



**UNIVERSIDADE ESTADUAL DE CAMPINAS  
FACULDADE DE ENGENHARIA DE ALIMENTOS**

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**AVALIAÇÃO DAS PROPRIEDADES FÍSICO-QUÍMICAS E FUNCIONAIS NO  
PROCESSAMENTO INTEGRAL DE UMÊ (*Prunus mume*)**

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DOUTOR EM TECNOLOGIA DE ALIMENTOS

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Este exemplar corresponde à versão final da tese AVALIAÇÃO DAS PROPRIEDADES FÍSICO-QUÍMICAS E FUNCIONAIS NO PROCESSAMENTO INTEGRAL DE UMÊ (*Prunus mume*) defendida por ERNESTO QUAST, aprovada pela comissão julgadora em \_\_\_\_/\_\_\_\_/\_\_\_\_ e orientado pelo Prof. Dr. Flávio Luís Schmidt.

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À minha esposa Leda Battestin Quast

e minha filha Catarina,

Com muito amor e carinho,

Dedico



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## **Resumo geral**

O umê (*Prunus mume*) é um fruto de caroço que apresenta alta rusticidade e adaptabilidade agrícola, sendo muito estudado como porta-enxerto de pêssegos, nectarinas e ameixas. Embora no Brasil não existam estudos visando o processamento dos frutos, o umê é muito consumido e apreciado em países asiáticos devido às propriedades nutracêuticas relacionadas ao consumo de concentrados de frutos verdes ou flores, por isso, o objetivo do presente trabalho é possibilitar o aproveitamento desta matéria prima pela elaboração de um produto apreciado pelo consumidor brasileiro, com apelo saudável. Foram utilizados frutos de diferentes localidades do Estado de São Paulo, caracterizados segundo suas dimensões, massa e rendimento no despolpamento, bem como seus aspectos botânicos. Os frutos tiveram a sua maturação acompanhada pela cor e após maduros, foram branqueados termicamente, despolpados, desaerados e envasados a quente na planta piloto de Frutas e Hortaliças da Faculdade de Engenharia de Alimentos da UNICAMP. Foram realizadas análises de textura instrumental em frutos verdes e maduros. Na Universidade Estadual de Ponta Grossa (UEPG) foram realizadas análises químicas de compostos fenólicos e da atividade antioxidante dos frutos colhidos em cinco diferentes localidades do Estado de São Paulo e em diferentes graus de maturação, bem como as análises reológicas. As dimensões dos frutos (2,0 a 3,6 cm de diâmetro) e suas massas foram significativamente inferiores (6 a 16 gramas) aos valores relatados em trabalhos científicos de autores asiáticos. Os frutos não apresentaram variação significativa do teor de compostos fenólicos e da atividade antioxidante durante a maturação, a partir de sua completa maturação fisiológica (88 dias após a floração), cujos valores foram de 147 a 226 mg catequina/ g base seca e 21 a 34 µMol Trolox/ g base seca, respectivamente. A reologia foi realizada para polpa de umê nas concentrações de 6 a 9 °Brix, nas temperaturas de 15 a 75 °C. O comportamento reológico da polpa de umê se mostrou independente do tempo, com comportamento não-Newtoniano e pseudoplástico (Herschel-Bulkley). Apresentou bom ajuste aos modelos reológicos de Herschel-Bulkley, Casson, Bingham e Lei da Potência. A equação de Arrhenius possibilitou o cálculo da energia de ativação para diferentes concentrações de polpa. A consistência da polpa de umê é altamente influenciada pela concentração dos sólidos solúveis do produto. A micro estruturação da polpa com alginato, para adição ao suco clarificado de umê mostrou-se estável com a elevação do pH para valores superiores a 3,5.

## **Abstract**

*Prunus mume* is a stone fruit that exhibits high robustness and agriculture adaptability. Its use is being extensively studied as rootstocks for peaches, nectarines and plums. Although in Brazil there are no studies for mume fruit processing, in Asian countries this fruit is widely consumed and appreciated due to the nutraceutical properties related to the consumption of concentrated or processed green-stage fruits or flowers. The present work aim is to produce a highly acceptable product with health appeal. Fruits from different locations in São Paulo State were characterized according to their size, weight and yield pulping, as well as its botanical characteristics. Fruits were evaluated during maturation by color and after fully-ripe they were thermally blanched, pulped, deaerated and hot filled in the pilot plant of Fruits and Vegetables of the Faculty of Food Engineering at UNICAMP. Instrumental texture analyses of green and ripe fruits were performed. At the State University of Ponta Grossa (UEPG) were done chemical analysis of total phenolic compounds and antioxidant activity of fruits collected in 5 different locations in the State of São Paulo and at different stages of maturity, as well as rheological analyses. The fruit dimensions (2.0-3.6 cm diameter) and their mass were significantly lower (6-16 g) than values reported in scientific studies done in Asian countries. Total phenolic content and antioxidant activity did not vary during maturation of the fruit, from its full physiological maturity (88 days after flowering), with values of 148-226 mg catechin/ g dry basis and 21-34 mMol Trolox/ g dry basis, respectively. Pulp concentrations at 6-9 °Brix were used to study the rheological properties from 15 to 75 °C. The rheological behavior of mume pulp is non-Newtonian pseudoplastic (Herschel-Bulkley). Herschel-Bulkley, Casson, Bingham and Power Law rheological models described well the mume pulp behavior. The Arrhenius equation was used to calculate the activation energy for different concentrations of pulp and was shown that consistency is highly influenced by soluble solids concentration. Micro structured particles of pulp with alginate for adding into clarified mume juice just showed stability with pH increase over 3.5.

## Introdução geral

O presente trabalho mostra uma nova alternativa para a produção de frutas. Embora a cultura estudada, o umê (*Prunus mume*) seja conhecido quase que exclusivamente entre descendentes asiáticos, existe grande potencial de plantio e crescimento da cultura no Brasil, cujo manejo é semelhante à cultura do pêssego e da ameixa. As características organolépticas do fruto de umê, que apresenta elevada acidez e amargor acima do aceito pela maior parte dos consumidores, fazem com que a aptidão deste fruto seja o processamento industrial, onde as características originais dos frutos podem ser ajustadas.

Outra importante característica do umê é sua rusticidade agrícola e facilidade de adaptação. Sendo do gênero *Prunus*, o umê apresenta alta compatibilidade para ser utilizado com porta-enxerto de outras culturas de maior importância econômica no Brasil, como o pêssego (*Prunus persica*) e a ameixa japonesa (*Prunus salicina*). As pesquisas para o uso do umê como porta-enxerto baseiam-se na sua baixa susceptibilidade a doenças, podendo ser cultivado até mesmo comercialmente sem o uso de defensivos químicos. Porém, no Brasil não existem trabalhos científicos para o aproveitamento dos frutos. No mundo, os estudos em sua maior parte têm como objetivos a produção de extratos concentrados, para fins medicinais, ou o consumo na forma tradicional em conserva, geleia ou licor.

Primeiramente, o trabalho teve como principal objetivo a caracterização e avaliações físico-químicas e reológicas da polpa de umê para a produção de suco. Visto que o sabor lembra o pêssego, pode-se vislumbrar um *blend* de umê com pêssego, com algumas vantagens nutricionais devido ao poder antioxidante do umê. Em seguida, avaliou-se a aceitação do néctar de umê como um produto diferenciado, visto que o mercado de sucos encontra-se em expansão, com o lançamento de novos sabores de frutas. Além disso, foi desenvolvido um produto contendo polpa micro reestruturada, de modo a fornecer uma experiência sensorial diferente do que se encontra atualmente na maior parte das bebidas à base de frutas.

O presente trabalho está dividido em artigos que serão submetidos, ou que já foi publicado em revistas científicas.

O primeiro capítulo é uma **Revisão Bibliográfica** reunindo informações de trabalhos científicos realizados mundialmente, para facilitar o entendimento da importância e do potencial da cultura do umê na produção agrícola nacional.

O segundo capítulo refere-se ao artigo intitulado: “**Fruit characteristics of *Prunus mume* from different regions for human consumption**”. Neste artigo foram analisados frutos provenientes de diferentes regiões do Estado de São Paulo, a fim de verificar a variação entre as cultivares trazidas há mais de vinte anos por descendentes de japoneses e chineses no Brasil e comparar os frutos de umê nacionais com aqueles dos países asiáticos, onde os trabalhos de melhoramento genético visam o aumento do rendimento, produtividade e propriedades nutracêuticas. Além da caracterização do fruto, foram realizadas caracterizações florais visando montar um banco de dados genético da espécie no Brasil.

O capítulo 3 refere-se ao artigo “***Prunus mume* – fruit characteristics and phenolic content capacity**”, publicado na revista internacional **Fruit Processing**. O teor de compostos fenólicos foi analisado para diferentes partes de frutos branqueados ou não e expostos ao ar por períodos diferentes, sendo observado que o caroço e a pele apresentam maior teor de compostos fenólicos, quando comparado à polpa. Além disso, foi possível notar que os frutos devem ser branqueados antes do despolpamento, para que haja um menor decréscimo do teor de compostos fenólicos, principalmente na polpa do fruto.

O artigo “**Chemical characteristics and phenolic content of mume fruits collected at different locations and at different maturity stages in Southeast Region of Brazil**” é apresentado no capítulo 4 e deverá ser submetido à revista Ciência e Tecnologia de Alimentos. Neste artigo, objetivou-se conhecer melhor as características de diferentes cultivares de umê plantadas na Região Sudeste do Brasil. Foram estudadas as alterações da atividade antioxidante de frutos fisiologicamente maduros (verdes para o consumo) até sua completa maturação. Estes resultados podem permitir destacar as diferenças das propriedades antioxidantes atribuídas ao consumo de frutos verdes de umê, largamente

estudadas, com as características nutricionais e funcionais de frutos maduros. Estes resultados podem auxiliar no direcionamento de trabalhos futuros para a valorização de alimentos produzidos a partir de frutos maduros de umê, bem como trabalhos de melhoramento genético, a partir de variedades nacionais ou de outros países.

O artigo intitulado: “***Prunus mume* pulp rheological evaluation**” (capítulo 5) deverá ser submetido à revista Journal of Food Engineering. Neste artigo, o comportamento reológico da polpa de umê foi avaliado em diferentes concentrações, visando fornecer subsídios técnicos para o processamento industrial de produtos formulados a partir da polpa concentrada de umê.

No capítulo 6 intitulado: “**Micro reestruturação da polpa de umê e adição ao suco clarificado**” foram estudados os parâmetros para a produção de partículas de polpa estáveis às condições de processamento e estocagem do néctar de umê. Posteriormente, estas partículas podem ser adicionadas ao néctar de umê, para avaliação de sua aceitação.

Dessa forma, foi possível propor a elaboração de um produto inovador, a partir de um fruto com grande potencial de expansão agrícola, forte apelo de saudabilidade e com características sensoriais agradáveis ao consumidor de néctares de frutas.

## **Revisão bibliográfica**

*Prunus mume* Siebold et Zucc. pertence à família *Rosaceae*. Suas flores e frutos são bastante apreciados pelo sabor na China, Coréia e Japão. Porém, sua importância tem crescido na área da saúde, devido a muitos benefícios à saúde, como a melhoria da fluidez do sangue, que reduz a possibilidade de problemas cardíacos e substâncias capazes de combater a formação de radicais livres e a multiplicação de células cancerosas. Estes benefícios estão relacionados com o consumo do fruto verde ou de produtos processados de umê (DAOZONG et al.,2010).

Nos países asiáticos, o consumo do fruto de umê é muito apreciado na forma de uma conserva salgada, produzida a partir de frutos ainda verdes. Esta conserva é chamada *Ume-boshi*. Outros produtos produzidos em geral de forma artesanal, são o licor de umê (*Ume-shu*), o fruto desidratado e o concentrado de suco de fruta (TSUBAKI; OZAKI e AZUMA, 2010). No Brasil, o fruto de umê é mais consumido na forma de conserva salgada ou licor, mais conhecido e apreciado entre os descendentes asiáticos.

O umê é cultivado comercialmente em países como Japão, China e Coréia e apresenta uma produtividade média de 6,7 ton/ha (JUN e CHUNG, 2008). Topp, Noller e Russel (2007) realizaram um levantamento da produção, comercialização e consumo de umê no mundo e fizeram recomendações técnicas e econômicas para o fomento da cultura na Austrália. Embora Campo Dall'Orto et.al. (1998), tenham relatado diversas vantagens agronômicas para o cultivo do umê no Brasil, tais como a menor susceptibilidade a doenças comuns em culturas de frutos de caroço e a redução do porte da planta, que possibilita o maior adensamento da cultura, não existem trabalhos científicos na área de processamento de alimentos para o aproveitamento do umê em grandes escalas de produção.

Os frutos de umê apresentam comportamento tipicamente climatérico, ou seja, são capazes de continuar o processo de maturação mesmo após destacados da árvore, desde que já tenham atingido a sua maturidade fisiológica. Durante a maturação ocorre o amarelecimento da casca, amolecimento da polpa, diminuição da acidez total, aumento do

teor de açúcar, formação de substâncias voláteis, aumento da taxa de produção de dióxido de carbono e de etileno (ABDI et al., 1997).

No Brasil, a maior parte dos trabalhos científicos publicados relacionados com umê levam em consideração a elevada rusticidade agrícola, elevada resistência à pragas e doenças, elevada adaptabilidade e diminuição do vigor da planta, permitindo maior adensamento das culturas e portanto, um aumento na produção, com maior número de plantas/ha e consequentemente, maior produção de frutos/ha (MAYER, PEREIRA e SANTOS, 2005; MAYER, PEREIRA e MORO, 2008). Nestes trabalhos, o foco é o uso do umê como porta-enxertos de outros frutos de caroço como o pêssego, nectarina e ameixa.

Algumas pesquisas revelaram um elevado poder antioxidante do extrato etanólico de flores de umê, principalmente pela presença de ácidos clorogênicos (SHI et al., 2009). No entanto, não foram realizados testes *in vivo* para comprovar os efeitos destes compostos no metabolismo humano.

Entre as propriedades nutracêuticas, atribui-se ao suco concentrado de umê a capacidade de aumentar a fluidez do sangue humano, em parte pela presença de ácidos orgânicos em elevada quantidade e de uma substância chamada mumefral (1-[5-(2-formilfural)metil] di-hidrogenio 2-hidroxipropano-1,2, 3-tricarboxilato), formado junto com 5-hydroxymethyl-2-furfural (HMF) durante o aquecimento do extrato de umê para a sua concentração (CHUDA et al., 1999), o que pode auxiliar na prevenção de doenças cardiovasculares (UTSUNOMIYA et l., 2002), além de exercer ação anti-câncer (ADACHI et al., 2007; MORI et al., 2007). JEONG et al. (2006) isolaram um composto na fração n-hexano do extrato metanólico de umê, de fórmula molecular C<sub>19</sub>H<sub>22</sub>O<sub>6</sub>, que inibiu o desenvolvimento de células cancerosas humanas e apresentou pouco efeito sobre células saudáveis. Outras pesquisas mostram que a ingestão de extrato concentrado de frutos de umê pode inibir a formação endógena de nitrosaminas, substâncias carcinogênicas que podem ser formadas no organismo humano após uma refeição rica em aminas e nitratos (CHOI, CHUNG & SUNG, 2002).

