences from those with a $CaCl_2$ concentration of 0.33%. At a concentration of 0.66% KCl, the product had a higher average value of cohesiveness. This finding was not expected because this formulation, with lower hardness, disintegrated quickly. This event may have occurred because F7 contained more fluid, and when introduced into the container, the material tended to return to its initial state, making the area of the second compression cycle closest to the area of the first compression cycle. The areas of the first and second compression cycles of the formulation F7 areas were lower than those of the other formulations.

According to Oliveira et al. (2009), gumminess determines the force required to chew a semi-solid food. The parameter of gumminess presented with a higher value for the formulation with 0.165% $CaCl_2$ (F2), which indicates that this formulation was more rigid. At higher levels of $CaCl_2$ (F3 and F4), there was a decrease in gumminess. For formulations with KCl, at all concentrations added, gumminess was higher than in F1 (without added salts), except for formulation F7, in which a reduction in gumminess with increasing concentration of KCl was observed. Thrimawithana et al. (2010), in research on κ- and ι- carrageenan gels in the presence of ions, found that depending on the concentration and type of salt used, the conditions can become insufficient to form a uniform gel matrix, which may explain the decrease in the parameters of cohesiveness and gumminess with increasing salt concentration.

3.2. Stress relaxation test

When a constant load is applied to the materials, different relaxation behaviors can be observed in materials with different viscoelastic properties; ideal elastic materials do not relax, ideal viscous materials relax instantly, and

viscoelastic solids gradually relax and reach a equilibrium (Steffe, 1992; Li et al., 2010).

Figure 1 shows the stress versus time curves of different formulations of functional sugar-free guava preserves with different levels of KCl or $CaCl_2$. There was the graphic with the first 40 points only to observe the behavior of the products in different types and concentrations of salts.

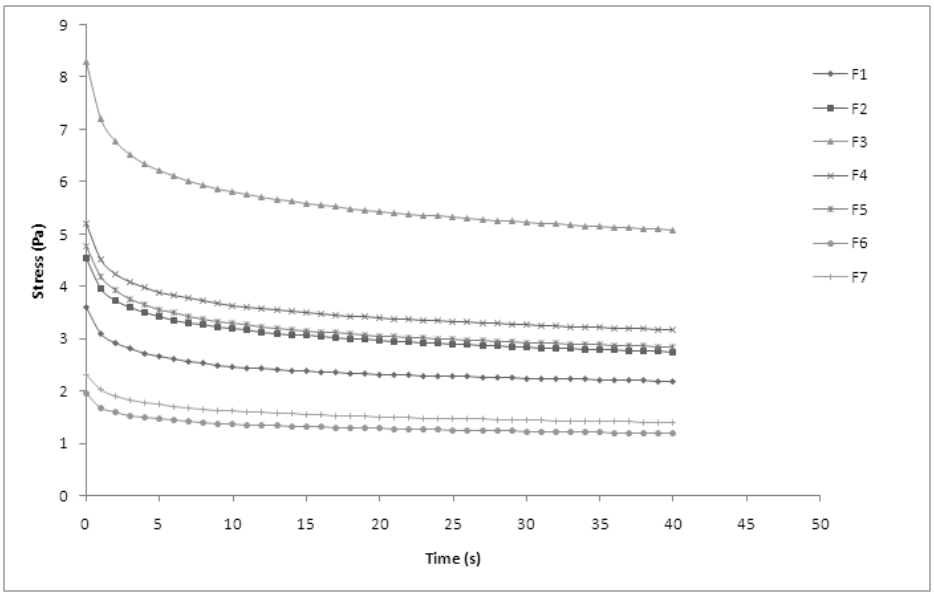


Figure 1 Stress versus time curves of different formulations of functional sugar-free guava preserves with different levels of KCl or $CaCl_2$.

Figure 1 illustrated that addition of 0.33% $CaCl_2$ (F3) increased the stress values. However, higher concentrations of salt decreased the stress, but values are greater for the stress of the formulation without added salt (F1). The addition of 0.165% KCl (F5) to guava preserves caused a stress increase, but after that, the concentration stress decreased to values lower than F1.

Figure 2 shows the linearized relaxation curves of Peleg's model (equation 4) for different formulations of functional sugar-free guava preserves with different levels of KCl or $CaCl_2$. There was the graphic with the first 30 second only to observe the behavior of the products in different types and concentrations of salts.

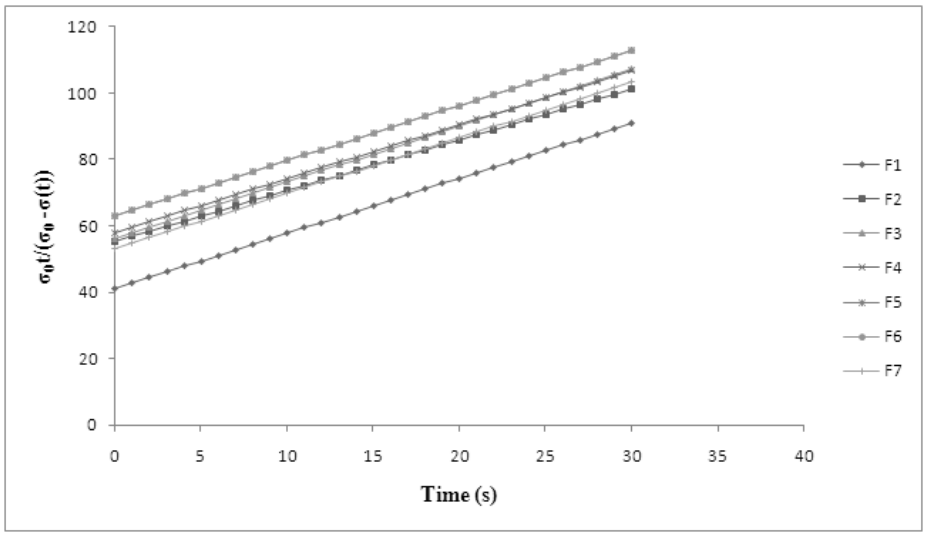


Figure 2 Linearized relaxation curves of Peleg's model of different formulations of functional sugar-free guava preserves with different levels of KCl or $CaCl_2$.

According Tang et al. (1998), Bhattacharya et al. (2006), Sozer & Dalgic (2007), Sozer et al. (2008) and Rodríguez-Sandoval et al. (2009), application of Peleg's model to describe the relaxation data is a simple way to describe and compare the stress relaxation with the literature data on rheology because it uses only two properties: the initial decay rate ($1/k_1$) and the normalized stress ($1/k_2$). The k_1 property is a measure of the ease with which the material deforms, i.e., higher values of k_1 suggest a harder material, which

dissipates less energy, and therefore requires more force to be compressed (Guo et ai., 1999; Rodríguez-Sandoval et al., 2009). The k_2 property represents the degree of relaxation of the material (Guo et ai., 1999; Bellido & Hatcher, 2009; Rodríguez-Sandoval et al., 2009). According Peleg (1980), $1/k_2$ represents the equilibrium conditions of the material, i.e., the portion of the material that remains without relaxing at equilibrium. Figure 3 clearly shows that the linearized relaxation curves of guava preserves are too close to discriminate between them (Scott-Knott test at 5% probability was performed (data not shown), and no significant difference was found), which means that Peleg's model is not suitable for formulation discrimination.

Table 3 presents the average values of viscoelastic properties of Maxwell's model for functional sugar-free guava preserves with different levels of KCl or $CaCl_2$. This model was chosen because there was no considerable improvement (up R^2) when generalized model of Maxwell's model of two elements and spring in parallel were tested.

Table 3 Viscoelastic properties of Maxwell's model for functional sugar-free guava preserves added salts.

Formulations	E_e (Pa)	E_1 (Pa)	λ (s)	η (Pa.s)
F1 (no added salts)	52.39±0.1^b	21.28±0.3^c	105.13±0.7^b	2237.17±0.4^d
F2 (0.165% $CaCl_2$)	52.05±0.3^b	33.64±0.4^b	140.22±0.2^a	4717.00±0.7^b
F3 (0.33% $CaCl_2$)	74.29±0.9^a	42.85±0.3^a	132.06±0.1^a	5658.77±0.3^a
F4 (0.66% $CaCl_2$)	31.48±0.1^c	25.19±0.2^c	159.07±0.9^a	4006.97±0.3^c
F5 (0.165% KCl)	46.77±0.3^b	22.95±0.5^c	79.40±0.3^b	1822.23±0.9^d
F6 (0.33% KCl)	16.65±0.7^c	10.81±0.1^d	143.72±0.3^a	1553.61±1.3^d
F7 (0.66% KCl)	20.17±0.2^c	12.51±0.2^d	121.47±0.1^a	1519.59±1.1^d

Means followed by same letter in columns do not differ statistically among themselves by Scott-Knott test at 5% probability.