Produtos desidratados de umê ou de ameixa podem ser visualmente muito parecidos, porém, o preço da conserva de umê é mais elevado. Dessa forma, pesquisas são realizadas para evitar a fraude de conservas de umê (NG et al., 2005).

Grande parte do apelo saudável dos frutos é decorrente do alto teor de compostos fenólicos, que pode ser quantificado analiticamente pelo método de Folin-Ciocalteau, utilizando-se ácido gálico ou catequina como padrões para expressão dos resultados. RUPASINGHE; JAYASANKAR e LAY (2006) estudaram a correlação do teor de compostos fenólicos totais e a atividade antioxidante de genótipos de ameixa europeia e encontraram uma relação direta, que depende do tipo de composto fenólico e este, do fruto, cultivar, localidade e dos tratos culturais empregados.

No processamento de alimentos, a adição de extrato de umê pode inibir a formação de radicais livres na fase de iniciação da oxidação de óleos e gorduras insaturadas em carnes durante o armazenamento. A diminuição da velocidade de formação de radicais livres, responsáveis pela reação de quebra das cadeias de triglicerídeos que compõe a maior parte dos óleos e gorduras e consequente menor formação de ácidos graxos livres, podem evitar a formação do odor de “warmed-over flavor” em produtos cárneos durante a estocagem, após seu processamento térmico. JO et al. (2006) verificaram que a aplicação de extrato de umê em frangos pré-cozidos e resfriados diminui a incidência de formação de odores desagradáveis pela inibição de etapas importantes na oxidação de lipídeos, na formação de radicais livres.

Além dos benefícios à saúde, é importante que um alimento seja saboroso. Um dos fatores importantes na aceitação de determinada fruta é a relação entre o teor de açúcares e sua acidez total titulável (ATT). Esta relação Brix:ATT é chamada “ratio”. De acordo com o tipo de fruto, o clima, os tratos agronômicos e o seu estádio de maturação, o “ratio” pode assumir valores elevados (acima de 200 para laranja lima, por exemplo), ou inferiores a 10. Frutos de umê “in natura”, apresentam “ratio” que varia de 2,5 a 5 e têm baixa perspectiva de aceitação no mercado nesta forma, pois raramente um produto apresenta grande aceitação com valores de “ratio” abaixo de 10, onde a sensação de acidez é muito alta (JAYASENA e CAMERON, 2008; SANTANA, 2009). Este “ratio” pode ser ajustado pela incorporação de açúcar ou pela diluição do ácido na elaboração de néctar de fruta.

Outro aspecto sensorial importante para a aceitação de um produto alimentício é o seu aroma (OLIVEIRA, 2009). O fruto verde de umê não apresenta odor nem aroma perceptível. No entanto, durante sua maturação ocorre a formação de compostos voláteis responsáveis por aromas similares ao de pêssego, de maior intensidade e mais floral e adocicado, que foram identificados e quantificados por Miyazawa et al. (2009).

Com o crescimento do setor de sucos, a formulação de sucos com sabor adequado e apelos saudáveis pode levar ao desenvolvimento de um produto de grande aceitação por parte do consumidor. Para o desenvolvimento de um suco, é necessário conhecer o comportamento reológico da polpa concentrada e do suco sob diversas temperaturas. Normalmente sucos de frutas apresentam comportamento reológico do tipo pseudoplástico, onde existe uma elevada viscosidade do líquido não submetido a tensões, que decresce com o aumento da tensão aplicada ao líquido (BEZERRA et al., 2008).

O conhecimento do comportamento reológico de uma polpa ou um suco é importante para garantir o controle do processo, a manutenção dos aspectos sensoriais e para os cálculos de dimensionamento do projeto industrial de uma linha de produção. A concentração do produto bem como a variação da temperatura pode afetar no seu escoamento, o que pode acarretar em mudanças nos parâmetros de transferência da quantidade de movimento, de calor e de massa (ALTAN e MASKAN, 2005; NINDO et al., 2005; CHIN et al., 2009; FALGUERA e IBARZ, 2010). A falta deste conhecimento pode resultar na perda de competitividade industrial devido ao sobre processamento térmico, que acarreta no aumento da energia utilizada no processamento industrial e perda da qualidade organoléptica e nutricional do produto, ou no processamento térmico inadequado que pode permitir o desenvolvimento de micro organismos patogênicos, acarretando em perigo ao consumidor final, especialmente em produtos prontos para o consumo.

Assim, com a produção de derivados a partir do umê e com o conhecimento de suas características físico-químicas em função de parâmetros de processo, é possível o desenvolvimento de produtos seguros e prontos para o consumo, aliando baixa necessidade do uso de defensivos agrícolas, sabor agradável e benefícios à saúde.

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# **Fruit characteristics and pollen morphological characterization of *Prunus mume* from different regions of Southeast Region of Brazil for human consumption**

**Artigo a ser encaminhado para a revista Bragantia. Assunto: crop production.**

## **Comments to the Editor**

The present work describes mume (*Prunus mume*) fruit characteristics from different locations Southeast Region of Brazil. Although many scientific research have been published world-wide about benefits related to consumption of mume products, in Brazil all researches have been focused its use as rootstock for *Prunus* species, such as peach and plums.

The objective of the present work is to determine the main fruit characteristics planted in Southeast Region of Brazil, such as average weight, size, total soluble solids and titratable acidity content, as well as texture and color during full maturation of fruits. The morphological pollen characterization helps to organize a data base of this culture in Brazil and the correct classification of the culture.

The information presented can lead to new lines in genetic improvement new crops, for fruits processing, due to its high agricultural resistance, great similarity to peach fruits and food products that present health benefits. The present work was not submitted to any other journal until the evaluation on the present periodic.

## **ABSTRACT**

The present work compared mume (*Prunus mume*) fruits from different locations in Southeast Region of Brazil. Fruit sizes and mass were smaller than values reported in Asiatic reports. Firmness of fruits collected at green-mature stage for consumption provided about  $14.1 \pm 2.6$  g to tear the peel and  $5.3 \pm 1.1$  g during inner pulp compression using a 2 mm diameter tip. In fully ripe fruits, the peel did not show mechanical resistance and it was noted about  $3.4 \pm 0.8$  g during compression. Since these fruits are climacteric, they can be collected at green-mature stage and left to mature. During maturation it is possible to notice a strong sweet floral odor formation and a significant change in color, specially “a” color parameter (L,a,b system), from  $-14.5 \pm 5.0$  (-a) to  $1.8 \pm 2.1$  (+a). Although total soluble solids did not vary significantly during fruit maturation, total titratable acidity lowered from 4.0-5.7 g (100g<sup>-1</sup>) to 2.0-3.8 g (100g<sup>-1</sup>) expressed in citric acid. Moreover, the morphological pollen characterization showed common characteristics between *Prunus mume* and other species from *Prunus* genus presented as average, isopolar, tricolorate, colporate, difficult to see with edges and triangular framework.

Key words: *Prunus mume* Sieb. et Zucc, fruit characteristics, pollen characterization

Título em português: **Características da fruta e caracterização morfológica do pólen de umê proveniente de diferentes localidades da Região Sudeste do Brasil, para o consumo humano**

## RESUMO

O presente estudo comparou frutos de umê provenientes de diferentes localidades da Região Sudeste do Brasil. As dimensões e a massa dos frutos foram menores que os valores reportados por artigos científicos realizados na Ásia. A firmeza dos frutos verdes, mas fisiologicamente maduros, forneceu força de ruptura da pele de  $14,1 \pm 2,6$  g e em seguida, compressão da polpa igual a  $5,3 \pm 1,1$  g, utilizando um probe cilíndrico de diâmetro 2 mm, velocidade de teste de 5 mm ( $s^{-1}$ ) e profundidade 3 mm. Em frutos completamente maduros, a pele não mostrou nenhuma resistência mecânica e a força de compressão média foi de  $3,4 \pm 0,8$  g. Os frutos de umê são climatéricos e podem ser colhidos no estádio de maturação verde. Durante a maturação dos frutos, foi possível notar a formação de fortes odores florais adocicados e uma significativa alteração da cor, especialmente no parâmetro “a” (sistema L,a,b), de  $-14,5 \pm 5,0$  (-a) para  $1,8 \pm 2,1$  (+a). Embora o teor de sólidos solúveis totais não tenha se alterado durante a maturação, a acidez total titulável diminuiu de 4,0 a 5,7 para 2,0 a 3,8 g ( $100g^{-1}$ ), expressa em ácido cítrico. Ainda, os grãos de pólen analisados se apresentaram como médios, isopolares, tricolporados, colporos com extremidades de difícil visualização e âmbito triangular, mostrando características comuns entre o *P. mume* e outras espécies do gênero *Prunus*.

Palavras chave: *Prunus mume* Siebold et Zucc., características do fruto, caracterização polínica.

## 1 INTRODUCTION

The *Rosaceae* family has about 300 genus and 3,000 species, one of the largest of *Angiosperms* family. *Rosaceae* members occur in a variety of habitats around the world, especially on the northern hemisphere, with a few species native from Brazil. Regarding the economic aspect, it is one of the most important families, including apple (*Malus sylvestris*), pear (*Pyrus communis*), peach (*Prunus persica*), plum (*Prunus salicina*), strawberry (*Fragaria vesca*), yellow plum (*Eriobotrya japonica*), cranberry (*Rubus idaeus*) and mume (*Prunus mume*). These plants are cultivated in the South and Southeast Regions of Brazil because of the milder weather conditions, favorable for the development of temperate fruits (LORENZI and SOUZA, 2005).

*Prunus mume* Siebold et Zucc. presents several common names, such as Chinese plum, Japanese apricot, ume (from Japanese), mei (Chinese), maesil (Korean) or mume. There are over 200 cultivars of mume, which have been widely cultivated in Asia for about 3,000 years (TSUBAKI, OZAKI and AZUMA, 2010). Fruiting mume is consumed mainly in Korea, China and Japan. The tree is cultivated in about 2,700 ha in Korea, 9,000 ha in China and 17,000 ha in Japan with an average production yield of 6.7 ton (ha<sup>-1</sup>) (JUN and CHUNG, 2008; TOPP, NOLLER and RUSSEL, 2007). Price of fruits in Japan is about 1.68 US dollar (kg<sup>-1</sup>).

The plant exhibits an arboreal growth and 4-6 meters high. Its leaves are simple, deciduous, hairless and paper-like consistency leaves, with color difference between top and bottom of the leaves and 3-7 cm long. The flowers are white, androgynous and bloom from June to August. Fruits are drupaceous and show firm pulp with bitter and sour taste and maturity ranging from October to December (LORENZI et al., 2006).

In Brazil most of the rootstocks of peach and plums are 'Okinawa' peach cultivar. *Prunus mume* Siebold et Zucc. has been studied as peach (*Prunus persica*) rootstock, showing promising characteristics, such as rusticity, high resistance to plagues and diseases, adaptation and for reducing the peach and nectarines trees sizes, reducing the size of the trees allows a higher density of planting (MAYER, PEREIRA and MORO, 2008;

MAYER, PEREIRA and SANTOS, 2005). High resistance to plagues and diseases, as well as rusticity enables fruit production without or with less use of pesticides.

Mume fruits display a typical climacteric pattern of respiration and ethylene production. Green fruits can be collected directly from the tree or shaken to fall in large clothes or nets. When there is interest in using ripe fruits, the fruits are left to drop by their own into nets while still green but physiologically mature. Then, they are matured at temperature above 20 °C ventilated (LUO, 2006). By doing these procedures, insects and flies presence during harvest period can be significantly reduced.

Mume cultivar, ripeness point of harvest, temperature and ambient of storage can influence in the quality or shelf-life of fresh fruit (KITA et al., 2007). Texture, color and aroma of mume fruits change significantly during maturation (MIYAZAWA et al., 2009).

Several processed products of mume fruits have been consumed as health food and for the treatment of several diseases because they are rich in bioactive compounds, such as anti-cancer and antioxidant substances (TSUBAKI, OZAKI and AZUMA, 2010; LIU et al., 2009; SHI et al., 2009; ADACHI et al., 2007; SHI and MOY, 2005). Because of the similarity of unripe apricot and mume fruits, JUN and CHUNG (2008) developed sequence-characterized amplified region (SCAR) markers to differentiate mume fruits due its higher value and demand. This identification can be used both for germplasm classification and to detect unripe apricot fruits commercialized as mume fruits. JO et al. (2006) used mume extract as a natural source of antioxidant compounds, able to inhibit formation of “warmed over flavor” volatiles in cooked chicken meat during storage because free radical formation during initiation stage of lipid oxidation is inhibited and thus the breakage of the triglyceride chain and formation of free carboxylic acid and other volatiles compounds.

In Asia there are different cultivars that include early and late fruit producing plants so the harvest interval increases to three or four months. Among the same plant, the interval for harvesting does not exceed three weeks.

The objective of this research was to characterize Brazil mume fruits for future use as ingredient for food production with high nutritional value and great acceptance by most

consumers. Fruits were characterized in terms of average size, weight, color, density, geometry, yield of pulp, solids content and total titration acidity. In addition, the morphological pollen characterization can provide better understanding of *Prunus mume* species, also helping to characterize the mume plants planted in Southeast Region of Brazil.

## 2 METHODS

### 2.1. Plant material

Fruits were green-to-yellow color soon after natural fruit dropping occurs, approximately 88 days after flowering (DAF) and were held for over 8 days at 26 °C to monitor the ripening of fruits.

Mume fruits were collected in São Paulo State, Brazil, from different locations:

- ▲ S – Angatuba, SP- Latitude 23°30'43"S; Longitude 48°16'38"W; Elevation 737 m;
- ▲ V – Valinhos, SP- Lat. 22°59'28"S; Long. 47°02'38"W; Elev. 640 m;
- ▲ CB – Capão Bonito, SP- Lat. 24°02'52"S; Long. 48°21'19"W; Elev. 705 m;
- ▲ B – Botucatu, SP- Lat. 22°57'32"S; Long. 48°27'09"W; Elev. 841 m;
- ▲ SUN – Botucatu, SP- Lat. 22°57'56"S; Long. 48°27'25"W; Elev. 843 m.

All locations show climate are characterized as Cwa by Koeppen classification (tropical of altitude) with rain in summer and lack of rain during the winter. Average temperature in the hottest month is over 22 °C.

Preliminary tests were performed to follow the maturation of fruits. Green-stage fruits were collected from 60 DAF every 7 days and these fruits were stored at 26 °C ventilated and had the color evaluated every day. Fruits collected before 81 DAF did not ripe well. This could be noticed by the change of color from green to brown with no characteristic odor formation. Only fruits collected 88 DAF presented yellow color and

mature characteristic odor formation. After the 7<sup>th</sup> day at 26 °C these fruits presented change of color to brown and wrinkle of the peel.