It was observed that, contrary to Peleg's model, Maxwell's model was able to discriminate between the formulations because there were significant difference between formulations (Table 3) in all parameters of model.

The properties of elasticity (E_e and E_1) quantify the rigidity of the material (Peleg, 1987; Rodríguez-Sandoval, 2009). The formulation F3 (0.33% $CaCl_2$) had a larger average E_e and E_1 (Table 3), which indicates that functional sugar-free guava preserves with 0.33% $CaCl_2$ are more rigid. Additionally, the formulations with added KCl showed lower values for these parameters, which indicates that these are less rigid.

The use of 0.165% KCl does not significantly alter the relaxation time (λ) of functional sugar-free guava preserves in relation formulation without salt (F1). However, the increase of salt concentration caused the relaxation time to increase and then reach a plateau. For $CaCl_2$, the addition of 0.165% always led to a significant increase in the relaxation time and stabilization at higher concentrations. According Nobile et al. (2007), Bhattacharya (2010), and Campus et al. (2010), lower relaxation time values indicate that the material is less elastic and less firm.

The viscosity (η) behaved similarly to the properties of elasticity. Larger values were observed for formulation F3 (0.33% $CaCl_2$), which indicates that this behavior is stronger than the others. There was no significant difference in the viscosity of the formulation without added salt (F1) and those with KCl (F5, F6 and F7).

3.3. Measurement of the uniaxial compression

Table 4 shows the averages of the resistance to compression parameters (true rupture stress, true rupture strain, modulus of elasticity and work of rupture) for functional sugar-free guava preserves with added salts. Significant differences in all parameters analyzed were able to discriminate between the samples of the functional sugar-free guava preserves with added salts.

Table 4 Resistance to compression parameters analysis for functional sugar-free guava preserves with added salts.

Formulations	σ_{rup} (kPa)	ε_{rup}	E (kPa)	W_{rup} (kJ/m^2)
F1 (no added salts)	25.35±0.3^{d}	0.29±0.6^{b}	47.68±0.2^{b}	3.67±0.1^{c}
F2 (0.165% $CaCl_2$)	41.55±0.9^{c}	0.28±1.1^{b}	82.13±0.6^{a}	5.20±0.1^{c}
F3 (0.33% $CaCl_2$)	74.41±0.1^{b}	0.53±0.2^{a}	80.82±0.2^{a}	19.36±0.2^{b}
F4 (0.66% $CaCl_2$)	88.58±0.4^{a}	0.51±0.8^{a}	37.10±0.3^{c}	24.67±0.5^{a}
F5 (0.165% KCl)	39.17±0.9^{c}	0.24±0.5^{b}	59.20±0.1^{b}	5.74±0.2^{c}
F6 (0.33% KCl)	9.44±1.3^{e}	0.22±0.2^{b}	16.97±0.1^{d}	2.75±0.1^{c}
F7 (0.66% KCl)	18.12±0.6^{d}	0.29±0.3^{b}	53.43±0.5^{b}	2.98±0.9^{c}

Means followed by same letter in columns do not differ statistically among themselves by Scott-Knott test at 5% probability.

True rupture stress (σ_{rup}) is defined as the stress required to break the food matrix (Cunha, 2002). According to Marudova & Jilov (2003), higher true rupture stress pre-supposes a more elastic behavior. It is observed that there was no significant difference in the value of true rupture stress between the formulations F2 (0.165% $CaCl_2$) and F5 (0.165% KCl). The formulations with the addition of $CaCl_2$ showed the highest mean, which indicates that the addition of calcium chloride caused guava preserves to strengthen. When using low

methoxyl pectin, the addition of Ca^{2+} is necessary because this type of pectin forms gel in the middle of zones of junction between the free carboxyl and Ca^{2+} and is supplemented by hydrogen bonds (Fiszman, 1989). Depending on the type of fruit used, it may not be necessary to add calcium. Such is the case with calcium rich guava, according El-Buluk et al. (1997). However, as noted in this study, the formulation without the addition of $CaCl_2$ (F1) had a lower true rupture stress, i.e., were less strong, than those with addition of $CaCl_2$ (F2, F3, F4). The formulation F6 (0.33% KCl) showed the lowest true rupture stress that the formulation F1 and F7, and F7 did not differ from the formulation without salts (F1). Marudova & Jilov (2003) noted that in low methoxyl pectin gels with added monovalent cations, the true rupture stress was lower, which made the gels become brittle. These authors suggest that the decreased true rupture stress was due to the decrease in cross links between pectin chains influenced by the addition of these salts. This increase in true rupture stress with increasing $CaCl_2$ concentration may be due to interaction with the calcium in both pectin and carrageenan with the resulting increase in gel strength. This result occurs because calcium carrageenan induces conformational changes in gel form (MacArtain et al., 2003) and induces gel formation of pectin LMP (Fiszman, 1989). Michel et al. (1997) studied the phase diagram of carrageenan in the presence of sodium, copper, potassium and calcium. In the case of divalent cations (Cu^{2+} and Ca^{2+}), these authors found that at low concentrations of these (<0.02 M) at all concentrations of carrageenan studied, there is gel formation. They also found that the addition of cations above a critical value does not affect the overall viscoelasticity (G') of the system and contributes to only some heterogeneity in the gel. Lai et al. (2000) also studied the effects of calcium on carrageenan gum and reported that there was a decrease in gel strength at a

calcium concentration above 0.5 M, which suggests that this decrease was due to syneresis.

In experiments on carrageenan gum system with sodium, potassium and calcium, Takemasa et al. (2001) observed that the after system gelation with calcium showed a high modulus of elasticity. This system was also characterized by a low stress optical coefficient, which indicates low anisotropy of the polymer chains and little reorientation of these chains caused by mechanical deformation of the gel networks. According MacArtain et al. (2003), these results indicate that calcium induces a high increase in branching during the gelation of the carrageenan, most likely due to aggregation after cooling coil.

True rupture strain (ε_{rup}) indicates the brittleness of the food's texture, i.e., the extent to which the product can be deformed without tearing (Cunha, 2002). Materials with high true rupture stress and true rupture strain are rigid and strong, whereas materials with high true rupture stress but with low values of true rupture strain are hard and brittle. Formulations F3 (0.33% $CaCl_2$) and F4 (0.66% $CaCl_2$) differed statistically with respect to true rupture strain of the other formulations showing higher mean values (0.53 and 0.51, respectively). The increase in KCl concentration did not affect the true rupture strain of guava preserves. Yoo et al. (2009) studied the characteristics of enzymatically deesterified pectin gels produced in the presence of monovalent ionic salts and found that the gelatinization depended on the degree of methoxylation of the pectin and that the pectin gels produced with 0.2 M KCl were brittle.

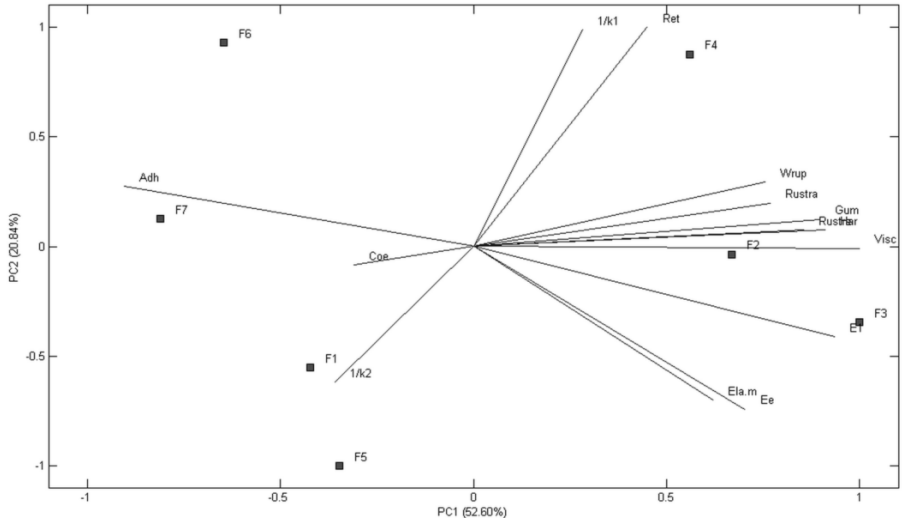
Gels with high values of elastic modulus (E) are more rigid (Fraeye et al., 2010). Formulations F2 (0.165% $CaCl_2$) and F3 (0.33% $CaCl_2$) had higher average modulus of elasticity and were not significantly different. However, F2 was rigid and brittle, and when the level of calcium was duplicated (F3), the gel was hard, but strong. According Fraeye et al. (2010) in studies with pectin with

different degrees of methoxylation of the concentration, an increase of Ca^{2+} leads to an increase in the modulus of elasticity, which makes more rigid gel until it reaches a plateau. Dunstan et al. (2001) reported that the concentration of salt (KCl) solution containing carrageenan gum and locust bean gum causes the modulus to increase to a maximum and then decrease. This result occurs because there is an increased rigidity of the resulting three dimensional networks and syneresis (Cardenas et al., 2008).

The work of rupture, which is the parameter which indicates the energy required to induce rupture of gel (Roopa & Bhattacharya, 2009), showed values between 2,75 kJ and 24,67 kJ. The guava preserve with 0.66% $CaCl_2$ (F4) had a higher work of rupture. The formulation with KCl did not differ among them in that respect. Roopa & Bhattacharya (2009) observed that in alginate gels, the concentration of $CaCl_2$ causes the energy of rupture (W_{rup}) to increase.

3.4. Principal component analysis

The principal components analysis was performed to obtain an overview about the behavior of salts on the rheological properties of functional sugar-free guava preserves (Figure 3).



Coe: cohesiveness; Adh: adhesiveness; Rustre: True rupture stress; Ret: relaxation time; Gum: gumminess; Har: hardness; Rustra: True rupture strain; Visc: viscosity; Ela.m: modulus of elasticity; Wrup: work of rupture; E_e: equilibrium elastic moduli; E_1: elastic moduli of the elastic body ideal; $1/k_1$: initial decay rate; $1/k_2$: hypothetical value of the asymptotic normalized force

Figure 4 Principal components analysis for rheological properties of functional sugar-free guava preserves

Principal component analysis (PCA) reduces a large number of variables to a few orthogonal variables called principal components (PC), which describe the largest covariance in the data analyzed (Lu et al., 2011). Variables found close to one another in pairs or groups show a positive correlation (Fredriksson et al., 1998). The first principal component (PC1) explains 52,60 %, and the second (PC2) explains 20,84 % of the variance of the model. We observed two distinct groups. One consisted of the formulation F1 (without added salts) and formulations with KCl (F5-0.165%, F6-0.33% and F7-0.66%), and another contained the formulations with $CaCl_2$ (F2-0.165%, F3-0.33% and F4-0.66%), the latter being positively influenced by most of the rheological parameters.

3.5. Correlation between the rheological properties

Pearson's correlation coefficients between the different rheological properties of the functional sugar-free guava preserves with/without added salt are shown in Table 5.

Table 5 Pearson's correlation coefficients between the rheological properties.

	Har	Adh	Coh	Gum	E_e	E_1	λ	η	$1/k_1$	$1/k_2$	σ_{rup}	ε_{rup}	E	W_{rup}
Har	1													
Adh	-0.83*	1												
Coh	-0.57	0.15	1											
Gum	0.99**	-0.84*	-0.50	1										
E_e	0.52	-0,71	-0,30	0.48	1									
E_1	0.82*	-0.87*	-0.32	0.78*	0.78*	1								
λ	0.41	-0.23	-0.07	0.47	-0.32	0.26	1							
η	0.84*	-0.90**	-0.22	0.84*	0.66	0.97**	0.46	1						
$1/k_1$	-0.84*	-0.02	-0.57	-0.22	0.74	0.66	-0.95**	0.46	1					
$1/k_2$	-0.45	0.59	-0.14	-0.56	-0.07	-0.43	-0.70	-0.65	-0.36	1				
σ_{rup}	0.51	-0.01	-0.70	0.47	-0.11	0.28	0.66	0.31	-0.65	-0.20	1			
ε_{rup}	0.47	-0.45	-0.10	0.41	0.36	0.78*	0.49	0.75	0.31	-0.37	0.44	1		
E	0.46	-0.80*	0.21	0.49	0.77*	0.66	-0.20	0.66	-0.75	-0.27	-0.41	0.26	1	
W_{rup}	0.46	-0.80*	-0.57	0.21	0.49	0.77*	0.66	-0.20	0.66	0.18	-0.27	0.33	0.26	1

* $p<0.05$; ** $p<0.01$

Har: hardness; Adh: adhesiveness; Coh: cohesiveness; Gum: gumminess; E_e: equilibrium elastic moduli; E_1: elastic moduli of the elastic body ideal; λ: relaxation time; η: viscosity; $1/k_1$: initial decay rate; $1/k_2$: hypothetical value of the asymptotic normalized force; σ_{rup}: True rupture stress; ε_{rup}: True rupture strain; E: modulus of elasticity; W_{rup}: work of rupture

Hardness (Har) was negatively correlated with the adhesiveness (Adh) (in absolute value) (-0.83, $p<0.05$) and initial decay rate ($1/k_1$) (-0.84, $p<0.05$) and positively with the gumminess (Gum) (0.99, $p<0.01$), the elastic moduli of

the elastic body ideal (E_1) (0.82, $p<0.05$) and the viscosity (η) (0.84, $p<0.05$). Goldner et al. (2012) obtained opposite results for the correlation of hardness with the adhesiveness studies on cooked tubers because the texture profile analysis simulates mastication, and therefore requires large deformations (20% to 50%) (Huang et al., 2007). These strains cause the sample to collapse, and so it is not suitable for the calculation of certain parameters, such as adhesiveness (Pons & Fiszman, 1996) because this parameter is a surface characteristic (Adhikari et al., 2001; Huang et al., 2007; Besbes et al., 2009). According to the observations in this study, the increase of $1/k_1$ is related to the softening and this occurs because materials with higher $1/k_1$ dissipate more energy and are therefore softer (Guo et al. 1999; Rodríguez-Sandoval et al. 2009). The gumminess determines the force required to chew a semi-solid food (Oliveira et al., 2009), i.e., the higher the hardness, the greater the gumminess, which confirms the results obtained. In this study, it was found that as the hardness increased, the elastic modulus of the elastic body ideal (E_1) increased. According to Peleg (1980), the elastic moduli is a property that can be used to measure the hardness of a material, such that samples with higher values of elastic moduli are harder materials. The increase in the hardness of viscoelastic materials causes the viscosity to increase. Such a result was reported by Rodríguez-Sandoval et al. (2009), which found that materials with higher hardness have greater relaxation times and thus higher viscosities.

Adhesiveness (Adh) was negatively correlated with the gumminess (Gum) (-0.84, $p<0.05$), the elastic moduli of the elastic body ideal (E_1) (-0.87, $p<0.05$), the viscosity (η) (-0.90, $p<0.01$), the modulus of elasticity (E) (-0.80, $p<0.05$) and the work of rupture (W_{rup}) (-0.80, $p<0.05$). As previously mentioned, the adhesiveness, a surface feature the use of large deformations is not appropriate for calculating the parameter because small deformations

(approximately 2.0%) and long contact times with the sample probe (approximately 300s) must be used for the calculation of this parameter (Borde et al. (2010)).

Gumminess (Gum) was positively correlated with the elastic moduli of the elastic body ideal (E_1) (0.78, $p<0.05$) and the viscosity (η) (0.84, $p<0.05$). This finding is consistent with other studies (Peleg, 1980; Bellido & Hatcher, 2009; Oliveira et al., 2009; Rodríguez-Sandoval et al., 2009), which report that materials with higher elastic moduli of the elastic body ideal and viscosity are harder, and being gummy, require more strength to chew, which closely correlated with these parameter.

The equilibrium of the elastic moduli (E_e) was positively correlated with the elastic moduli of the elastic body ideal (E_1) (0.78, $p<0.05$) and the modulus of elasticity (E) (0.77, $p<0.05$). E_e and E_1 properties are closely related because are the properties of the elastic element in Maxwell's model (Kaur et al., 2002; Bellido & Hatcher, 2009). The higher solid behavior of material with the highest values of these properties and the modulus of elasticity (E) is related to the rigidity of the material (Fraeye et al., 2010).

The elastic moduli of the elastic body ideal (E_1) are positively correlated with the viscosity (η) (0.97, $p<0.01$), the true rupture strain (ε_H) (0.78, $p<0.05$) and the work of rupture (W_{rup}) (0.77, $p<0.05$). Viscosity is calculated according to equation 3. The greater the E_1, the higher the η. According Fraeye et al. (2010), the higher the solid behavior of the material, the higher the true rupture strain. Roopa & Bhattacharya (2009) found that the greater the W_{rup}, the greater the energy required to rupture the material, which indicates that the material has solid behavior.

Relaxation time (λ) was negatively correlated with the initial decay rate ($1/k_1$) (-0.95, $p<0.01$). That finding is in line with the literature (Guo et ai., 1999;

Nobile et al., 2007; Rodríguez-Sandoval et al., 2009; Bhattacharya, 2010; Campus et al., 2010), in which stronger materials have longer relaxation and higher k_1 values.