## 2.2. Physical characterization

Physical analysis were performed in 40 different green-mature fruits using caliper Marberg with accuracy of 0.05 mm and scale Mettler Toledo, AB204 with accuracy of 0.1 mg. The fruits were portioned into three different fractions (pulp, peel and stone) and weight separately.

Full-mature fruits were manually portioned into three different fractions: peel, pulp and stone, and weight separately. The relation between the mass of (pulp+peel):stone gives the maximum yield that can be obtained during fruit pulping.

The specific weight was calculated based on the average dimensions and mass of fruits and considering the fruit as a sphere with diameter equal to the average of the diameter and the height of these fruits. The fruit volume was calculated using Equation 1.

$$V = \frac{\pi}{6} \times D^3 \quad (1)$$

The bulk density was calculated using estimated particle porosity obtained in page 630 of FOUST et al. (1982). Mume fruits were considered as non-uniform spheres and the relation diameter of fruits:diameter of bed equal to 0.1. Thus, porosity resulted 0.34 and bulk density calculation was provided by Equation 2.

$$\text{Bulk density} = (\text{Specific weight}) \times (1 - \text{Porosity}) \quad (2)$$

The force necessary for perforation of the skin of fresh fruits in different maturation stages was determined using a TA-XT2i texture analyzer (Stable Micro Systems) incorporating a P2N needle probe with parameters, pre-test speed 5 mm s<sup>-1</sup>, test speed 0.5 mm s<sup>-1</sup>, post-test speed 5 mm s<sup>-1</sup>, depth 3 mm. Firmness was measured on opposite

sides along the equatorial region of the fruit. The first measurements were done on the incident sun side and were analyzed 40 fruits for each stage of maturation.

Color of the skin was measured in 20 different fruits, taking picture on opposite sides using digital camera Sony DSC-H2, fluorescent white lamp and no flash. Five distinct points of each picture were exported computer and transformed to Lab color system using Corel-PhotoPaint, version 12 (Corel®).

### 2.3. Chemical characterization

For chemical characterization of mume fruits, each sample was obtained from manual pulping of 8 fruits, using knife and latex gloves. Soluble solids content were analyzed in duplicate for each sample and were used 4 different samples for each treatment or location. Results were obtained using a digital refractometer Reichert, AR200, expressed in Brix. Total titration acidity was expressed in terms of citric acid, analyzed in duplicate for each sample and used 4 different samples for each treatment or location (INSTITUTO ADOLFO LUTZ, 2008).

Solids content were analyzed with four repetitions of 10 g of fruits from each location and dried in circulated heater at 105 °C for 24 hours (INSTITUTO ADOLFO LUTZ, 2008).

### 2.4. Morphological pollen characterization

The material used for pollen characterization was collected from S location: Quast, September 10, 2010 (HUPG17130), Angatuba, SP.

The polinic material was prepared as described by ERDTMAN (1952), with modifications proposed by MELHEM et al. (2003) for observation in optical microscope.

From the material, were taken randomly 25 measurements from the polar diameter (PD) and equatorial diameter (ED) of pollen grains from equatorial view and 10 measurements of the equatorial diameter from polar view (EDPV) and apocolpus side (AS)

from polar view, distributed in 10 glass slides. Statistical analysis was performed by calculating the arithmetic mean, standard deviation of the sample, deviation from the mean, coefficient of variation, confidence interval at 95% and range of variation.

For other characteristics such as openings, layers of exine and the diameter of the material, were measured 10 randomly pollen grains distributed in 10 glass slides and calculated the arithmetic mean using Bioestat 5.0 software.

## 2.5. Statistical Analysis

All the experiments were conducted in triplicate and the mean and standard deviation were calculated using MS Excel software (Microsoft®). Data for LSD at  $p < 0.05$  for significant difference in treatment means were analyzed using Statistica software (version 5.5, StatSoft Inc., Tulsa, OK, USA).

# 3 RESULTS AND DISCUSSION

## 3.1. Physical characteristics

Fruits from locations S, V and CB were not different from each other and showed characteristics similar to Koushiushinkou cultivars from Japan, with very small fruits (ZHI-HONG et al., 2004). Fruits were small compared to other locations, which presented fruits with 15-30 g, as reported by JUN and CHUNG (2008). Regarding all fruits and plants of different locations used in this present work, were not applied pesticides.

Fruits dimensions and weight are shown in Table 1. The values shown are the mean and standard deviation of 40 measurements for each location. Different letters in the same column mean the values are significantly different ( $p < 0.05$ ).

**Table 1** – Dimensions and mass of green-mature mume fruits

Location	Diameter (cm)	Height (cm)	Mass (g)
S	2.130±0.174 <sup>c</sup>	2.304±0.199 <sup>c</sup>	5.701±1.298 <sup>c</sup>
V	2.242±0.247 <sup>c</sup>	2.447±0.281 <sup>c</sup>	6.111±1.934 <sup>c</sup>
CB	2.239±0.125 <sup>c</sup>	2.310±0.125 <sup>c</sup>	6.892±1.058 <sup>c</sup>
B	2.502±0.215 <sup>b</sup>	2.662±0.206 <sup>b</sup>	9.095±2.099 <sup>b</sup>
SUN	3.153±0.264 <sup>a</sup>	3.164±0.215 <sup>a</sup>	16.909±3.667 <sup>a</sup>

\*Different letters in the same column mean the values are significantly different ( $p<0.05$ ).

\*\*Least significance difference (LSD) was 0.063, 0.063 and 0.643 for diameter, height and mass, respectively.

Fruits from location B showed intermediate sizes and mass. As reported by farmers, cultivar plants from B and SUN location have the same origin, from China for over 20 years. Both locations are very close to each other and differences are mostly in agronomic handling. Probably in commercial planting were used fertilizers and small fruits were collected at early stage of development to enable remaining fruits to become larger.

Location SUN is the only commercial fruit producer and the mass is at the lower limit of values presented by JUN and CHUNG (2008) probably due to genetic improvement in Asian countries, where mume fruits are consumed in larger scale and are part of traditional food. The trees in SUN are over 20 years old and no genetic improvement work was carried out during this period. In occidental countries, these fruits are not consumed by most people due to lack of knowledge and because they are normally planted by oriental descendants, although other *Prunus* species, such as peach (*P. persica*), nectarine (*P. persica* var. *nucipersica*), apricot (*P. armeniaca*), european plums (*P. domestica*) and japanese plums (*P. salicina*) are greatly appreciated, cultivated and genetically improved and adapted to each region (BARBOSA et al., 1997). In Brazil, most

researches over *P. mume* intend to use the plant as rootstock for peach, nectarine and Japanese plums because of great resistance to diseases (MAYER et al., 2005).

Pulp corresponded to approximately 71.3%  $\pm 2.7$  of total weight of fresh fully ripe fruit. The skin and stone represented 13.8%  $\pm 1.7$  and 14.9%  $\pm 2.0$  respectively. The relation between mass of (pulp+peel):stone resulted 5.71 $\pm$ 1.08, lower than the same relation found by Lima et al. (1999) for peaches (between 10 and 20). This means the mume stones in the studied fruits are relatively larger than the ones in peaches. Although the relation (pulp+peel):stone is important for large scale production design, little information was found regarding to mume fruits. This relation can increase using bigger fruits that can be obtained using cultivars that produce larger fruits or collecting small fruits right after flowering so the energy of the plant is used for less fruits, providing better quality and larger fruits.

The specific weight of green-mature mume fruits ranged from 0.906 to 1.119 g ( $\text{mL}^{-1}$ ). It was not observed any relation between the specific weight and stage of maturation of fruits. The bulk density ranged from 0.6 to 0.7 g ( $\text{mL}^{-1}$ ). The knowledge of the bulk density is important for the design of storage and transportation bins.

Mume fruits presented significant different texture values during maturation. The equivalent force (in mass) necessary to tear the peel of green-stage firm fruits (89 days after flowering - DAF, or 1 day after harvest - DAH) was 14.147 $\pm$ 2.590 g and occurred after a cylindrical tip with 2 mm diameter penetrated 1.3-1.7 mm. After tearing, the peel average compression force of these fruits was obtained over a period of constant force when the probe penetration was between 1.9 and 2.8 mm and was equivalent to 5.251 $\pm$ 1.080 g. This shows that green-stage fruits have high resistance to compression.

Ripe fruits (6 DAH) showed significant differences in color, odor and texture. After this period, fruits showed senescence characteristics and were not used. Differently from green mume fruits, the peel of fully mature-stage fruits did not present significant mechanical resistance. Most fruits presented nearly linear increase of compression strength, although a few showed maximum force around 2-2.5 mm of penetration. The average texture of mature mume fruits was 3.433 $\pm$ 0.803 g. Fruit softening during ripening is

associated with modifications of cell wall polysaccharides, depolymerization of hemicellulose, increasing pectin solubility and loss of neutral sugars. At this stage of maturation, fruits are easily smashed and should be processed soon or stored under refrigeration.

Color showed different values during fruit maturation, with (-a) indicating green, (+a) red, (+b) yellow and "L" parameter varying from 0 (dark) to 100 (white). At green-stage, the color index was  $-14.53\pm4.98$  (-a),  $31.13\pm5.81$  (+b) and  $43.30\pm12.17$  (L), whereas ripe fruits was  $1.77\pm2.10$  (+a),  $35.43\pm2.78$  (+b) and  $37.73\pm5.04$  (L). Results of color of mume fruits during maturation were similar to reported for 'Nanko' cultivars (KITA et al., 2007), that showed change of color from green to yellow and high ethylene biosynthesis 3 DAH.

The green-mature fruits studied reached full ripeness after 5 to 6 days at 26 °C, which shows a fast change in color and texture after harvesting. Previous studies showed that mume fruits collected before this maturation stage did not reach complete maturity. Although most fruits need to be collected from the plant, mume fruits fall when physiological maturity is fully achieved, still green-mature. An alternative for high quality fruits is to keep neatness under the tree and collect the fruits every day during harvest time, as common practice for macadamia nuts (FRANÇA, 2007).

### 3.2. Chemical characteristics

Total soluble solids (TSS) did not vary significantly during maturation of fruits and ranged from 9.5 to 10.0 °Brix. Total solids content ranged from 10.2 to 12.2%. The pH value did not change much and showed values between 2.55 and 2.65 due to a high acid content. Total titratable acid (TTA) values lowered from 4.0-5.7 g (100g<sup>-1</sup>) green-mature fruit to 2.0-3.8 g (100g<sup>-1</sup>) ripe fruit, expressed in citric acid. TSS and TTA are important parameters of fruit maturity and quality. Climacteric fruits, such as mume are capable to further metabolize and increase respiration rates after harvesting. During the last days of maturation, some acids present in the fruit are broken to form volatile compounds, although

even in ripe fruits TTA is high, comparable to those found in lemons and limes (PENNISTON et al., 2008).

Ratio value under 10 makes product unacceptable for fresh fruit consumption for most people. This value is much under values found for fruit intended for direct consumption, such as grapes and peaches, with ratio values between 16 and 20 (JAYASENA and CAMERON, 2008; SANTANA, 2009). Green stage mume fruits showed ratio ranging from 1.5 to 2.5 and strong bitter taste. Although the bitterness practically vanished in fully ripe mume fruits, ratio varied from 2.5 to 5.0. Thus, the consumption of mume fruits is recommended as processed food products, with health proven health benefits due to the antioxidant capacity of fruits (QUAST et al., 2011).

### 3.3. Morphological pollen characterization

The observation of samples prepared in glass slides was done according to BARTH and MELHEM (1988).

*P. mume* pollen grains showed average size and are shown in Figures 1 to 3. Still, pollen grains are isopolar with radial simetry, triangle extent, prolate spheroidal shape (Table 2). They presented long and narrow colporate (Figures 4 and 5) with difficulty to visualize edges and median slight constriction. Endoaperture is elongated without median constriction (Table 3). Sexine has no cave and has striated surface reticulate (Figures 5 and 6) which makes it slightly wavy. The thickness of the sexine is twice the nexin.

Comparing mume with other species of *Prunus* genus, pollen grains analyzed showed common characteristics, with the scope within the genus classified as obtuse, convex and triangular. The measurements from the polar and equatorial diameter among the genre vary according to different species. Thus, measurements of *P. mume* are within the range of measurements described in scientific literature.

**Table 2** – Measurements ( $\mu\text{m}$ ) from equatorial view of pollen grains from *Prunus mume* species, Rosaceae, Angatuba (location S), SP

Species	Polar Diameter					Equatorial Diameter					P/E
	x	$s_x$	s	CV%	IC95%	x	$s_x$	s	CV%	IC95%	
<i>Prunus mume</i>	36.4	0.6	3.2	8.8	35.1-37.7	34.8	0.4	1.8	5.1	34.1-35.5	1.04

\*n=25, where: x- arithmetic mean;  $S_x$ - standard deviation of mean; s- standard deviation of sample; CV- coefficient of variation; IC- confidence interval at 95%; P/E- relation between polar and equatorial diameter.

**Table 3** – Mean measurements ( $\mu\text{m}$ ) of apertures and thickness of the exine of pollen grains of *Prunus mume* species, Angatuba (location S), São Paulo (n=10)

Species	Colpo		Endoaperture		Thickness of exine		
	Width	Length	Width	Length	Sexine	Top	Nexine
<i>P. mume</i>	2.72	27.35	2.15	10.97	1.53	0.81	0.63



**Figure 1** – Polar view



**Figure 2** – Equatorial view



**Figure 3** – Colpo detail



Figure 4 – Detail of LO1 (high foccus)



Figure 5 – Detail of LO2 (low focus)

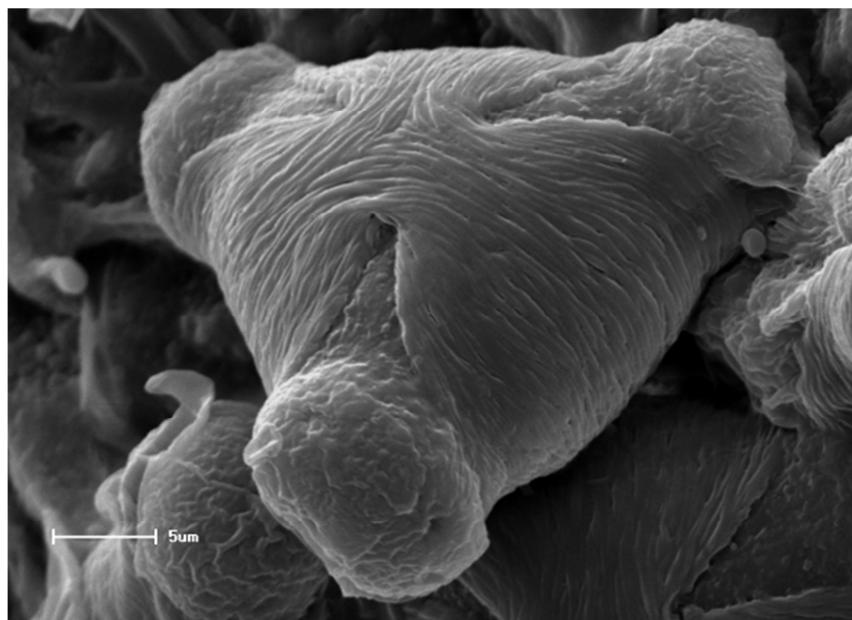


Figure 6 *Prunus mume* pollen in scanning microscopy

#### 4 CONCLUSIONS

The present work showed that green-mature mume fruits planted in Brazil showed diameter ranging from 2.1-3.1 cm, mass 5.7-16.9 g and bulk density 0.6-0.7 g (mL<sup>-1</sup>). In fully ripe fruits pulp and peel represented 71.3% and 13.8% respectively. The peel of green-stage fruits showed high mechanical resistance and was necessary about 14.1 g to tear the peel with a 2 mm diameter cylindrical probe. In fully ripe fruits the peel did not

represent significant mechanical resistance. Color changed significantly during the six days of fruits maturation specially parameter “a” of color in Lab system, that increased from -14.53 (-a) to 1.77 (+a). Although total soluble solids content did not change significantly during maturation, TTA lowered from 4.0-5.7 to 2.0-3.8 g (100g<sup>-1</sup>) expressed in citric acid and ratio increased until 5, which is low for fresh fruit consumption. Comparing the fruit characteristics presented in this work with Asian mume cultivars, it is necessary to plant new *P. mume* cultivars or study the influence of soil, fertilizers and climate for commercial production with higher yield to produce food products with health benefits and low need of pesticides.

The pollen grains analyzed are presented as average isopolar, tricolorate, colporate, difficult to see with edges and triangular framework. The species analyzed presented similar characteristics to other species of the genus *Prunus* described in the literature. In addition to morphological pollen characterization, the present work contributes to the study of taxonomy of the species through the pollen.

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# ***Prunus mume* – fruit characteristics and phenolic content capacity**

**Artigo publicado na revista Fruit Processing, p.238-242, nov/dec 2011.**

*Prunus mume*; fruit characterization; phenolic content

## **FRUIT PRODUCTION AND CHARACTERISTICS**

*Prunus mume* Sieb. et Zucc (with fruit name in Chinese *Wu Mei*) belongs to *Rosaceae* family and has been widely cultivated in Asia for about 3,000 years. There are about 200 cultivars of mume. Initially the plants were cultivated because of their flowers, but about 2,000 years the interest has been focused in the fruit medicinal properties (Shi & Moy, 2005; Shi et al., 2009; Tsubaki et al., 2010).

Fruiting mume is consumed mainly in Korea, China and Japan. The tree is cultivated in about 2,700 ha in Korea, 9,000 ha in China and 17,000 ha in Japan with an average production yield of 6.7 ton/ha (Jun & Chung, 2008; Topp et al., 2007). Price of fruits in Japan is about 1.68 US dollar/kg.

In Brazil, most of the rootstocks of peach and plums are 'Okinawa' peach cultivar. *Prunus mume* Siebold et Zucc. has been studied as peach (*P. persica*) rootstock, showing promising characteristics, such as rusticity, high resistance to plagues and diseases, adaptation and for reducing the peach and nectarines trees scales (Mayer et al., 2005; Mayer et al., 2008). Reducing the size of the trees allows a higher density of planting. High resistance to plagues and diseases, as well as high rusticity enables fruit production without or with less use of pesticides.

Fruits are commonly collected in the middle spring before ripening to produce extremely sour products, rich in glucoside prudomenin, malic acid and succinic acid (Shi & Moy, 2005) that are conserved by lowering of water activity with salt impregnation and dehydration. In Asia there are different cultivars that include early and late fruit producing plants, so the harvest interval increases to three or four months. Among the same plant, the interval for harvesting does not exceed three weeks. Green fruits can be collected directly

from the tree or shaken to fall in large clothes or nets. When there is interest in using ripe fruits, the fruits are let to fall by their own into nets while still green, but physiologically mature, and matured above 20 °C with ventilation.



**Figure 1** – Flowers of fruiting mume (© E. Quast)



**Figure 2** – Mume fruit tree in Brazil (© E. Quast)

Mume fruits are small (15 to 30 g) with a green to yellow skin color and shows high acidity and has a high citric acid content. Apricot (*P. armeniaca*) has bigger fruits (30 to 80 g) and yellow to orange skin when fully ripe with low to medium acidity and high percentage of malic acid. They are closely-related species that have high cross-compatibility and many natural hybrids. Because of the similarity of unripe apricot and mume fruits, Jun & Chung (2008) developed sequence-characterized amplified region (SCAR) markers to differentiate mume fruits due its higher value and demand. This identification can be used both for germplasm classification and to detect apricot unripe fruits commercialized as mume fruits.



**Figure 3** – Hole and cut mume fruit collected in São Paulo, Brazil (© E. Quast)

The stones of mume fruits cling the pulp, similar as observed in many peach and plum cultivars. Stones are relatively small, and have a slightly smooth surface.

Ripeness point of harvest, temperature and ambient of storage can influence the quality or shelf-life of fresh fruit, as well as the mume cultivar. Kita et al. (2007) observed significant post-harvest differences between ‘Orihime’ and ‘Nanko’ cultivars. Natural fruit dropping was observed approximately 105 and 120 days after flowering (DAF), respectively. At harvest stage, the skin color of ‘Orihime’ presented more yellow-to-red color, probably because of the early ethylene production, 92 to 95 DAF. In ‘Nanko’ cultivars high ethylene production was observed just 3 days after harvest (DAH).

### **Mume medicinal properties**

Several mume processed products have been consumed as health food and for the treatment of several diseases because they are rich in bioactive compounds, such as anti-cancer and antioxidants substances (Shi & Moy, 2005; Shi et al., 2009; Tsubaki et al., 2010).

The effect of a mume juice concentrate (MJC) in rats was examined by Kubo et al. (2005), which showed an improving effect on blood fluidity. Citric acid and mumefral were identified as the main principles on the inhibitory effects of the collagen-arachidonic acid and ADP-induced platelet aggregations and on the thrombin-induced conversion of fibrinogen to fibrin.

The protective influence of processed foods made from *Prunus mume* Sieb. et Zucc. in gastric disorders, including chronic active gastritis, peptic ulcers, metaplasia and cancer are traditionally known. Otsuka et al. (2005) showed that MJC in doses of 1% for 10 weeks suppressed chronic active gastritis in the glandular stomachs of *Helicobacter pylori* infected Mongolian gerbils.

Mori et al. (2007) showed that the dried hydrophobic extract, MK615, obtained by heating and concentration of mume extract has an anti-neoplastic effect against three human colon cancer cells, SW480, COLO, and WiDr. The effect may be exerted by induction of apoptosis and autophagy.

Still about cancer, Jeong et al. (2006) isolated a new compound from mume fruit that showed significant inhibitory effects against cancer cells but little against normal cells. On the basis of HPLC analysis, the amount of the novel compound B-1 in *P. mume* fruit is

approximately 1.47-1.70 g/kg. It can be used as a nutraceutical compound for alcohol beverages and fruit juices of *P. mume* fruit, which are currently sold in the market.

In addition to the health benefits presented, Yingsakmongkon et al. (2008) reported the ability of mume fruit juice concentrate to inhibit human influenza A virus infection. Treatment of the cells with the fruit-juice concentrate was very effective only before viral adsorption; hence, the product probably acts via blockage of viral hemagglutinin attachment to host cell surfaces, with a lectin-like molecule(s) that could bind competitively with influenza viral hemagglutinin for host cell surface N-linked glycoprotein necessary for influenza virus entry and infection.

Oxidative damage of biological molecules in human body is involved in degenerative or pathological process such as cancer.

Therefore, the potency of these compounds could provide a chemical basis for some of the health benefits such as anti-mutagenic, anti-carcinogenic and antioxidant activities.

The reactive oxygen scavenging capacities of food products are related to medicinal effects in animals' body. However, food properties or nutritional contents may change during processing.

## **FRUIT COLLECTED**

Mume fruits were collected in São Paulo State, Brazil, Latitude: 23°30'43"S; Longitude: 48°16'38"W; Elevation: 737m. Fruits were green-to-yellow color soon after natural fruit dropping occurs, approximately 88 DAF and were held for 6 days at 26 °C until complete maturation. Mume were collected from the same farmer and were carried out physical and chemical characterization of the fruits.

## **PHYSICAL AND CHEMICAL CHARACTERISTICS**

Fruits weight were  $6.11 \pm 1.93\text{g}$ , significantly smaller than those reported by Jun & Chung (2008) with 5 to 30 grams in mass, probably due to genetic improvement in Asian countries, where mume fruits are consumed in larger scale and are part of traditional food. In occidental countries, mume fruits are not consumed by most people because of lack of knowledge and are normally planted by oriental descendants with no use of pesticide because of the high resistance of the crop, although other *Prunus* species, such as peach (*P. persica*), nectarine (*P. persica var. nucipersica*), apricot (*P. armeniaca*), European plums (*P. domestica*) and Japanese plums (*P. salicina*) are greatly appreciated, cultivated and genetically improved and adapted to each region (Barbosa et al., 1997). In Brazil, most researches over *P. mume* intend to use the plant as rootstock for peach, nectarine and Japanese plums because of great resistance to diseases (Mayer et al., 2005). The plants were not fertilized nor the excess of fruits removed. This probably led to small fruits with small relation pulp/stone. There is also a need to know the cultivar of *P. mume* adapted to Brazil, with low demand of cold.

Total soluble solids (TSS) and total titratable acid (TTA) are important parameters of fruit maturity and quality. Climacteric fruits, such as mume are capable to further metabolize and increase respiration rates after harvesting. During the last days of maturation, some acids present in the fruit are broken to form volatile compounds. Mume fruits presented TTS ( $^{\circ}\text{Brix}$ ) of  $9.125 \pm 0.177$  and TTA (g citric acid/ 100 g product) of  $5.205 \pm 0.098$ . Ratio value of 1.75 makes the fresh fruit unacceptable for consumption for most people. This value is much under values found for fruit intended for direct consumption, such as grapes and peaches, with ratio values between 16 and 20 (Jayasena & Cameron, 2008; Santana, 2009).

## TOTAL PHENOL CONTENT (TPC)

In order to facilitate understanding of analysis of TPC has assembled a Table that summarizes the different treatments to which mume fruits were submitted.

**Table 1** – Identification of different treatments for TPC

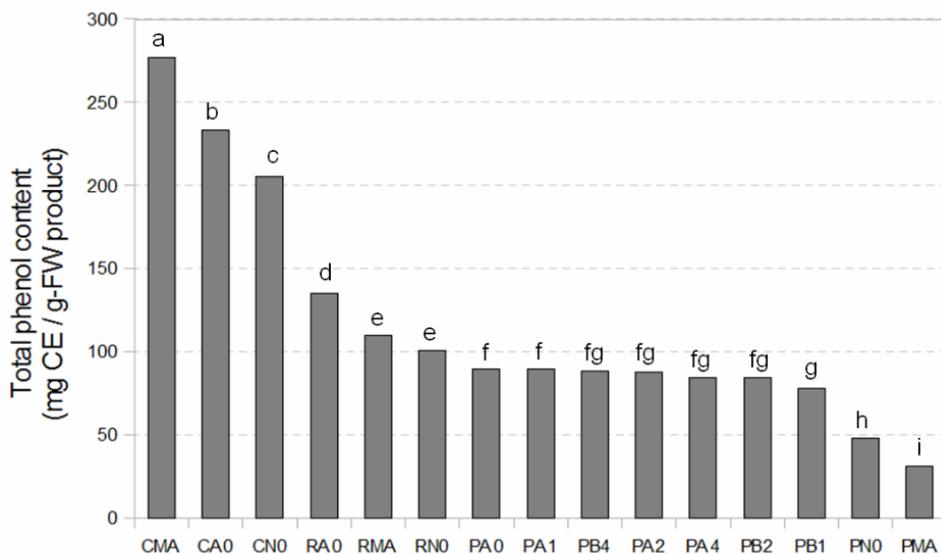
Code	Part of the fruit and treatment before and after pulping
CMA	Peel, manual separation, without air incorporation, after 1 hour at 26 °C
CA0	Peel, with thermal blanching, soon after pulping
CN0	Peel, without thermal blanching, soon after pulping
RA0	Stone, with thermal blanching, soon after pulping
RMA	Stone, manual separation, without air incorporation, after 1 hour at 26 °C
RN0	Stone, without thermal blanching, soon after pulping
PN0	Pulp, without thermal blanching, soon after pulping
PA0	Pulp, with thermal blanching, soon after pulping
PA1	Pulp, with thermal blanching, without vacuum, after 1 hour at 100 °C
PA2	Pulp, with thermal blanching, without vacuum, after 2 hours at 100 °C
PA4	Pulp, with thermal blanching, without vacuum, after 4 hours at 100 °C
PB1	Pulp, with thermal blanching, with vacuum, after 1 hour at 100 °C
PB2	Pulp, with thermal blanching, with vacuum, after 2 hours at 100 °C
PB4	Pulp, with thermal blanching, with vacuum, after 4 hours at 100 °C
PMA	Pulp, manual separation, without air incorporation, after 1 hour at 26 °C

It can be seen that the peel (C) has higher TPC values (over 20 mg catechin equivalent per gram of fresh fruit), followed by the stone (R). These results are similar to results of phenolic compounds in peach (*Prunus persica*) cultivars reported by Andreotti et al. (2008) that detected higher TPC in peel tissues compared to pulp.

CMA and CA0 presented 35% and 14% higher values compared with CN0, respectively. Manual separation of the peel (CMA) resulted in higher TPC probably because the tissue was not injured as much as during mechanical pulping. It is important to note that the CMA samples were left one hour exposed to air at 1 atm and 26 °C before

immersion in ethanolic solution. Comparing TPC values on mume fruit tissues right after mechanical pulping (CA0 and CN0) it can be seen that blanching of the fruits helped to preserve phenolic compounds in mume peel.

TPC of mume stones presented mean values between 10 and 13.5 mg CE/ g-FW fruit. Samples of stones in blanched mume fruits mechanically pulped presented higher TPC probably because of intense contact with mume peel at high temperature and relative humidity at the disposal of the pulper. As previously described, the peel presented higher TPC compared to stones and pulp.



**Figure 4 – TPC of different treatments and parts of mume fruits.**

\*Difference significance was verified to 95% confidence level, identified with letters above the bars.

Values are given in mg catechin equivalent (CE) per gram of fresh weigh (FW) product.

Different letters mean the values are statistically different.

In mume fruit pulp TPC did not vary significantly, except for non-blanchered fruits (PN0 and PMA). It can be noticed that the pulp of non-blanchered mume fruits manually removed (PMA) presented the lowest TPC, as opposed to peel (CMA). This can be explained because of the fast action of enzymes located in pulp. TPC in PMA samples were immersed one hour after exposure in air ambient at room temperature. During this time, it can be noticed that TPC lowered about 35% compared to non-blanchered mume fruit pulp right after mechanical pulping (PN0).