4. CONCLUSIONS

The rheological properties of functional sugar-free guava preserves varied according to the type and concentration of added salts. $CaCl_2$ was more effective for improving the characteristics of texture, especially gel strength (concentration near the F3: 0.33%), whereas KCl addition degraded gel strength. In testing the parameters of relaxation, Maxwell's model discriminated better between the samples than the Peleg's model parameters. Positive and negative correlations were observed, and the parameters of hardness, adhesiveness and elastic moduli of the elastic body ideal (E_1) were the most correlated with the other rheological parameters.

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ARTIGO 4: EFFECT OF GELLING AGENT CONCENTRATION ON THE TEXTURE AND SENSORY CHARACTERISTICS OF FUNCTIONAL SUGAR-FREE GUAVA PRESERVES

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ABSTRACT

Response surface methodology (RSM) was used to determine the effect of gelling agents and their concentrations on the sensory characteristics (flavor, texture, sweetness and overall liking) and texture (texture profile analysis and stress relaxation) of functional sugar-free guava preserves. The gelling agents used were locust bean gum (0.16% to 1.84%), carrageenan (0.16% to 1.84%) and low methoxyl pectin (1.16% to 2.84%). The effects on the sensory and texture characteristics were studied using a central composite design (CCD). The results showed that the carrageenan and locust bean gums strongly influenced the texture and sensory characteristics of the functional sugar-free guava preserves. It is suggested that low methoxyl pectin at a concentration of 2.0% can be used together with carrageenan and locust bean gums. The results suggest that for the best scores of the sensory attributes, locust bean gum and carrageenan concentrations ranging from 0.16% to 0.41% must be used. Higher instrumental values of the texture characteristics were achieved with higher concentrations of these two gums.

Keywords: carrageenan, locust bean gum, low methoxyl pectin, acceptance test, texture profile test, stress relaxation test

1. INTRODUCTION

The food industry has relied mostly on incremental innovation for its new product launches, but it is becoming increasingly aware that breakthrough, "new to the world" innovations are needed to remain competitive. The modification of food structures to generate novel flavor and texture sensations in products that provide consumers with unique eating experiences is increasing the importance of understanding the relationships between food structure, mastication and sensory perception (Foster et al., 2011). Moreover, due to an alarming increase in diabetes, obesity and other diseases (Hracek et al., 2010), there is an increased demand for products without added sugar (Acosta et al., 2008) and products that provide health benefits with ingredients, such as bioactive compounds and prebiotics (Foster et al., 2011). However, there are technological problems in the replacement of sugar in food systems because it serves several important functions, such as the development of sweetness and viscosity, contribution to the desired texture, and lowering of the water activity of others functions (Sandrou & Arvanitoyannis, 2000). Therefore, the development of sugar-free products requires the inclusion of many additives, including gelling agents, to provide all the functions of sugar (Hracek et al., 2010). Among all gelling agents, carrageenan, locust bean gum (LBG) and low methoxyl pectin (LMP) stand out. They act as thickeners, stabilizers and inhibitors of syneresis (Dunstan et al., 2001; Spagnuolo et al., 2005; Arda et al., 2009; Ngouémazong et al., 2012). The combined use of these agents have several advantages due to synergistic effects (Dunstan et al., 2001; Mandala et al., 2004; Arda et al., 2009), but depending on the type and amount of each gelling agent used, they may change the texture and sensory perception of food (Lucas et al., 2002; Bayarri et al., 2003, Ishihara et al., 2011, Mesquita et al.,

2012). According to Wilson & Brown (1997), Costell et al. (2000) and Bayarri et al. (2007), the higher the concentration of the gelling agents, the lower the perception of taste of the food. This is because increasing the concentration of gelling agents modifies the mechanical properties of the product (Bayarri et al., 2004; Bayarri et al., 2006); thereby, the perception of taste becomes more difficult (Koliandris et al., 2008).

Fruit preserves, such as guava preserves, are largely consumed in Brazil. However, consumption of traditional preserves (with sugar added) is decreasing due to the factors previously described (Menezes, 2011). Therefore, to retain the historical importance of this product and couple it with new market trends, a new product must be developed with similar properties, both sensory and textural, to the traditional product. It is extremely important to study the interactions between the gelling agents used as well as the optimal concentration of the product.

Response surface methodology (RSM) is one of the most commonly used optimization techniques in food science due to its comprehensive theory, high efficiency and simplicity (Pua et al., 2010). RSM encompasses a group of techniques used to study the relationship between one or more measured responses and input variables. RSM can be used in problems that have ingredients and/or processing conditions as variables (Arteaga et al., 1994). It has been successfully applied to optimize food processing operations by many researchers (Frank, 2001; Lee et al., 2006; Mirhosseini et al., 2008). RSM is an appropriate experimental design in applications where several responses are measured for each set of experimental conditions and a model is fitted for each response.

The objective of this study was to optimize and to evaluate the effect of the types of gelling agents and their concentrations on the sensory characteristics and texture of functional sugar-free guava preserves.

2. MATERIALS AND METHODS

2.1. Materials

We used ripe Pedro Sato guava cultivars from a local market, which were processed in the pilot plant of the Department of Food Science at the Federal University of Lavras.

The ingredients used were as follows: fructooligosaccharides (FOS) (Beneo P95, São Paulo, SP, Brazil), thaumatin (Nutramax, Catanduva, SP, Brazil), stevioside (Nutramax, Catanduva, SP, Brazil), sucralose (Nutramax, Catanduva, SP, Brazil), locust bean gum (LBG) (Danisco, São Paulo, SP, Brazil), carrageenan (Danisco, São Paulo, SP, Brazil), low methoxyl pectin (LMP) (Danisco, São Paulo, SP, Brazil), polydextrose (Litesse, São Paulo, SP, Brazil), citric acid monohydrate (Nuclear, São Paulo, SP, Brazil), calcium chloride (Vetec, São Paulo, SP, Brazil) and potassium sorbate (Vetec, São Paulo, SP, Brazil).

The use of fructooligosaccharides (FOS) and polydextrose provided body to the product and made them functional because they are not digested or absorbed in the small intestine by modifying the intestinal habitat, thereby causing an increase in stool production and normalization of stool frequency (Cherbut, 2002; Ribeiro et al., 2003; Rodriguez-Cabezas et al., 2010; Rodriguez et al., 2011; Martínez-Cervera et al., 2012).

2.2. Preparation of guava preserves and experimental design

The different formulations of guava preserves were processed in open stainless steel pots, according to the methodology proposed by Menezes (2011).

A mixture of pulp and polydextrose was heated to 45 °Brix then added to a mixture of gum, pectin LMP and calcium chloride that was previously homogenized under high stirring in water at 80 °C. Cooking was continued until a soluble solids content of 50 °Brix was achieved. FOS diluted 1:1 in water at room temperature was then added, followed by more cooking until a total soluble solids of 65 °Brix was obtained. Citric acid and potassium sorbate were added at the end of the cooking process (diluted 1:1 in water at room temperature) to prevent degradation at the high temperature. The sweeteners were added in this step. The guava preserves were placed in polypropylene containers with the filling performed at a high temperature (85 °C). The containers were then closed, cooled to room temperature and stored in a BOD at 20 °C for later analysis.

All the formulations used the following fixed concentrations: 60.0% guava pulp, 0.025% sucralose, 0.030% stevioside, 0.046% thaumatin, 13.18% FOS, 26.18% polydextrose, 0.2% citric acid, 0.3% calcium chloride and 0.05% potassium sorbate.

2.3. Sensory evaluation

The acceptance tests for flavor, consistency, sweetness and overall liking of the guava preserves were conducted in a laboratory with 60 consumers using a hedonic scale of 9 points (1 = dislike extremely to 9 = like extremely) (Stone & Sidel, 1985).

The samples, approximately 10.0 g each (Acosta et al., 2008), were served in 50mL disposable cups at room temperature, following the order of presentation proposed by Wakeling and MacFie (1995). The cups were coded

with three-digit numbers taken from a table of random numbers. The tests were performed in individual booths under white light.

2.4. Texture profile analysis

The texture profile analyses (TPA) were performed in a Stable Micro Systems Model TA-XT2i texturometer (Goldaming, England) at the following speeds over a 20.0-mm distance: pre-test speed of 1.0 mm/s, test speed of 1.0 mm/s, and post-test speed of 1.0 mm/ (Szczesniak, 1963a; Szczesniak, 1963b). Compression was performed with a cylindrical aluminum probe of 6.0 mm. The parameters analyzed were hardness, cohesiveness and gumminess. The tests were performed in quadruplicate. The analyses were conducted in the jars containing the guava preserves.

2.5. Stress relaxation test

There are several mathematical models that can explain the behavior of viscoelastic food products, but the Maxwell model is used most frequently to describe the behavior of gels and alimentary systems (Morales et al., 2007; Andrés et al., 2008; Bellido & Hatcher, 2009; Khazaei & Mohammadi, 2009).

The Maxwell model involves two simple elements combined in a series to represent different behaviors. The first of these two elements is the ideal elastic element, which can be represented by a spring and has a behavior defined by the elastic constant E. and the second element is the ideal viscous element, which can be represented by a dashpot and has a behavior defined by the viscosity η (Campus et al., 2010).

In the Maxwell model with a constant strain (ε_0), σ describes the tension applied from σ_0 to σ(t) after a time t (Nobile et al., 2007) as follows:

$$\sigma(t) = \varepsilon_0 \left(E.\exp(-\frac{t}{\lambda}) + E_e \right) \quad (1)$$

where E is the elastic modulus of the material, E_e is the equilibrium elastic modulus and λ is the relaxation time given by η/E. Some foods do not follow Maxwell's simplified viscoelastic model, and thus, the description of their behavior requires more complex models. An example of this case is the generalized Maxwell model, which consists of an infinite number of Maxwell models in parallel over a spring.

The stress relaxation curves (stress versus time) can be adjusted by means of equation 2, which provides the viscoelastic parameters of the generalized Maxwell model:

$$\sigma(t) = \varepsilon_0 \left(E_1 \exp(-\frac{t}{\lambda_1}) + E_2 \exp(-\frac{t}{\lambda_2}) + ... + E_e \right) \quad (2)$$

where E_1, E_2 ... are the elastic modulus of the ideal elastic body, E_e is the equilibrium elastic modulus and λ_1, λ_2 … are the relaxation times.

The viscosity of element i can be calculated according to equation 3:

$$\eta_i = E_i \lambda_i \quad (3)$$

The stress relaxation tests were performed in a texturometer (Stable Micro Systems Model TA-XT2i). The samples were cut into cylindrical shapes 2.0 cm in both diameter and height and then compressed to 5.0% of the original height of the sample, with a speed of 1.0 mm/s. The deformation was kept constant for 10.0 minutes, which allowed the stress to reach equilibrium. During that time, the relaxation of tension was measured at a rate of 1.0 measure per second. A 7.0 cm diameter probe cylinder, which had been lubricated to eliminate the influence of friction between the sample and the equipment, was used. Four measurements were performed for each treatment. The nonlinear regression program R (2011) was used to determine the constants of the Maxwell model.

2.6. Experimental design and statistical analysis

This study evaluated the effects of three factors (low methoxyl pectin concentration, locust bean gum concentration and carrageenan concentration). A central composite rotational design (CCRD) with $2^3 + 6 + 4$ points, including axial center points, was applied. The coded and real values of the factors are specified in Table 1.

Table 1 Levels and values of the independent variables of the central composite rotational design (CCRD).

Trials	Coded Variables			Real Variables		
	x_1	x_2	x_3	X_1 (%)*	X_2 (%)*	X_3 (%)*
1	-1	-1	-1	1.5	0.5	0.5
2	+1	-1	-1	2.5	0.5	0.5
3	-1	+1	-1	1.5	1.5	0.5
4	+1	+1	-1	2.5	1.5	0.5
5	-1	-1	+1	1.5	0.5	1.5
6	+1	-1	+1	2.5	0.5	1.5
7	-1	+1	+1	1.5	1.5	1.5
8	+1	+1	+1	2.5	1.5	1.5
9	-1.68	0	0	1.16	1.0	1.0
10	+1.68	0	0	2.84	1.0	1.0
11	0	-1.68	0	2.0	0.16	1.0
12	0	+1.68	0	2.0	1.84	1.0
13	0	0	-1.68	2.0	1.0	0.16
14	0	0	+1.68	2.0	1.0	1.84
15	0	0	0	2.0	1.0	1.0
16	0	0	0	2.0	1.0	1.0
17	0	0	0	2.0	1.0	1.0
18	0	0	0	2.0	1.0	1.0

x_1 = pectin LMP; x_2 = locust bean gum; x_3 = carrageenan.
* percentage relative to the whole system

To enable the adjustment of a regression model, axial points were added to make the number of data points greater than the number of estimated parameters. The results of all analyses were evaluated by the response surface methodology using the software STATISTICA™, Version 8.0 for Windows (StatSoft®).

The criterion used to accept the proposed model were the high coefficient of determination (R^2) and lack-of-fit.

To correlate the rheological properties with the sensory attributes, the Pearson correlation in SAS for Windows, version 5.0 was used.

3. RESULTS AND DISCUSSION

3.1. Sensory evaluation

The results of the sensory evaluation of the functional sugar-free guava preserves are presented in Table 2 (dependency on factor levels). The results of the analysis of variance (*F*-test) are shown in Table 3. The determination coefficients (R^2) were high and varied between 0.80 and 0.92 (properly adjusted to the model); the regression coefficients and their significance are presented in Table 4.

Table 2 Results of the sensory evaluation of the functional sugar-free guava preserves.

Trials	Flavor	Consistency	Sweetness	Overall liking
1	7.02	7.07	7.10	7.12
2	6.82	6.75	6.93	6.63
3	7.15	7.42	6.17	6.83
4	5.65	5.70	5.75	5.83
5	6.93	5.68	6.17	6.02
6	5.70	5.15	5.70	5.45
7	4.87	4.83	5.08	5.05
8	4.85	4.68	5.05	4.92
9	5.87	6.27	5.98	5.93
10	6.57	6.18	6.23	6.13
11	7.27	6.85	7.32	7.15
12	5.18	5.07	5.55	5.30
13	7.27	7.47	7.47	7.30
14	5.22	5.30	5.45	5.13
15	6.78	6.28	6.58	6.45
16	6.20	6.30	5.90	6.18
17	6.17	6.22	6.23	6.22
18	6.02	5.95	6.15	6.00

Table 3 Analysis of variance of the sensory attributes of the functional sugar-free guava preserves.

Factors	df	Flavor		Consistency		Sweetness		Overall liking	
		Mean square	*F*-ratio	Mean square	*F*-ratio	Mean square	*F*-ratio	Mean square	*F*-ratio
1 LMP(L)	1	0.23	0.79	0.60	3.43	0.03	0.30	0.25	2.37
LMP(Q)	1	0.03	0.10	0.03	0.16	0.17	1.52	0.13	1.24
2 LBG(L)	1	4.08	14.00**	1.84	10.44*	3.41	31.32**	2.38	22.41**
LBG(Q)	1	0.03	0.09	0.25	1.43	0.00	0.00	0.01	0.13
3 CAR(L)	1	4.38	15.05**	7.69	43.66**	3.95	36.28**	5.44	51.24**
CAR(Q)	1	0.02	0.07	0.00	0.01	0.00	0.01	0.02	0.16
1 x 2	1	0.00	0.00	0.13	0.74	0.00	0.04	0.00	0.01
1 x 3	1	0.03	0.09	0.23	1.31	0.00	0.01	0.08	0.73
2 x 3	1	0.44	1.50	0.05	0.27	0.02	0.16	0.02	0.20
Error	8	0.29	-	0.18	-	0.11	-	0.11	-
Total	17	-	-	-	-	-	-	-	-
Lack-of-fit	5	0.40	3.57	0.27	10.15	0.17	1.61	0.15	4.36
R^2	-	-	0.80	-	0.88	-	0.90	-	0.91

L- linear; Q- quadratic; R^2- coefficient of determination; *p<0.05; **p<0.01

Table 4 Coefficients of the regression models for the sensory attributes of the functional sugar-free guava preserves.

Coefficients	Flavor	Consistency	Sweetness	Overall liking
β_0	6.30**	6.20**	6.23**	6.22**
β_1	-0.13	-0.21	-0.05	-0.14
β_{11}	-0.05	-0.05	-0.11	-0.10
β_2	-0.55**	-0.37*	-0.50**	-0.42**
β_{22}	-0.05	-0.14	0.00	-0.03
β_3	-0.57**	-0.75**	-0.54**	-0.63**
β_{33}	-0.04	0.01	0.01	-0.04
β_{12}	-0.01	-0.13	0.02	-0.01
β_{13}	0.06	0.17	0.01	0.10
β_{23}	-0.23	-0.08	0.05	-0.05

*$p<0.05$; **$p<0.01$

Figure 1 shows the effects of LBG and carrageenan on the sensory attributes of the functional sugar-free guava preserves. The concentration of low methoxyl pectin was fixed at the value of the center point because it does not show any significant effect on the sensory attributes (Table 4).