Further, blanched pulp did not show significant difference in TPC regarding to time submitted at high temperature (until 4 hours at 100 °C), even without the use of vacuum. These results are consistent with the heat resistance of phenolic compounds reported by Lin et al. (2007), were the application of sterilization temperatures did not lower TPC.

Mume pulp presented higher TPC values (7.8 to 9 mg CE/ g FW) compared to most commercial frozen fruit pulps that ranged from 0.05 to 7.88 mg CE/ g product, were the highest TPC content was found in acerola pulp (Melo et al., 2008).

## CONCLUSIONS

The fruits studied were smaller than reported in literature, probably due to genetic difference and agricultural practices. This impacted in a lower relation pulp/stone and consequently lower yield during pulping. Depending on processing parameters the yield can change and part of the mume peel can be incorporated to the pulp. It was observed that the peel presents a higher TPC. Further researches are needed to evaluate the influence of peel incorporation over the product characteristics and acceptance.

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# **Chemical characteristics and phenolic content of mume fruits collected at different locations and at different maturity stages in Southeast Region of Brazil**

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## **ABSTRACT**

*Prunus mume* is widely studied due to its health benefits regarding to cardiovascular and cancer aspects. However, in Brazil this culture is found only among oriental descendants. The present study shows difference between mume fruits collected in three different locations in Southeast Region of Brazil. SUN location is the only commercial mume fruit producer and had average fruits of 16.9 g and in other locations weight varied from 5.7-6.9 g. Pectin content decreased from 11.2 to 10.8% during fruit maturation. Total phenolic compounds content varied from 147-226 mg catequin ( $\text{g}^{-1}$ ) dry basis and did not vary during ripening, as well as antioxidant power, that ranged from 96-169  $\mu\text{Mol Trolox (g}^{-1}\text{)}$  dry base or 21-34  $\mu\text{Mol Trolox (g}^{-1}\text{)}$  wet basis. Furthermore it was established a direct relation between TPC and antioxidant power for mume fruits from CB and SUN locations. FRAP analysis in hot filled pasteurized pulp stored at room temperature for one year showed that at least 50% of the original antioxidant power is left in the product, showing promising possibilities for mume product conservation with health appeal.

Key words: fruit characterization, health product.

## 1 INTRODUCTION

The *Rosaceae* family is one of the largest *Angiosperm* family. Regarding the economic aspect, it is one of the most important families, including apple (*Malus sylvestris*), pear (*Pyrus communis*), peach (*Prunus persica*), plum (*Prunus salicina*), strawberry (*Fragaria vesca*), yellow plum (*Eriobotrya japonica*), cranberry (*Rubus idaeus*) and mume (*Prunus mume*). These plants are cultivated in the South and Southeast Regions of Brasil because of the milder weather conditions, favorable for the development of temperate fruits (LORENZI and SOUZA, 2005). The plant exhibits an arboreal growth and 4-6 meters high. The flowers are white, androgynous and bloom from June to August. Fruits are drupaceous and show firm pulp with bitter and sour taste and maturity ranging from October to December (LORENZI et al., 2006).

In Asia there are different cultivars that include early and late fruit producing plants, so the harvest interval increases to three or four months. Among the same plant, the interval for harvesting does not exceed three weeks.

Mume fruits display a typical climacteric pattern of respiration and ethylene biosynthesis. Green fruits can be collected directly from the tree or shaken to fall in large clothes, nets or trimmed grass. When there is interest in using ripe fruits, the fruits are dropped by their own while still green but physiologically mature. Then they are matured at temperature above 20 °C ventilated (LUO, 2006). By doing these procedures, and removing over-ripe and injured fruits, insects and flies presence during harvest period can be significantly reduced. It is important to protect fruits during maturation with the use of protective screens.

The point of harvest, temperature and ambient of storage can influence the quality or shelf-life of fresh fruit, as well as the mume cultivar. Normally mume fruits are refrigerated to 0-10 °C when intended to extend the shelf-life of fresh fruit, but no studies were found about using modified atmosphere with this objective (KITA et al., 2007). Texture, color and perfume of these fruits change significantly during maturation (MIYAZAWA et al., 2009).

Even though in Brazil there are no studies to improve productivity and quality of fruits, over the world there are over 200 cultivars of *Prunus mume* Siebold et Zucc., which fruits are consumed mainly in Korea, China and Japan (TSUBAKI, OZAKI and AZUMA, 2010). In Japan fruits price is about 1.68 US dollar ( $\text{kg}^{-1}$ ), and present an average production yield of 6.7 ton ( $\text{ha}^{-1}$ ) (JUN and CHUNG, 2008; TOPP, NOLLER and RUSSEL, 2007).

*P. mume* has been studied in Brazil as peach (*Prunus persica*) and plums (*Prunus salicina*) rootstock, showing promising characteristics, such as rusticity, high resistance to plagues and diseases (MAYER; PEREIRA and MORO, 2008). High resistance to plagues and diseases, as well as rusticity enables fruit production without or with less use of pesticides.

Several mume processed products have been consumed as healthy food and for the treatment of several diseases because they are rich in bioactive compounds, such as anti-cancer and antioxidants substances (TSUBAKI et al., 2010; LIU et al., 2009; SHI et al., 2009; ADACHI et al., 2007; SHI and MOY, 2005). Because of the similarity of unripe apricot and mume fruits, JUN and CHUNG (2008) developed sequence-characterized amplified region (SCAR) markers to differentiate mume fruits due its higher value and demand. This identification can be used both for germplasm classification and to detect apricot unripe fruits commercialized as mume fruits. JO et al. (2006) used mume fruit extract as a natural source of antioxidant compounds, that can inhibit lipid oxidation and formation of “warmed over flavor” volatiles during storage of cooked chicken meat. Mume extract inhibits free radical formation during initiation stages of oxidation. In high concentrations, these highly reactive free radicals break triglycerides chains to produce free carboxylic acid that provide product alteration.

The objective of this research was to characterize Brazilian mume fruits for future use as ingredient for food production with high nutritional value and acceptance by most consumers. To accomplish this, fruits were characterized chemically and the phenolic content and the antioxidant activity were studied in fruits from different regions during ripening after harvest.

## **2 MATERIAL AND METHODS**

### **2.1. Plant material**

The fruits used were green-to-yellow color soon after natural fruit dropping occurred, approximately 88 DAF (days after flowering) and were held for up to 8 days at 26 °C ventilated until complete ripening of fruits.

Mume fruits were collected in São Paulo State, Brazil, from different locations:

- S – Angatuba, SP- Latitude 23°30'43"S; Longitude 48°16'38"W; Elevation 737 m;
- CB – Capão Bonito, SP- Lat. 24°02'52"S; Long. 48°21'19"W; Elev. 705 m;
- SUN – Botucatu, SP- Lat. 22°57'56"S; Long. 48°27'25"W; Elev. 843 m.

Full-mature fruits were used for total soluble solids (TSS) and total titration acidity (TTA) content analysis. Analyses were performed in duplicate for each sample and were used 4 different samples for each treatment or location. For each sample were used 8 fruits, manually pulped. TSS results were obtained using a digital refractometer Reichert, AR200, expressed in Brix. TTA was expressed in terms of citric acid, analyzed in duplicate for each sample and used 4 different samples for each treatment or location. Total solids content were analyzed with four repetitions of 10 g of fruits from each location and dried in circulated heater at 105 °C for 24 hours (INSTITUTO ADOLFO LUTZ, 2008).

Pectin content was analyzed mixing 4 g of lyophilized mume with 1:50 nitric acid 50 mM at 80 °C for 25 minutes. After filtration and cooling to 4 °C, the acid extract was mixed with 1:2 ethanol 96 °GL at 4 °C and allowed to stand still for 30 minutes. After this period, was filtered and kept inside permeable bags overnight with ethanol 70% under agitation. Then, washed again with ethanol 95%, and dried at 40 °C. Pectin analysis was performed in triplicate.

Antioxidant power of mume fruits was analyzed in hot filled pasteurized pulp kept for one year at room temperature. This pulp was obtained from fruits thermally blanched at

100 °C for 120 seconds, followed by mechanical pulping in a brush pulper with 2.2 mm perforations aperture, deaeration at -0.7 bar for 30 minutes at 50-60 °C and pasteurized at 90 °C for 10 minutes.

For total phenol content and antioxidant power analysis, samples containing 8 fruits were frozen at -86 °C for at least 2 days before being lyophilized (Terroni, LD1500A). After, 2.5 grams of dried pulp with peel were mixed with 25 mL of solution containing 1mL formic acid 30% + 200 mL distilled water + 800 mL ethanol 70% in a centrifuge tube. The product was left to stand at 15 °C for 24 hours and then immediately centrifuged (Celm, Combate) for 10 min at 2,232 g (3,500 rpm). The samples were identified and maintained in closed flasks inside a freezer for up to 4 months for total phenol content and antioxidant activity analysis.

For each location, samples of fruits were prepared from 1 day after harvest (DAH) until complete maturation, up to 8 DAH.

## 2.2. Determination of total phenol content

Total phenol content of the ethanol extracts was determined with Folin-Ciocalteu colorimetric method (SINGLETON and ROSSI, 1965) with some modifications. Briefly, 0.1 ml extract was diluted with 8.4 ml distilled water and mixed with 0.5 ml Folin-Ciocalteu reagent. The contents were mixed by manual shaking for 15-20 s.

After 3 min, 1.0 ml of 20% sodium carbonate solution was added. The reaction mixture was incubated at room temperature for 1 h and its absorbance was measured at 720 nm using a dual beam UV-Vis spectrophotometer (Shimadzu, Mod. UV-Mini 1240). Catechin (SIGMA, C-1251) was used as an analytical standard for total phenol quantification and it was expressed as milligrams of catechin equivalents (CE) per gram wet basis (g-WB) of fruit.

TPC analyses were performed in triplicate.

### 2.3. Ferric-reducing antioxidant power (FRAP) assay

Hermetically closed hot filled pasteurized pulp inside glass jars with painted metallic cover was kept for one year at room temperature. This pulp was clarified using pectinase enzyme and centrifuged in a Hitachi HIMAC CR 21GH refrigerated centrifuge with a R12A6 support at 12,300g (11,000 rpm) for 15 minutes. Other samples were just physically separated by centrifugation to study the effect or pectinase enzyme over antioxidant power. Samples were lyophilized and prepared an ethanol extract and kept at freezing temperature (-16 °C) for over 4 months to perform antioxidant analysis.

The reducing ability was determined by using FRAP assay (BENZIE and STRAIN, 1996). The FRAP reagent was freshly prepared from 300 mmol ( $L^{-1}$ ) acetate buffer (pH 3.63), 10 mmol ( $L^{-1}$ ) tripyridyltriazine (TPTZ) made up in 40 mmol ( $L^{-1}$ ) HCl and 20 mmol ( $L^{-1}$ )  $FeCl_3$ . All three solutions were mixed together in the ratio of 10:1:1 (v/v/v). An aliquot of 0.1 mL of the tested sample solution was mixed with 3.0 mL of FRAP reagent. The absorption of the reaction mixture was measured at 593 nm after 15 min incubation at 26 °C. The reducing power was expressed as trolox equivalent (TE) concentration.

FRAP analyses were performed in duplicate.

### 2.4. Statistical Analysis

Analysis were performed randomly and analysis for significant difference in treatment means were analyzed using STATISTICA software (version 5.5, StatSoft Inc., Tulsa, OK, USA) using Tukey ( $p<0.05$ ).

### 3 RESULTS AND DISCUSSION

#### 3.1. Plant material

Fruits differed in size and weight by the location. Table 1 shows the average size and weight and their standard deviation in parentheses. Different letters in the same column mean the values are significantly different ( $p<0.05$ ).

**Table 1** – Dimensions and mass of mume fruits

Location	Diameter (cm)	Height (cm)	Mass (g)
CB	$2.239 \pm 0.125^b$	$2.310 \pm 0.125^b$	$6.892 \pm 1.058^b$
S	$2.130 \pm 0.174^b$	$2.304 \pm 0.199^b$	$5.701 \pm 1.298^b$
SUN	$3.153 \pm 0.264^a$	$3.164 \pm 0.215^a$	$16.909 \pm 3.667^a$

Location SUN is the only commercial mume fruit producer and the mass of the fruits were higher than those from the locations CB and S. In spite of this, the SUN fruits were still at the lower limit of weight as reported by JUN and CHUNG (2008) probably due to genetic improvement in Asian countries, were mume fruits are consumed in larger scale and are part of traditional food. The trees at SUN are over 20 years old and no genetic improvement was carried out during this period. In all fruits and plants used in the present work were not applied pesticides.

Fruit flesh represented  $71.3\% \pm 2.7$  of total weight of fresh fruit. The skin and stone represented  $13.8\% \pm 1.7$  and  $14.9\% \pm 2.0$  respectively. This relative yield of pulp is lower than values reported by TSUBAKI, OZAKI and AZUMA (2010) that obtained  $82.7\% \pm 2.9$  of flesh. This difference is probably due to de smaller size of the fruits of the present work.

Mume fruits are climacteric, in other words, are capable to further metabolize and increase respiration rates after harvesting. TSS did not vary significantly during maturation of fruits and ranged from 9.5 to 10.0 °Brix. Total solids content ranged was 10.2-12.2%. The pH value did not vary much and showed values between 2.5 and 2.7 due a high acid content with TTA values of 4.0-5.7 g (100g<sup>-1</sup>) green-stage fruit and 2.0-3.8 g (100g<sup>-1</sup>) mature-stage fruit, expressed in citric acid. These values are similar to those found in lemons and limes (PENNISTON et al., 2008).

Full-ripe mume fruits presented ratio values under 5. These values are very low and makes product unacceptable by most consumers of fresh fruit. Thus, the consumption of mume fruits is recommended as processed food products, were ratio can we increased by sugar incorporation or acid dilution. These products show a healthy appeal, due to the high antioxidant capacity of these fruits (QUAST et al., 2011).

Pectin content decreased slightly during maturation, from 11.2±0.1% to 10.8±0.1% expressed in dry basis. This provides approximately 1.32% in wet basis that represents about half of pectin content in citric fruits and is similar to apple pomace (BAKER, 1997).

### 3.2. Determination of total phenol content (TPC)

Table 2 shows the mean and standard deviation of total phenol content of mume fruits collected at green-stage maturation from different locations and during maturation until complete ripeness. Results are expressed in grams of fresh fruit.

**Table 2** – Total phenol content of mume pulp+peel collected at different locations and in different stages of maturation (days after harvest – DAH)\*

Samples	Phenolic content (mg CE / g-WB)		
CB - 1 DAH	199	±	21 <sup>abcd</sup>
CB - 2 DAH	207	±	27 <sup>abc</sup>
CB - 3 DAH	228	±	16 <sup>a</sup>
CB - 4 DAH	203	±	31 <sup>abc</sup>
CB - 5 DAH	226	±	2 <sup>a</sup>
CB - 8 DAH (ripe fruit)	215	±	15 <sup>ab</sup>
S - 3 DAH	147	±	27 <sup>e</sup>
S - 4 DAH	161	±	8 <sup>de</sup>
S - 5 DAH	162	±	3 <sup>de</sup>
S - 6 DAH	144	±	11 <sup>e</sup>
S - 7 DAH (ripe fruit)	160	±	31 <sup>de</sup>
SUN - 3 DAH	226	±	7 <sup>a</sup>
SUN - 4 DAH	178	±	20 <sup>bcd e</sup>
SUN - 5 DAH	164	±	15 <sup>cde</sup>
SUN - 6 DAH	148	±	11 <sup>e</sup>
SUN - 7 DAH (ripe fruit)	169	±	7 <sup>cde</sup>

\*Expressed in mg of catechin-equivalent per gram of fruit in wet basis

\*\*Different letters means that the results are significantly different (p<0.05)

\*\*\*Least significance difference (LSD) was 22

According to Table 2 it can be seen that CB fruits presented a trend of higher TPC values compared to other locations. This difference can be a result of difference among cultivars, climate or handling. It also can be observed that TPC did not vary during maturation of fruits, except for SUN-3DAH, that showed higher values compared to other fruits collected at S and SUN locations. This information is highly relevant because most studies about *Prunus mume* products are related to health benefits of green fruits consumption. Therefore, it is possible to develop different products from ripe mume fruits keeping health appeal.

Mume fruits collected from CB location showed mean TPC values of 225 mg CE(g<sup>-1</sup>) wet basis. These values are higher than pineapple, guava, mango, passion fruit, mandarin and grapes frozen pulps (MELO et al., 2008). However, acerola (*Malpighia glabra*) frozen pulp showed TPC values ten times higher than the fruits in the present work.

### 3.3. Ferric-reducing antioxidant power (FRAP) assay

Table 3 shows the FRAP assay of mume fruits collected from different locations and at different stages of maturation. Antioxidant power was expressed as trolox equivalent (TE) in dry basis (DB) or wet basis (WB). Samples were identified with letters according to its origin followed by the number of days after harvest (DAH). Different letters in superscript beside the numbers on the same column denotes significant difference (p<0.05).

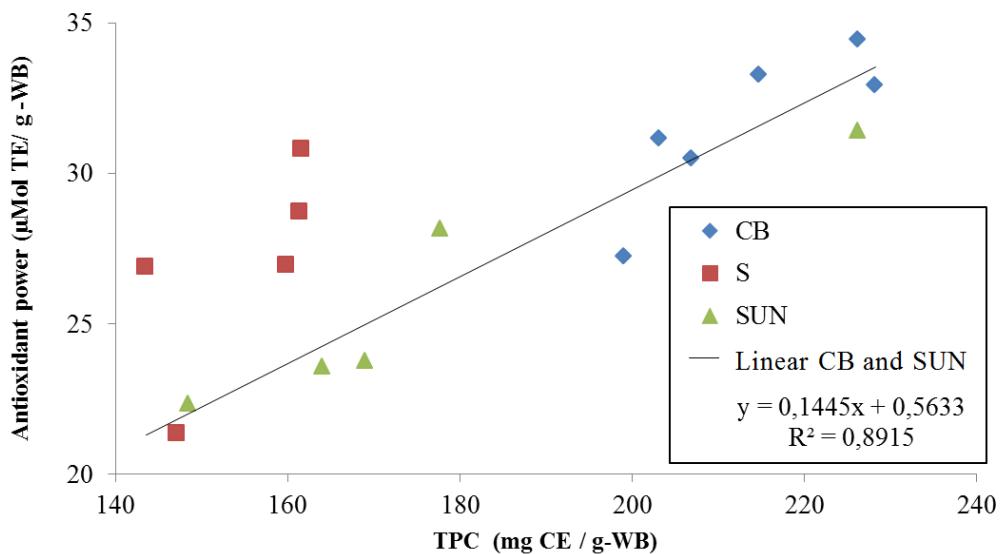
FRAP values did not show any tendency of results during maturation or difference between different regions. This is probably due to the methodology or the fact of the analyses was performed just in duplicate for each treatment. As result, FRAP analysis did not show differences observed in TPC analysis.

**Table 3** – FRAP assay of mume fruits collected at different locations  
and different stages of maturation (days after harvest – DAH)

Samples	Antioxidant power ( $\mu\text{Mol TE g}^{-1}$ ) DB	Antioxidant power ( $\mu\text{Mol TE g}^{-1}$ ) WB
CB - 1 DAH	145.65 $\pm$ 18.62 <sup>ab</sup>	27.24 $\pm$ 3.44 <sup>ab</sup>
CB - 2 DAH	154.21 $\pm$ 24.18 <sup>a</sup>	30.51 $\pm$ 4.07 <sup>ab</sup>
CB - 3 DAH	166.63 $\pm$ 11.09 <sup>a</sup>	32.94 $\pm$ 2.73 <sup>ab</sup>
CB - 4 DAH	153.01 $\pm$ 9.55 <sup>a</sup>	31.17 $\pm$ 2.84 <sup>ab</sup>
CB - 5 DAH	169.26 $\pm$ 5.84 <sup>a</sup>	34.45 $\pm$ 4.85 <sup>a</sup>
CB - 8 DAH (ripe fruit)	162.31 $\pm$ 3.88 <sup>a</sup>	33.27 $\pm$ 1.51 <sup>ab</sup>
S - 3 DAH	96.48 $\pm$ 22.59 <sup>b</sup>	21.35 $\pm$ 5.47 <sup>b</sup>
S - 4 DAH	125.48 $\pm$ 9.51 <sup>ab</sup>	28.74 $\pm$ 2.25 <sup>ab</sup>
S - 5 DAH	131.55 $\pm$ 10.14 <sup>ab</sup>	30.81 $\pm$ 2.58 <sup>ab</sup>
S - 6 DAH	116.52 $\pm$ 0.86 <sup>b</sup>	26.91 $\pm$ 0.39 <sup>ab</sup>
S - 7 DAH (ripe fruit)	116.34 $\pm$ 14.81 <sup>b</sup>	26.96 $\pm$ 4.65 <sup>ab</sup>
SUN - 3 DAH	159.34 $\pm$ 6.95 <sup>a</sup>	31.42 $\pm$ 1.39 <sup>ab</sup>
SUN - 4 DAH	136.59 $\pm$ 12.33 <sup>ab</sup>	28.18 $\pm$ 2.36 <sup>ab</sup>
SUN - 5 DAH	119.21 $\pm$ 21.10 <sup>b</sup>	23.57 $\pm$ 3.95 <sup>b</sup>
SUN - 6 DAH	116.99 $\pm$ 32.34 <sup>ab</sup>	22.35 $\pm$ 6.43 <sup>ab</sup>
SUN - 7 DAH (ripe fruit)	117.25 $\pm$ 10.63 <sup>b</sup>	23.78 $\pm$ 1.88 <sup>ab</sup>

\* LSD was 25.50 and 5.88  $\mu\text{Mol trolox-equivalent (g}^{-1}\text{)}$  in dry basis and wet basis,  
respectively

The antioxidant power is related to the presence of substances capable to react rapidly with oxygen or free-radicals. Therefore, plotting the values of TPC vs. FRAP can show if the antioxidant power is significantly influenced by TPC. This graphic is presented in Figure 1.



**Figure 1** – Relation between antioxidant power and total phenol content

Mume fruits collected in S location showed lower TPC values and different relation FRAP:TPC, compared with other locations. Although fruits from S and CB locations did not show significant difference in weight and dimensions (Table 1), it can be seen that antioxidant power and specially TPC values differed significantly. Probably fruits from location S present different phenol composition with higher antioxidant power that can be result of difference in cultivar, soil and climate or agricultural handling. In fruits collected at CB and SUN locations, increase of antioxidant power was directly proportional to the TPC increase, similar to studies in different European plum genotypes (RUPASINGHE; JAYASANKAR & LAY, 2006).

Table 4 shows the antioxidant power clarified and precipitated fraction of hot filled pasteurized pulp kept for one year at room temperature submitted to physical separation or

using pectinase enzyme. Results are expressed in  $\mu\text{Mol Trolox (g}^{-1}\text{)}$  of dry basis. It can be observed that pectinase clarified pulp presents significantly lower FRAP values. Also, samples clarified just by centrifugation showed higher FRAP values. Thus, although the separation yield is lower, but the antioxidant power is higher in samples separated just using centrifugation.

**Table 4** – FRAP analysis of hot filled pasteurized mume pulp  
kept for one year at room temperature

Samples	Antioxidant power ( $\mu\text{Mol Trolox / g-DB}$ )
Pectinase clarified	28.43 $\pm$ 8.40 <sup>b</sup>
Pectinase precipitate	65.42 $\pm$ 10.28 <sup>a</sup>
Not depectinized clarified	66.50 $\pm$ 1.48 <sup>a</sup>
Not depectinized precipitate	80.52 $\pm$ 15.24 <sup>a</sup>

\*Different letters means that the results are significantly different ( $p<0.05$ )

It can be seen that antioxidant power of hot filled pasteurized mume pulp maintains at least 50% of the original power, when comparing values from Table 4 and values expressed in  $\mu\text{Mol Trolox (g}^{-1}\text{)}$  in dry basis (Table 3). This fact can give an idea of the kinetics of antioxidant power decline in processed products and may aid in the development of products with health properties to the ready to drink juice consumer.

Further, it can be seen that in clarified fraction treated with pectinase enzyme the antioxidant power decreased significantly compared to not depectinized clarified. Mechanical separation can give a clarified juice with antioxidant power close to the whole fruit. QUAST et al. (2011) reported that the peel of mume fruits contain the highest TPC. This peel is incorporated during fruit pulping and contributes to the health benefits of

mume products. Appropriate technology should give an acceptable product without losing its advantageous properties.

#### 4 CONCLUSIONS

The present work showed that mume fruits planted in Southeast Region of Brazil are significantly smaller and yield of pulping is lower when compared to Asian countries fruits. TPC values ranged from 148 to 228 mg CE (g<sup>-1</sup>) WB. Regarding to TPC, it was observed significant differences between CB and S location (208 and 160 mg CE g-WB, respectively). Antioxidant power using FRAP assay showed lower differences between analyzed samples. Values ranged from 96-169 µMol TE (g<sup>-1</sup>) DB and 21-34 µMol TE (g<sup>-1</sup>) WB. TPC and antioxidant power did not vary during fruit maturation, from green-mature fruits. This shows promising applications for fully ripe mume products that can show the same health properties widely presented for green-stage mume fruits. The use of pectinase enzyme decreased significantly the antioxidant power of clarified fraction. This behavior was not observed in mechanical separation. Also, over 50% of antioxidant power was maintained in hot filled pulp kept at room temperature for one year. Thus, *Prunus mume* shows promising characteristics related to crop planting due its rusticity and low need for fertilizers and pesticides.

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## ***Prunus mume* pulp rheological evaluation**

**Artigo a ser encaminhado para a revista Journal of Food Engineering.**

### **ABSTRACT**

A study of the rheological behavior of mume pulp with 6, 7, 8 and 9 °Brix was investigated using a rotational viscometer at temperatures from 15 until 75°C. The data were fitted into rheological models of Bingham, Casson, Herschel-Bulkley and Ostwald-Waele (Power Law). Mume pulp was described as time independent with viscosity of 1.9 Pa.s at 15 °C and 1.1 Pa.s at 65 and 75 °C for 9 °Brix pulp. Non-Newtonian and pseudoplastic (Herschel-Bulkley) characteristic was confirmed by n values under 0.4 and under 0.68 using Ostwald-Waele and Hershel-Bulkley rheological models respectively. The linearization of Arrhenius rheological curve resulted in flow activation energy that ranged from 6.6-10.6 kJ.mol<sup>-1</sup>. Consistence index was influenced more by the total soluble solids concentration than temperature, with values from 22.9-18.0 Pa.s<sup>n</sup> in 9 °Brix pulp and 12.2-8.3 Pa.s<sup>n</sup> in 8 °Brix pulp at temperatures varying from 15 to 75 °C. All models represented high correlation value for all the rheological data obtained at the present work.

**Keywords:** mume pulp, rheological model, consistency, Non-Newtonian, concentration

### **1. Introduction**

*Prunus mume* Sieb. et Zucc (with fruit name in Chinese *Wu Mei*) belongs to *Rosaceae* family and has been widely cultivated in Asia for over 3,000 years. There are about 200 cultivars of mume. Initially the plants were cultivated because of their flowers, but in the past 2,000 years the interest has been focused in the fruit medicinal properties (TSUBAKI; OZAKI and AZUMA, 2010; SHI et al., 2009; SHI and MOY, 2005). Fruiting mume is

consumed mainly in Korea, China and Japan. The tree is cultivated in about 2,700 ha in Korea, 9,000 ha in China and 17,000 ha in Japan with an average production yield of 6.7 ton/ha (JUN and CHUNG, 2008; TOPP; NOLLER and RUSSEL, 2007).

*Prunus mume* Sieb. et Zucc. has been studied as peach (*P. persica*) rootstock, showing promising characteristics, such as rusticity, high resistance to plagues and diseases, adaptation and for reducing the peach and nectarines trees scales (MAYER, PEREIRA and MORO, 2008; MAYER, PEREIRA and SANTOS, 2005). Several mume processed products have been consumed as health food and for the treatment of several diseases because they are rich in bioactive compounds, such as anti-cancer and antioxidants substances (TSUBAKI, OZAKI and AZUMA, 2010; SHI et al., 2009; SHI and MOY, 2005).

Studies on the effects over human health of substances present in mume juice and fruit showed that mumefral, a citric acid derivative, improves the blood flow helping against cardiovascular diseases (UTSUNOMIYA et al., 2002). In Brazil, mume consumption is still almost restricted to the Asian descendants. Mume fruits can be used to make jam by mixing with peaches and plums (CAMPO DALL'ORTO et.al., 1995/1998).

In industrial scale, it is better to use fruit pulp other than handling with fresh fruits. Because of this, it is important to understand the physical-chemical and rheological properties that are largely influenced by the juice or pulp composition and it will depend on the fruit type and treatments to which it was subjected during the manufacturing process (VANDRESEN et al., 2009).

The knowledge of the rheology of fruit juices is useful in quality control, sensory evaluation and engineering applications when industrial plants are designed. The effect of temperature and concentration over the flow properties must be known to calculate and choose the right equipment and size to ensure adequate heat and mass transfer operations (FALGUERA and IBARZ, 2010; CHIN et al., 2009; ALTAN and MASKAN, 2005; NINDO et al., 2005). The aim of this work was to evaluate the rheological behavior of mume pulp at different concentrations and temperatures and fit the obtained data to well-known rheological models.