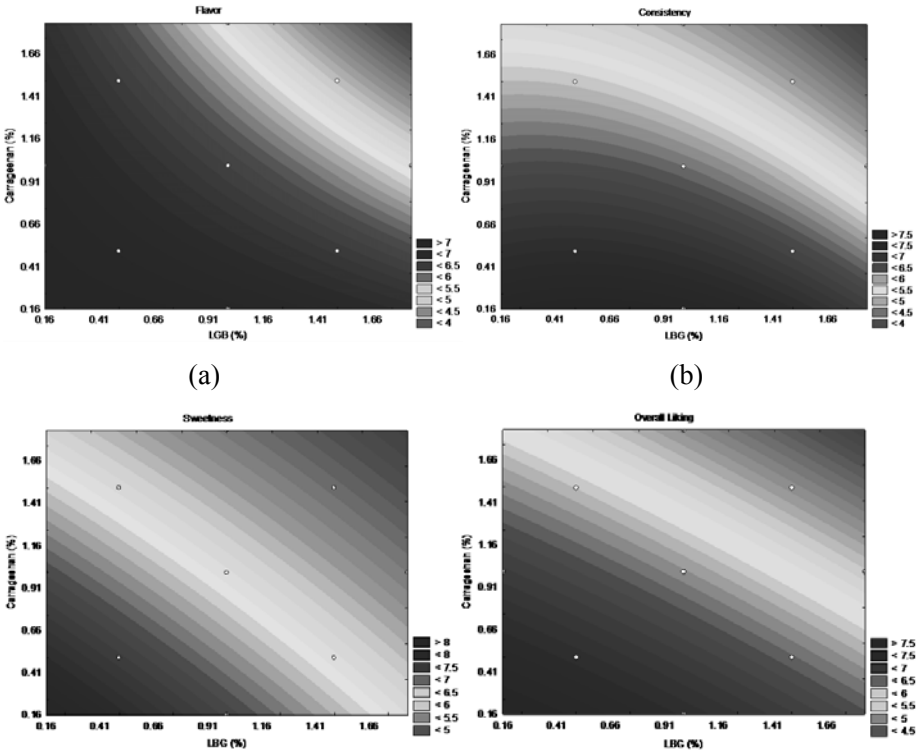


Figure 1 Effect of locust bean gum and carrageenan on the (a) flavor, (b) consistency, (c) sweetness, and (d) overall liking of the functional sugar-free guava preserves.

Based on the mean hedonic ratings of flavor, consistency, sweetness and overall liking, the preserves with lower levels of carrageenan and locust bean gum were more accepted (Figure 1).

Locust bean gum (x_2) and carrageenan (x_3) significantly decreased the flavor attribute. The coefficients of linear regression of both factors were negative (Table 4 and Figure 1a), indicating that as the concentrations of locust bean gum and carrageenan increased, the flavor notes decreased. The perception of flavor is considered to be a combination (in the brain) of two senses, the sense

of smell and the sense of taste. Therefore, flavor can be broken down into two major components, the volatile compounds that are sensed by the olfactory epithelium (aroma) and the non-volatile compounds that are sensed by the taste buds on the tongue (taste). As food is consumed, there are various factors that may influence the release of the volatile components, as well as the tastant. These factors include the breaking of the structure upon mastication and mixing with saliva (Koliandris et al., 2008). For solutions, it has been frequently reported that the intensity of flavor perception decreases with increased viscosity (Baines & Morris, 1989). According Wilson & Brown (1997), Costell et al., (2000) and Bayarri et al. (2007), the higher the concentration of gelling agents, the lower the perception of taste for food. Additionally, according Bayarri et al. (2004) and Bayarri et al. (2006), the concentration of gelling agents modifies the mechanical properties (diffusion) of the gels, influencing the perception of flavor. Figure 1a shows that the highest scores for the flavor attribute are achieved with carrageenan concentrations ranging from 0.16% to 0.91%. As for the locust bean gum, the variation was from 0.16% to 1.0%.

With regard to consistency, it is observed (Table 4 and Figure 1b) that increasing the concentration of carrageenan and locust bean gum reduces the sensory evaluation scores for consistency. Carrageenan had a greater effect on this decrease than locust bean gum (Figure 1b). Formulations with higher carrageenan concentrations exhibited higher values of hardness (Figure 2a). According to Cakir et al. (2012), the development of low-calorie products is often perceived by consumers as less texturally attractive because, according to Rogers et al. (2009), an increase in firmness of a product (by the presence of gelling agents) allows a lower degree of decomposition during mastication, thereby reducing its acceptance. According to Figure 1b, higher scores for the

consistency attribute is achieved with concentrations of carrageenan from 0.16% to 0.41% and concentrations of locust bean gum from 0.16% to 1.0%.

The sweetness was affected negatively by factors independent of the linear terms x_2 (locust bean gum) and x_3 (carrageenan) (i.e., the concentration of carrageenan and locust bean gum in the functional sugar-free guava preserves decreased sweetness). The highest scores for the sweetness attribute were obtained using concentrations of carrageenan and locust bean gum between 0.16% and 0.41% (Figure 1c). Several studies have reported that the concentration of gelling agents reduces the perception of the sweetness of the sweetener used (Mälkki et al., 1993, Cook et al., 2003; Bayarri et al., 2003; Bayarri et al., 2007; Koliandris et al., 2008; Holm et al., 2009). Bayarri et al. (2003) studied the sweetness of gels made of gellan gum and carrageenan. They reported that there is a greater perception of sweetness with lower concentrations of these gums. According to Gibson (1992), a gel with less firmness disintegrates more easily in the mouth, releasing the sweetener faster. Cook et al. (2003) noted that the sweetness of sucrose was reduced in guar solutions, while that of aspartame was reduced in λ-carrageenan solutions. Both the nature of the thickening agent (carboxymethylcellulose, xanthan and pectin) and viscosity influenced the sweetness of apple juice (Walker & Prescott, 2000). Increased concentrations of the gelling agent increased the firmness (instrumental) and decreased the sweetness perception in pectin gels (Lundgren et al., 1986), which were similarly observed for carrageenan, alginate, agar (Chai et al., 1991) and gellan gels (Costell et al., 2000).

Regarding overall liking, the same response as with the other attributes evaluated was observed; the concentration of the gums in this study reduced overall liking. Figure 1d shows that higher scores for this attribute are obtained

with concentrations of locust bean gum and carrageenan ranging between 0.16% and 0.41%.

3.2. Evaluation of the texture profile analysis

The results of the texture profile parameters of the functional sugar-free guava preserves are shown in Table 5 (dependency on factor levels). The *F*-test results are shown in Table 6. The determination coefficients (R^2) were high and varied between 0.79 and 0.92 (properly adjusted to the model). The regression coefficients and their significance levels are listed in Table 7.

Table 5 Results of the texture profile parameters of the functional sugar-free guava preserves.

Trials	Hardness (N)	Cohesiveness	Gumminess (N)
1	1.17	0.40	0.47
2	1.56	0.36	0.57
3	2.15	0.30	0.65
4	1.75	0.34	0.59
5	2.60	0.43	1.10
6	4.06	0.43	1.72
7	2.94	0.34	1.00
8	4.28	0.35	1.50
9	2.87	0.31	0.89
10	3.86	0.32	1.23
11	1.47	0.49	0.72
12	4.58	0.38	1.72
13	0.82	0.33	0.27
14	3.17	0.33	1.03
15	2.54	0.35	0.88
16	4.00	0.37	1.48
17	3.76	0.36	1.35
18	2.60	0.39	1.03

Table 6 Analysis of variance parameters of the texture profiles of the functional sugar-free guava preserves.

Fatores	df	Hardness		Cohesiveness		Gumminess	
		Mean square	*F*-ratio	Mean square	*F*-ratio	Mean square	*F*-ratio
1 LMP (L)	1	1.44	3.07	0.00	0.14	0.22	2.78
LMP (Q)	1	0.00	0.00	0.00	8.21*	0.03	0.34
2 LBG (L)	1	3.54	7.56*	0.02	42.30**	0.18	2.22
LBG (Q)	1	0.17	0.36	0.01	20.27**	0.00	0.01
3 CAR (L)	1	9.19	19.61**	0.00	3.64	1.37	17.24**
CAR (Q)	1	2.91	6.22*	0.00	4.83	0.47	5.95*
1 x 2	1	0.11	0.23	0.00	1.92	0.01	0.12
1 x 3	1	0.99	2.11	0.00	0.01	0.14	1.80
2 x 3	1	0.05	0.10	0.00	0.38	0.04	0.45
Error	8	0.47	-	0.00	-	0.08	-
Total	17	-	-	-	-	-	-
Lack-of-fit	5	0.40	0.69	0.0004	1.10	0.08	1.07
R^2	-	-	0.83	-	0.92	-	0.79