## 2. Materials and methods

### 2.1. Raw Material – Mume pulp

The mume fruits used for this study were collected in Angatuba, SP, Brazil, at green-mature stage. Before processing, fruits were maintained at room temperature (26 °C) ventilated for over six days until complete ripeness that can be noted by significant change of color to yellow and strong peach-like perfume with floral notes. Overripe fruits present brownish color and wrinkled skin. Batches of about 5 kg ripe fruits were thermally blanched by immersion in boiling water for 120 seconds and then immediately pulped in a pilot-scale brush pulper with 2.2 mm perforations aperture. This equipment was set for maximum speed rotation to maximize pulp extraction. The pulp was poured into a jacketed kettle equipped with vacuum for deaeration at -0.7 bar for 30 minutes at 50-60 °C. Then, valve was closed for pasteurization at 90 °C for 10 minutes with a paddle mixer. Pasteurized pulp was removed using a tube installed on the bottom of the pan and hot-filled into glass jars closed with metal covers. Hermetically closed glass jars were stored at room temperature until its use. Original mume pulp contained 9 °Brix. For values of 8, 7 and 6 °Brix, potable water was used for dilution and concentration was evaluated using a digital refractometer Reichert, AR 200.

### 2.2. Rheological measurements

The rheological behavior of the mume pulp was determined using a rotational viscometer equipped with concentric cylinders (Brookfield DVII + Pro) with SC4-27 spindle and a SC4-45Y assembly coupled to a thermostatic bath Brookfield TC-501 with accuracy of  $\pm 0.1$  °C. The measurements were made at 15, 25, 35, 45, 55, 65 and 75°C for each concentration level of 9, 8, 7 and 6 °Brix. Before each test, samples remained in a thermostatic bath at the same temperature programmed for rheological tests for 10 minutes. The equipment was connected for data acquisition: shear stress, shear rate and apparent viscosity.

To verify if the viscosity of mume pulp is time-dependent, pulp concentrations of 6 to 9 °Brix at 15 to 75 °C had their apparent viscosity measured for up to 180 seconds.

Analyses were carried out varying the shear rate from 0 to 70 s<sup>-1</sup> (ascending curve) and from 70 s<sup>-1</sup> to 0 (descending curve). The shear rate of 0-70 s<sup>-1</sup> was chosen because 10-100 s<sup>-1</sup> approximates the shear rate of tumbling and pouring while 100-1000 s<sup>-1</sup> approximates the shear rate of home mixers (CHIN et al., 2009). The experiments were conducted in duplicate and for each measurement a new sample was used.

During an industrial operation a fluid product is submitted to a range of shear rates and it is important to quantify the effect of the temperature over the viscosity for adequate design and use of equipment (MACEIRAS et al., 2007). The effect of temperature over the viscosity of mume pulp was determined using the Arrhenius equation that is commonly used in literature for rheological studies of juices and pulps (FALGUERA and IBARZ, 2010; VANDRESEN et al., 2009; BELIBGLI and DALGIC, 2007; ARSLAN et al., 2005) and can be expressed by:

$$\eta_a = \eta_0 \exp \frac{E_a}{RT} \quad (1)$$

Where  $\eta_a$  is the apparent viscosity (Pa.s),  $\eta_0$  is an empirical constant (Pa.s),  $E_a$  is the flow activation energy (J.mol<sup>-1</sup>), R value is 8.314J.mol<sup>-1</sup>K<sup>-1</sup> and represents ideal gas constant and T is the absolute temperature (K).

The Arrhenius equation can be linearized as shown:

$$\ln(\eta_a) = \ln(\eta_0) + \frac{E_a}{R} \frac{1}{T} \quad (2)$$

Applying Equation (2), the values of  $\eta_0$  and  $E_a$  is obtained from the linear and the angular coefficient of the plot of  $\ln(\eta_a)$  as function of  $(1/T)$ :

- $\ln(\eta_0)$  = linear coefficient
- $E_a/R$  = angular coefficient

Once analyzed the apparent viscosity behavior, Ostwald-Waele (Power law) model was used to determine rheological parameters as follows:

$$\tau = \kappa(\gamma)^n \quad (3)$$

Where:  $\tau$  is the shear stress (Pa),  $\kappa$  consistency index ( $\text{Pa.s}^n$ ),  $\gamma$  is the shear rate ( $\text{s}^{-1}$ ) and  $n$  is the fluid behavior index (dimensionless).

Herschel-Bulkley, Casson and Bingham models are commonly used for describing non-Newtonian fluids. They can be described as followed:

$$\text{Casson:} \quad \tau^{0.5} = \kappa_{oc} + \kappa_c (\gamma)^{0.5} \quad (4)$$

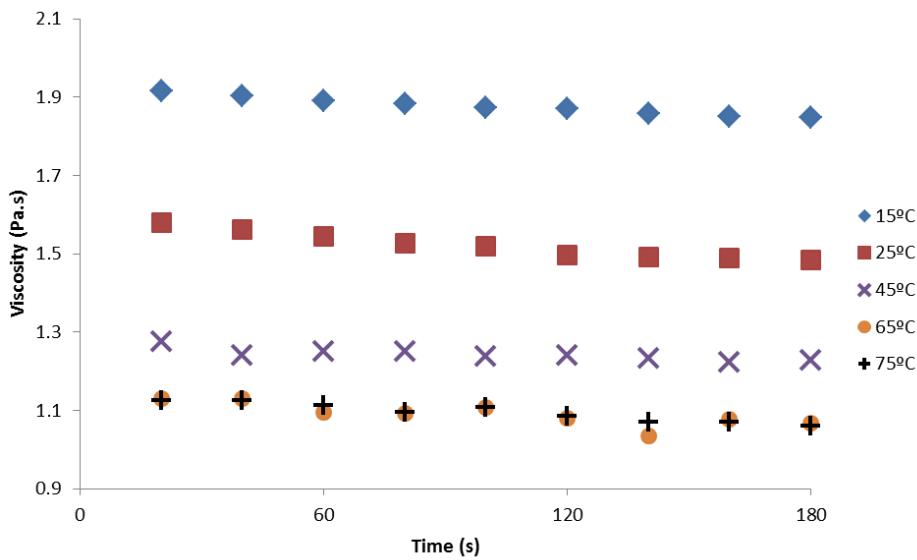
$$\text{Bingham:} \quad \tau = \tau_0 + \eta_\infty \gamma \quad (5)$$

$$\text{Herschel-Bulkley:} \quad \tau = \tau_0 + \kappa(\gamma)^n \quad (6)$$

Where:  $\tau_0$  and  $\kappa_{oc}$  are the yield stress (Pa);  $\kappa$  and  $\kappa_c$  are the consistency indexes ( $\text{Pa.s}^n$ ) and  $\eta_\infty$  is consistency index ( $\text{Pa.s}^n$ ) or Casson viscosity.

### 3. Results and discussion

Figure 1 shows the results of apparent viscosity and its variation with time using mume pulp with 9 °Brix concentration. Similar behavior was obtained for the other concentrations but data are shown at the end of the work, in Figures A.1, A.2 and A.3. Data were obtained maintaining the spindle rotation constant at 40 rpm.



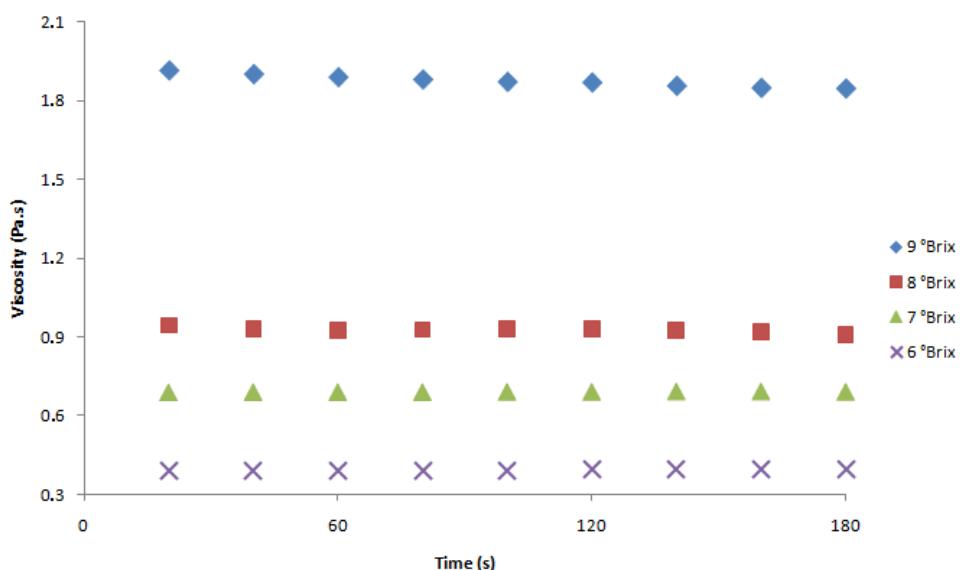
**Figure 1** – Apparent viscosity with time for temperatures of 15 to 75°C at 9 °Brix concentration

The apparent viscosity at temperatures ranging from 15 to 75°C and 9 °Brix concentration showed that constant apparent viscosity values were obtained after a period of 3 minutes. During this period, viscosity was practically constant with time. This behavior is characteristic of time non-dependent fluids. The knowledge of this information is important when designing equipment or industrial processes, such as pasteurization or sterilization of pulps or juices. The mume pulp exhibited a typical juice behavior, where apparent viscosity decreases with the increase of temperature.

In Figure 1 it also can be seen that the temperature effect over apparent viscosity was more notably at lower temperatures. The raise of temperature from 15 to 25 °C lowered apparent viscosity values in 21% from 1.9 to 1.5 Pa.s. Temperature raising in 20 °C, from

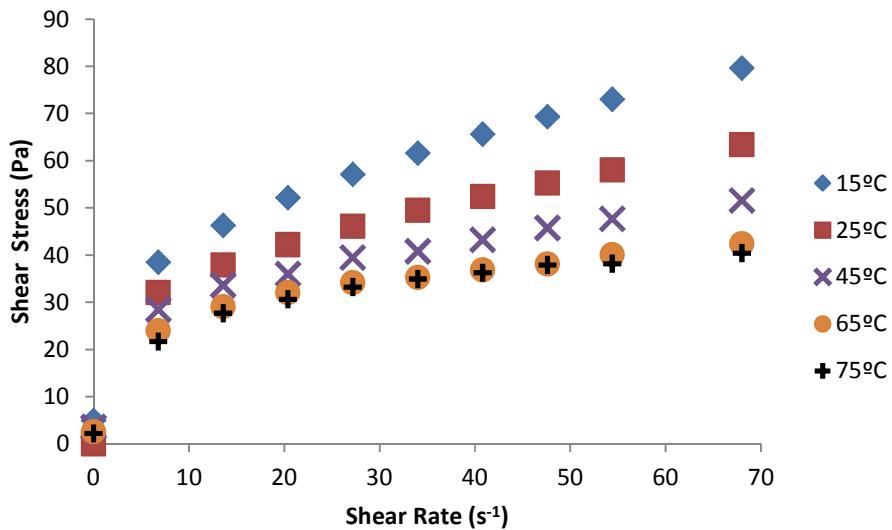
45 to 65 °C lowered apparent viscosity in 12% from 1.25 to 1.1 Pa.s and no difference was observed in apparent viscosity of mume pulp with 9 °Brix at 65 and 75 °C.

At the present study, as can be noted in Figure 2, the decrease of 1 °Brix resulted in significant reduction of the apparent viscosity. This same behavior was observed at higher temperatures, but is not shown. Thus, total soluble solids content should be strictly controlled in an industrial application such as heating treatment, where the apparent viscosity can greatly influence the final product quality.



**Figure 2** – Apparent viscosity with time at 15 °C for different concentrations

Figure 3 shows the Shear Stress (Pa) versus Shear Rate ( $s^{-1}$ ) variation. Measurements were performed at temperatures from 15 to 75 °C and constant 9 °Brix concentration. Similar graphs were obtained for the other concentrations but no new information can be obtained, so these data are shown at the end of the work, in Figures A.4, A.5 and A.6.



**Figure 3** – Shear Stress verus shear rate at temperatures from 15 to 75°C at 9 °Brix concentration.

All samples (9, 8, 7 and 6 °Brix) evaluated at temperatures from 15 to 75°C showed a non linear relation of shear stress and shear rate, indicating non-Newtonian behaviour as reported by several authors (FALGUERA and IBARZ, 2010; IGUAL et al., 2010; CHIN et al., 2009; TIZIANI and VODOVOTZ, 2005) for fruit juices. Preliminary experiments showed no difference between increase or decrease of shear stress for mume pulp indicating no thixotropic effect (BELIBAGLI and DALGIC, 2007).

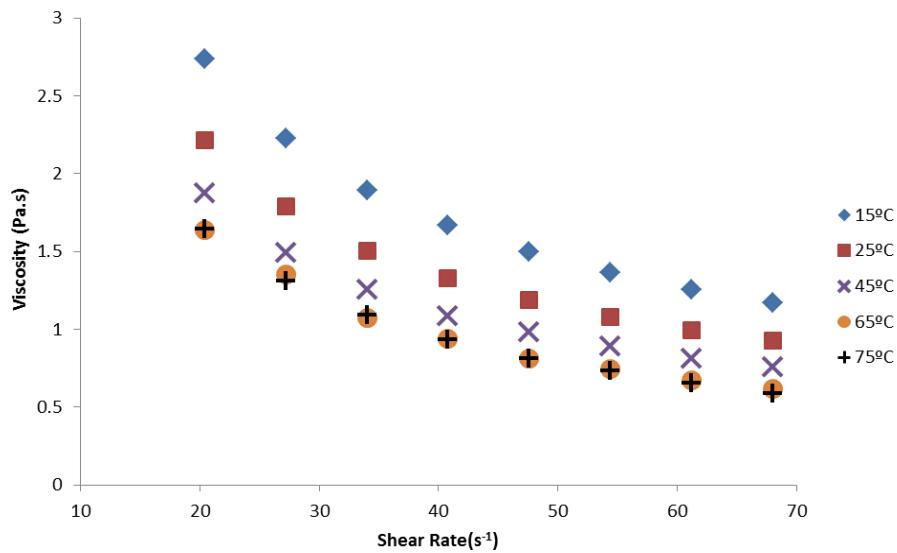
Table 1 presents the calculated values of  $\eta_0$  and  $E_a$  and the regression coefficient ( $R^2$ ) obtained from the linearization of Arrhenius equation for mume pulp flow at different shear rates. Graphics of Arrhenius equation linearization are shown at the end of the work, in Figures A.10 and A.11.