L- linear; Q- quadratic; R^2- coefficient of determination; *p<0.05; **p<0.01

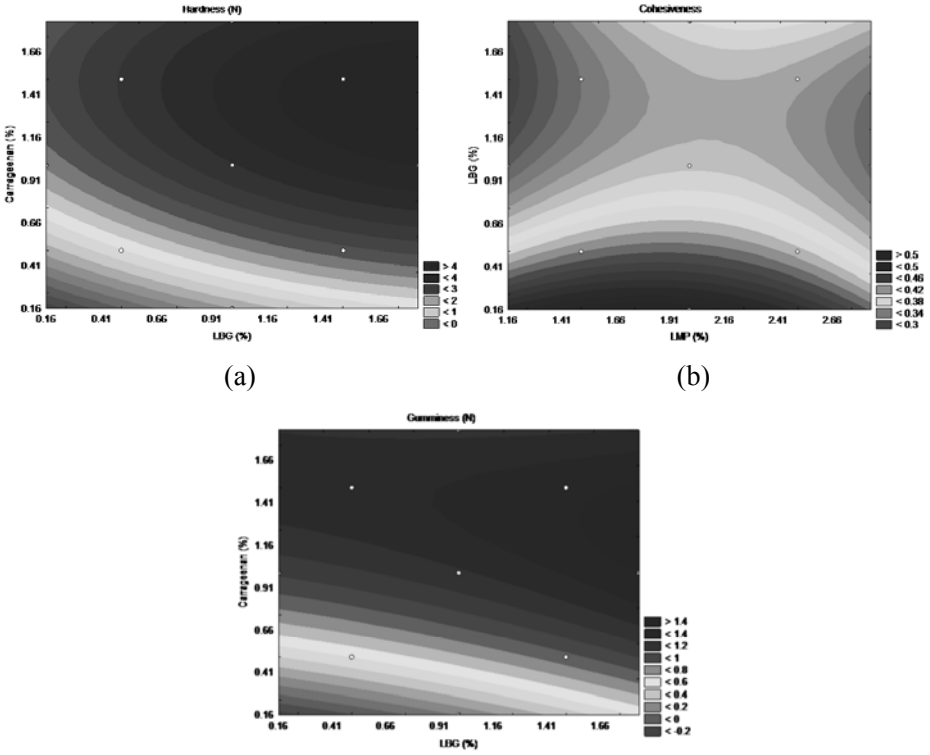
Table 7 Coefficients of the regression models for the texture parameters of the functional sugar-free guava preserves.

Coefficients	Hardness (N)	Cohesiveness	Gumminess (N)
β_0	3.23**	0.37**	1.19**
β_1	0.32	0.002	0.13
β_{11}	0.01	-0.02**	-0.05
β_2	0.51*	-0.03**	0.11
β_{22}	-0.12	0.03**	0.01
β_3	0.82**	0.01	0.32**
β_{33}	-0.48*	-0.01	-0.19*
β_{12}	-0.12	0.01	-0.03
β_{13}	0.35	0.001	0.13
β_{23}	-0.08	-0.004	-0.07

*$p<0.05$; **$p<0.01$

TPA is a method to evaluate sensory properties. The test consists of uniaxially compressing the food (study sample) twice in a reciprocating motion to mimic the action of the mandible. Therefore, an initial compression and relaxation followed by a second compression were performed during testing. This test yields a graph of force versus time, from which the texture parameters are calculated (Honikel, 1998; Lau et al., 2000; Bourne, 2002; Herrero et al., 2007).

Figure 2 shows the contour curves of the texture profile parameters of the functional sugar-free guava preserves. The value of low methoxyl pectin was fixed at the center point for the parameters of hardness and gumminess because this independent variable did not significantly affect these parameters (Table 7). For the analysis of cohesiveness, the level of carrageenan was set at the central point because it did not affect this parameter (Table 7).



(a) (b)

(c)

Figure 2 Contour curves of the texture profile parameters of the functional sugar-free guava preserves: (a) hardness, (b) cohesiveness, and (c) gumminess.

For hardness, only the linear effects of locust bean gum (x_2) and carrageenan (x_3) and the negative quadratic effect of carrageenan (x_3) were significant (Table 7 and Figure 2a). That is, the concentration of locust bean gum in the guava preserves increases hardness, and higher hardness values were obtained with LBG concentrations ranging from 1.16% to 1.66% (Figure 2a). LBG does not form gel on its own (Fernandes et al., 1991). However, when it is combined with other polysaccharides, such as carrageenan and pectin, locust

bean gum can form gels (Azero & Andrade, 2006; Hernandez et al., 2001; Bourbon et al., 2010). The increased hardness induced by increasing the amount of locust bean gum in the product may be due to complex interactions involving all the gelling agents used in this study, which make the gel networks more rigid. Similar results were obtained by Alloncle & Doublier (1991), who studied starch gels and hydrocolloids, and Rocha et al. (2009), who studied mixed gels of whey protein and locust bean gum. These authors concluded that the increase in hardness was caused by the increase in locust bean gum concentration, which was due to the modification of the balance between aggregation and separation of the gel. Arocas et al. (2009) studied white sauces with added starch, xanthan gum and locust bean gum, and they observed the same behavior with locust bean gum and that the concentration of starch increased the consistency of the dressing (measured in a rheometer). With regard to carrageenan, it increased hardness up to maximum concentrations between 1.0% and 1.41% (Figure 2a). Beyond these concentrations, hardness decreases. Spagnuolo et al. (2005) reported that the carrageenan molecule is very flexible and may form a more ordered structure in the form of a double helix, which may lead to gel formation at high concentrations. The gelation process is highly influenced by many factors, such as the type and concentration of salts in the solution, cooling and heating rates, concentration of the hydrocolloid and the presence of other biopolymers. Modifications of these factors greatly affect the gelling and the rheological properties of the gels (Baeza et al., 2002). In this study, we used 0.3% $CaCl_2$ to promote gelling of both the low methoxyl pectin and carrageenan. Increasing the concentration of carrageenan in the system destructured the double helices of this gelling agent (by binding of the sulphated groups of carrageenan to calcium), thus affecting the balance of the attractive and repulsive forces between the molecules, causing decreased rigidity of the gel

(Montero & Pérez-Mateos, 2002). Karim et al. (2009) evaluated the effect of carrageenan in tofu and also observed a decrease in hardness with increasing carrageenan content. These authors attributed this fact to the way protein interacts with calcium and other constituents (e.g., phytic acid) in soy milk and anions that form the microstructure that determines the hardness of tofu, which together with carrageenan, causes the gel strength to decrease.

Low methoxyl pectin (x_1) had a negative quadratic effect on cohesiveness (Table 7 and Figure 2b). That is, increasing pectin concentration increased cohesiveness up to a maximum value and then decreased, which was observed at concentrations between 1.16% and 2.0% (Figure 2b). The decrease may be due to syneresis because the concentration of pectin gels was strong enough to expel water from the system (Thrimawithana et al. 2010), making the gel more prone to disintegration in the first compression cycle (Extralab, 2010). As for locust bean gum (x_2), it had a linear and positive quadratic effect on this parameter of the texture profile. As shown in Figure 2b, higher values of cohesiveness are achieved with lower concentrations of locust bean gum (0.16% to 0.30%).

Gumminess was significantly influenced (positive linear effect and negative quadratic effect) by carrageenan (x_3) (Table 7 and Figure 2c). Increasing the concentration of carrageenan in the functional sugar-free guava preserves increased gumminess to a threshold value then subsequently decreased. The concentration of carrageenan in the functional sugar-free guava preserves an optimal region of hardness (Figure 2c) at concentrations ranging from 1.16% to 1.66%. At higher levels, there was a decrease in hardness, confirming the results obtained for the hardness parameter.

3.3. Evaluation of the stress relaxation test

The results of the stress relaxation tests on the functional sugar-free guava preserves are presented in Table 8 (dependency on factor levels). The *F*-test results are shown in Table 9. The determination coefficients (R^2) were high and varied between 0.79 and 0.83 (properly adjusted to the model). The regression coefficients and their significance are listed in Table 10.

Table 8 Results of the stress relaxation tests on the functional sugar-free guava preserves.

Trials	E_e	E_1	λ	η
1	5.62	6.95	69.67	483.94
2	11.47	8.87	65.33	574.12
3	6.81	6.86	57.43	391.77
4	8.79	6.88	78.35	533.44
5	13.55	23.93	96.65	2321.60
6	43.78	26.99	130.61	3444.81
7	23.96	18.02	117.55	2135.31
8	18.56	18.81	104.00	1980.68
9	32.63	28.39	83.00	2343.18
10	36.53	25.39	67.10	1652.01
11	14.72	25.77	169.82	4309.38
12	23.23	17.16	78.01	1340.97
13	2.31	3.30	57.96	191.13
14	36.08	30.98	132.22	3952.80
15	14.44	13.26	116.94	1489.66
16	19.88	13.57	81.72	1090.58
17	16.59	12.27	95.52	1149.97
18	11.08	9.52	124.94	1179.34

Ee: equilibrium elastic modulus (N/m^2); E1: elastic modulus of the ideal elastic body (N/m^2); λ: relaxation time (s); η: viscosity ($N/m^2.s$)

Table 9 Analysis of variance of the properties of the Maxwell model of the functional sugar-free guava preserves.

Factors	df	E_e		E_1		λ		η	
		Mean square	*F*-ratio	Mean square	*F*-ratio	Mean square	*F*-ratio	Mean square	*F*-ratio
1 LMP(L)	1	112.67	1.84	0.04	0.00	7.75	0.01	113	0.00
LMP(Q)	1	311.67	5.09	157.14	5.84	1875.48	3.21	82775	0.14
2 LBG(L)	1	0.30	0.00	68.85	2.56	1857.16	3.18	3359196	5.70
LBG(Q)	1	3.92	0.06	32.56	1.21	330.39	0.57	1766286	3.00
3 CAR(L)	1	1124.88	18.39**	803.27	29.86**	6718.77	11.51**	14816775	25.14**
CAR(Q)	1	2.90	0.05	0.07	0.00	327.54	0.56	145380	0.25
1 x 2	1	194.95	3.19	2.18	0.08	62.03	0.11	187994	0.32
1 x 3	1	36.12	0.59	0.45	0.02	1.84	0.00	67848	0.12
2 x 3	1	22.21	0.36	18.00	0.67	5.26	0.01	287877	0.49
Error	8	61.18	-	26.90	-	583.80	-	589364	-
Total	17	-	-	-	-	-	-	-	-
Lack-of-fit	5	89.69	6.56	41.01	12.08	699.74	1.79	923822	28.93
R^2	-	-	0.79	-	0.83	-	0.71	-	0.81

L- linear; Q- quadratic; R^2- coefficient of determination; *p<0.05; **p<0.01
Ee: equilibrium elastic modulus (N/m^2); E1: elastic modulus of the ideal elastic body (N/m^2); λ: relaxation time (s); η: viscosity ($N/m^2.s$)

Table 10 Coefficients of the regression of the Maxwell model properties of the functional sugar-free guava preserves.

Coefficients	Ee	E1	λ	η
β_0	15.91**	12.54**	105.16**	1271.55**
β_1	2.87	0.05	0.75	2.88
β_{11}	4.97	3.53	-12.19	81.01
β_2	-0.15	-2.25	-11.67	-496.17
β_{22}	-0.56	1.61	5.12	374.23
β_3	9.08**	7.67**	22.19**	1042.06**
β_{33}	-0.48	0.07	-5.10	107.37
β_{12}	-4.94	-0.52	-2.78	-153.29
β_{13}	2.12	0.24	0.48	92.09
β_{23}	-1.67	-1.50	-0.81	-189.70

Ee: equilibrium elastic modulus (N/m^2); E1: elastic modulus of the ideal elastic body (N/m^2); λ: relaxation time (s); η: viscosity ($N/m^2.s$); *p<0.05; **p<0.01

Figure 3 shows the contour curves of the Maxwell model properties (equation 1) of the functional sugar-free guava preserves. This model was chosen because there was a considerable improvement in R^2 when the generalized Maxwell model of two elements of a spring in parallel was tested. The low methoxyl pectin concentration was set at the central point for all Maxwell model parameters because this independent variable did not significantly affect any of the parameters (Table 10).

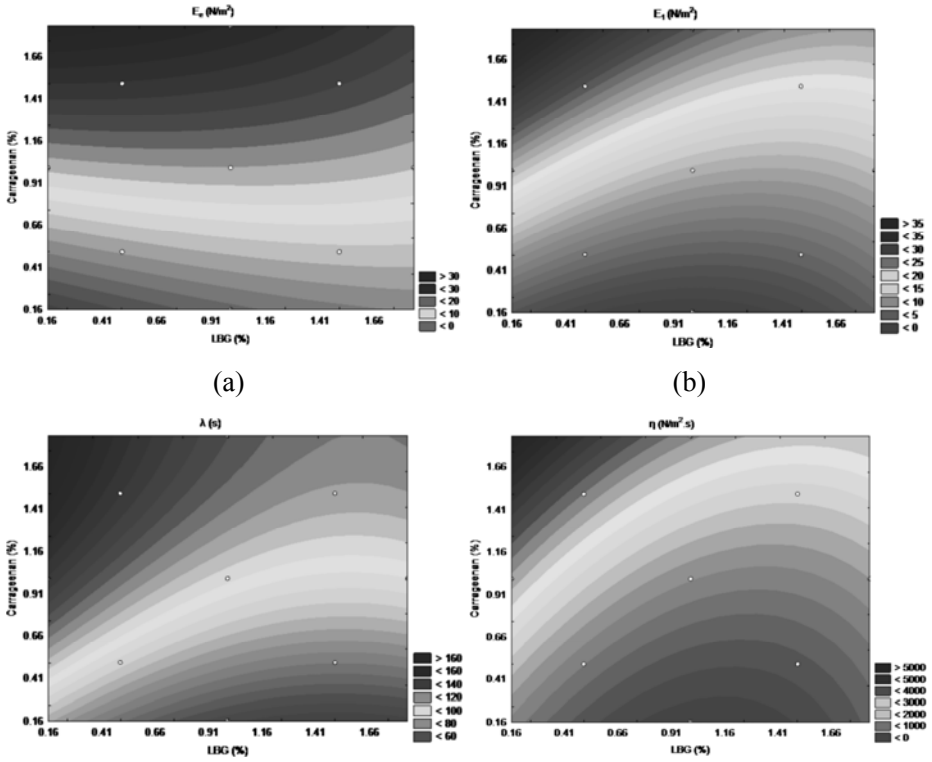


Figure 3 Contour curves of the stress relaxation of the Maxwell model parameters of the functional sugar-free guava preserves: (a) equilibrium elastic modulus (E_e); (b) elastic modulus of the ideal elastic body (E_1); (c) relaxation time (λ); and (d) viscosity (η).

It was observed that the only variable that was linearly independent and positively affected all the Maxwell model properties was carrageenan (x_3). The concentration of this gelling agent increased the rigidity of the material since the properties E_e and E_1 quantified this rigidity (Peleg, 1987; Rodriguez-Sandoval, 2009). The property λ indicated that the greater the relaxation time, the greater the elastic behavior and the firmer the material (Nobile et al., 2007; Bhattacharya, 2010; Campus et al., 2010). The property η indicated that the

higher its value, the more solid the material (Rodriguez-Sandoval, 2009). These results are similar to those obtained from the texture profile analysis. Higher values of E_e, E_1 and η were obtained with concentrations of carrageenan between 1.66% and 1.84% and higher values of λ were obtained at concentrations between 1.41% and 1.84% (Figures 3a-d).

3.4. Correlation between the sensory and rheological parameters

The Pearson correlation coefficient between the sensory and rheological properties of the functional sugar-free guava preserves is shown in Table 11.

Table 11 Pearson correlation coefficient between the sensory and rheological properties.

	Flavor	**Consistency**	**Sweetness**	**Overall liking**
Hardness (N)	-0.66**	-0.71**	-0.75**	-0.74**
Cohesiveness	0.22	-0.06	0.29	0.17
Gumminess (N)	-0.60**	-0.71**	-0.66**	-0.69**
E_e (N/m^2)	-0.53*	-0.57*	-0.54*	-0.63**
E_1 (N/m^2)	-0.34	-0.50*	-0.38	-0.49*
λ (s)	-0.24	-0.40	-0.15	-0.27
η ($N/m^2.s$)	-0.27	-0.43	-0.24	-0.37

* $p<0.05$; ** $p<0.01$

Hardness, gumminess and E_e were negatively correlated with all the sensory attributes studied, while E_1 was the only negatively correlated attribute with overall liking. Cohesiveness, λ and η were not correlated to any of the sensory attributes studied. These results indicate that increasing the texture parameters (i.e., the concentration of gelling agents in the product) decrease the

scores of the sensory attributes decrease, indicating that consumers prefer a functional sugar-free guava preserve with low concentrations of gelling agents. As reported in several studies (Gibson in 1992, Costell et al., 2000, Ronda et al., 2009; Rogers et al., 2009; Thrimawithana et al., 2010), the increase in gelling agents causes an increase in the rigidity of the gel but makes it more brittle and cohesive, therefore making it difficult to dissolve in the mouth and reduces the product's acceptance.

4. CONCLUSION

The present study indicated that response surface methodology was a useful experimental technique in the evaluation of the effects and appropriate concentrations of gelling agents on the texture and sensory characteristics of functional sugar-free guava preserves. The results indicated that the independent variables of carrageenan and locust bean gum had the most influence on the texture and sensory characteristics of the functional sugar-free guava preserves and that low methoxyl pectin can be used at a concentration of 2.0% together with locust bean gum and carrageenan. We also conclude that higher sensory scores were achieved at low concentrations of locust bean gum and carrageenan. In relation to the rheological properties, the highest values were obtained with high concentrations of the two gums. Negative correlations were observed between the sensory attributes and rheological properties, indicating that there is greater acceptability of functional guava preserves without added sugar and with concentrations of locust bean gum and carrageenan ranging from 0.16% to 0.41%.

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