**Table 1** – Model parameters obtained for Arrhenius equation

Samples	20 s <sup>-1</sup>			41 s <sup>-1</sup>			68 s <sup>-1</sup>		
	(°Brix)	$\eta_0$ (Pa.s)	Ea (kJ.mol <sup>-1</sup> )	R <sup>2</sup>	$\eta_0$ (Pa.s)	Ea (kJ.mol <sup>-1</sup> )	R <sup>2</sup>	$\eta_0$ (Pa.s)	Ea (kJ.mol <sup>-1</sup> )
6	0.0153	8.4739	0.9934	0.0055	9.9076	0.9969	0.003	10.6463	0.9991
7	0.0342	8.0023	0.9958	0.0179	8.4095	0.9959	0.0108	8.819	0.9958
8	0.0819	6.6012	0.9472	0.0484	6.6037	0.9429	0.0255	7.2672	0.9492
9	0.142	6.9361	0.9406	0.0573	7.9126	0.9466	0.0237	9.2167	0.9798

$E_a$  indicates the viscosity sensibility to temperature variations. High  $E_a$  values indicate that viscosity varies a lot with changes of temperature. As it can be seen in Table 1,  $R^2$  exceeded 94% in all cases, indicating an adequate adjustment. The value of flow activated energy growed slightly with the increase of shear rate. This fact can be seen in Figure 3, as well as the decrease of apparent viscosity with more intensive deformations. Thus, heat transfer processes can be helped by the agitation in different stages of processing (FALGUERA and IBARZ, 2010). Considering the largest shear rate (68 s<sup>-1</sup>), the  $E_a$  value to 9 °Brix was 10.64 kJ.mol<sup>-1</sup>. This result is similar to the obtained by ALTAN and MASKAN (2005), ( $E_a = 8.6$  kJ.mol<sup>-1</sup>) for pomegranate juice at 35.2 °Brix and by GRATÃO et al. (2007), ( $E_a = 11.59$  kJ.mol<sup>-1</sup>) for soursop juice at 9.3 °Brix. The values of  $E_a$  showed no clear trend regarding changes in concentration. Similar behavior was observed by GRATÃO et al. (2007) for soursop juice and CHIN et al. (2009) for pomelo juice.

Figure 4 shows the apparent viscosity (Pa.s) as a function of the shear rate. Measurements were performed at temperatures from 15 to 75 °C with 9 °Brix pulp concentration. Similar graphs were obtained for the other concentrations but no new information could be obtained, so these data are shown at the end of the work, in Figures A.7, A.8 and A.9.



**Figure 4** – Mume pulp apparent viscosity versus shear rate at temperatures from 15 to 75°C and 9 °Brix.

Regarding Figure 4, changes in viscosity with the increase of shear rate showed a pseudoplastic behavior and consequently a non-Newtonian characteristic fluid for all temperatures (MACEIRAS et al., 2007; TIZIANI and VODOVOTZ, 2005). The Non-Newtonian behavior of mume pulp can be attributed to the presence of soluble pectin around 11% in dry basis as well as other soluble fibers. Rheological properties of most liquid foods change during the processing stages due to their dependence upon temperature and concentration (GRATÃO et al., 2007). The apparent viscosity of mume pulp decreased with the raise of the temperature, as previously observed in Figure 1. This same behavior can be seen in Figures 3 and 4 (Shear Stress vs. Shear Rate).

Table 2 presents the data obtained for the adjustment in accordance with the Power Law model for mume pulp at different concentrations and temperatures. Due to an experiment limitation, it was not possible to obtain data for 6 °Brix at 75°C. The graphics of the linearization of Ostwald-Waele model are shown at the end of the work, in Figures A.12 and A.13.

**Table 2** – Power law parameters for mume pulp at different concentrations and temperature

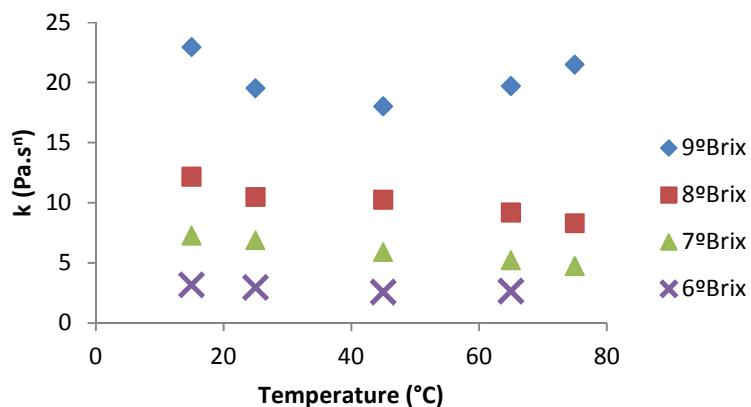
		Temperature (°C)				
Samples (°Brix)	Parameters	15	25	45	65	75
9	n	0.2937	0.2757	0.2465	0.1789	0.1519
	$\kappa$ (Pa.s <sup>n</sup> )	22.9439	19.5389	18.0164	19.7227	21.4914
8	R <sup>2</sup>	0.9996	0.9976	0.9937	0.9538	0.9706
	n	0.2702	0.2497	0.2275	0.2243	0.2249
7	$\kappa$ (Pa.s <sup>n</sup> )	12.1609	10.487	10.2519	9.1811	8.3157
	R <sup>2</sup>	0.9978	0.9981	0.9961	0.9963	0.9897
6	n	0.3288	0.3104	0.2913	0.2829	0.2752
	$\kappa$ (Pa.s <sup>n</sup> )	7.2623	6.8795	5.8905	5.2258	4.7333
	R <sup>2</sup>	0.9994	0.9991	0.9983	0.9989	0.9997
	n	0.405	0.384	0.3489	0.2903	--
	$\kappa$ (Pa.s <sup>n</sup> )	3.1756	2.9711	2.5834	2.6686	--
	R <sup>2</sup>	0.999	0.9987	0.9968	0.9962	--

Regarding the results shown in Table 2, it is possible to observe that n values were smaller than a unity indicating non-Newtonian and pseudoplastic behavior at all concentrations and temperatures levels. The n values decreased with the increase of concentration. Similar behavior was observed by ARSLAN et al. (2005) for sesame paste/concentrated grape juices blend and by GRATÃO et al. (2007) for soursop juice. Additionally, as expected, the flow behavior index slightly decreased with the raise in temperature for several concentrations.

For fruit juices, the flow behavior index usually decreases with the increase in concentration of soluble solids because dilute juices tend to show rheological behavior

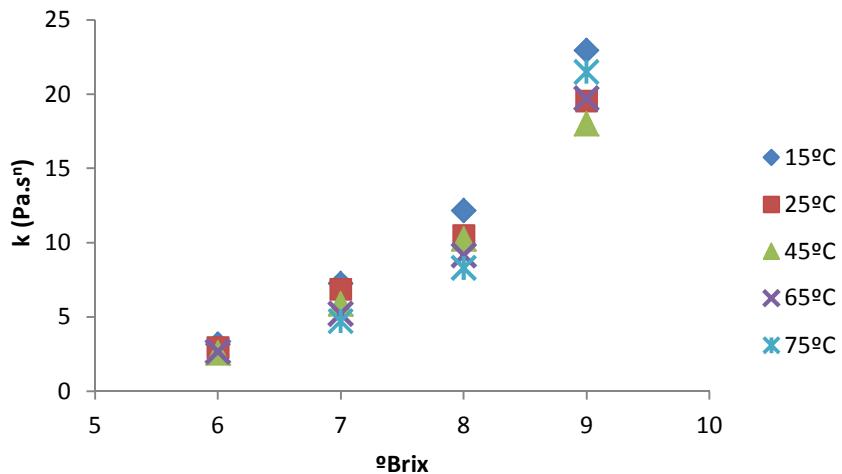
similar to water. The value of  $n$  usually increases slightly with the rise of temperature. Because these variations are quite small, some authors consider  $n$  as constant (GRATÃO et al., 2007). The fluid behavior for mume pulp at 8 and 9 °Brix were similar to those found to prune puree at 20 to 40°C studied by MACEIRAS et al. (2007).

Both analyses of  $n$  and  $\kappa$  values are important in juice processing. For example, the increase in consistency index will decrease the flowing rate of juice in the pipe due to the more resistance to flow. This means that holding time in flow lines during pasteurization at recommended temperature will be longer for juices with higher total soluble solids for the same pressure drop (CHIN et al., 2009). Figures 5 and 6 show the consistency index ( $\kappa$ ) as function of temperature and °Brix respectively.



**Figure 5** – Consistency index of mume pulp as a function of temperature for various concentrations.

It can be seen in Figure 5 that in general consistency index decreases with the raise of temperature, as expected. But the increase of the temperature over 60 °C for mume pulp with 9 °Brix showed decrease of  $\kappa$ . This can be result of chemical reactions and formation of products with higher consistency index. It also can be seen in Figure 6 that the mume pulp consistency index changed more significantly with the modification of concentration than variation of temperature. Thus, a small difference of pulp concentration form different products from the same production batch can give different consistency index and this effect can be more significant than the raise of temperature.



**Figure 6** – Consistency index of mume pulp as a function of Brix for various temperatures

The mume pulp consistency index changed more significantly with the modification of concentration than variation of temperature. The consistency index increased exponentially with the concentration increase. The  $\kappa$  value for mume pulp at 9 °Brix and 25°C was about 19.54 Pa.s<sup>n</sup>, which is higher when compared with soursop juice (1.45 Pa.s<sup>n</sup>) at 9.3 °Brix and 28.4°C (ARSLAN et al., 2005).

Tables 3, 4, 5 and 6 show the models parameters of Casson and Bingham for mume pulp at temperatures from 15 to 75°C and °Brix varying from 9 to 6. Due to an experiment limitation, it was not possible to obtain data for 6 °Brix at 75°C.

**Table 3** – Casson and Bingham models parameters for mume pulp at 6 °Brix

Temperature (°C)	Casson			Bingham		
	$\kappa_{0c}$	$\kappa_c$	R <sup>2</sup>	$\tau_0$	$\eta_\infty$	R <sup>2</sup>
15	2.213	0.242	1.000	8.231	0.142	0.995
25	2.132	0.214	0.999	7.383	0.116	0.995
45	1.965	0.171	1.000	5.950	0.081	0.997
65	2.633	0.294	0.996	5.419	0.057	0.996

**Table 4** – Casson and Bingham models parameters for mume pulp at 7 °Brix

Temperature (°C)	Casson			Bingham		
	$\kappa_{0c}$	$\kappa_c$	R <sup>2</sup>	$\tau_0$	$\eta_\infty$	R <sup>2</sup>
15	3.291	0.258	0.999	16.113	0.198	0.992
25	3.180	0.230	0.999	14.654	0.166	0.993
45	2.917	0.193	1.000	12.011	0.124	0.994
65	2.740	0.173	0.999	10.469	0.104	0.993
75	2.606	0.157	0.997	9.347	0.088	0.986

**Table 5** – Casson and Bingham models parameters for mume pulp at 8 °Brix

Temperature (°C)	Casson			Bingham		
	$\kappa_{0c}$	$\kappa_c$	R <sup>2</sup>	$\tau_0$	$\eta_\infty$	R <sup>2</sup>
15	4.154	0.247	0.999	23.651	0.220	0.993
25	3.830	0.203	0.995	19.522	0.161	0.985
45	3.735	0.177	0.998	18.068	0.134	0.991
65	2.431	0.323	0.874	8.273	0.273	0.723
75	3.389	0.152	0.973	14.731	0.103	0.949

**Table 6** – Casson and Bingham models parameters for mume pulp at 9 °Brix

Temperature (°C)	Casson			Bingham		
	$\kappa_{0c}$	$\kappa_c$	R <sup>2</sup>	$\tau_0$	$\eta_\infty$	R <sup>2</sup>
15	5.776	0.383	0.998	47.163	0.490	0.990
25	5.277	0.323	0.999	38.469	0.369	0.993
45	4.991	0.264	0.998	33.117	0.275	0.995
65	5.051	0.175	0.954	31.039	0.168	0.944
75	5.210	0.144	0.942	31.895	0.135	0.906

Tables 7 and 8 show the Herschel-Bulkley model parameters for mume pulp at temperatures from 15 to 65°C and °Brix varying from 9 to 6. Due to an experiment limitation, it was not possible to obtain data for 6 °Brix at 75°C.

**Table 7** – Herschel-Bulkley model parameters for mume pulp at 6 and 7 °Brix

Temperature (°C)	6 °Brix					7 °Brix				
	$\tau_0$	$\kappa$	n	R <sup>2</sup>	$\tau_0$	$\kappa$	N	R <sup>2</sup>		
15	4.236	4.236	0.587	1.000	7,203	3.011	0.471	1.000		
25	4.054	4.054	0.583	1.000	7.570	2.341	0.484	1.000		
45	4.408	4.408	0.678	1.000	7.720	1.300	0.537	1.000		
65	4.133	4.133	0.636	1.000	5.929	1.516	0.478	1.000		
75	-	-	-	-	1.979	3.333	0.325	1.000		

**Table 8** – Herschel-Bulkley model parameters for mume pulp at 8 and 9 °Brix

Temperature (°C)	8 °Brix					9 °Brix				
	$\tau_0$	$\kappa$	n	R <sup>2</sup>	$\tau_0$	$\kappa$	n	R <sup>2</sup>		
15	15.360	2.593	0.516	1.000	19.335	10.464	0.414	1.000		
25	7.947	4.862	0.360	0.998	25.633	3.900	0.535	1.000		
45	12.881	1.645	0.508	0.998	26.734	1.715	0.632	0.998		
65	1.272	8.235	0.237	1.000	19.606	4.689	0.371	0.956		
75	2.531	10.496	0.196	0.991	4.884	17.371	0.172	0.968		

Determination coefficients obtained in the adjustment of experimental data to Herschel-Bulkley and Casson where equal or similar to 100%, as shown in Tables 3 to 8. The non-linear regression for Herschel-Bulkley model showed that yield stress ( $\tau_0$ ) did not show consistent behavior regarding to temperature variation. This situation was reported by FALGUERA and IBARZ (2010) to concentrated orange juice at temperatures from -12 until 30°C. However, it was observed that yield stress values increased with the raise of

concentration. Similar behavior was observed for the consistency index ( $\kappa$ ). Taking as an example, the mume pulp with 8 °Brix at 15 to 25 °C, showed consistence index of 3.73 Pas<sup>n</sup>. At the same temperature range, for carrot juice at 8.04 °Brix, the consistency index is 0.03067 Pas<sup>n</sup> (VANDRESEN et al., 2009), which indicates that the mume pulp has, in addition of soluble solids, other components which increase its consistency. The consistency values for mume pulp were similar to those obtained for concentrated orange juice (FALGUERA and IBARZ, 2010). Regarding to Tables 7 and 8, flow behavior index (n) values were higher than those determined by the power law. However, all values remained less than unity confirming a pseudoplastic (Herschel-Bulkley) behavior.

#### 4. Conclusions

Modeling of the rheological behavior of mume pulp showed that it is time non-dependent, non-Newtonian and presents a pseudoplastic (Herschel-Bulkley) behavior, similar to other fruit juices. The consistency of mume pulp depends strongly on its concentration and in less importance, the temperature. Herschel-Bulkley, Casson, Bingham and Ostwald-Waele models represented well the rheological data with high correlation coefficient values.

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# **Micro reestruturação da polpa de umê para adição ao suco clarificado**

**Artigo a ser enviado para revista científica, em definição.**

## **RESUMO**

Durante o despolpamento do umê, as células vegetais que compõem a polpa são destruídas. Porém, este material pode ser micro reestruturado e permanecer novamente na forma de partículas em suspensão, processo este geralmente auxiliado por hidrocolóides. Para o processo de micro reestruturação, podem-se utilizar diferentes tipos de gomas, como gelana e alginato de sódio, levando-se em consideração os valores de pH do meio onde os pedaços de goma com polpa serão mantidos. Em valores de pH abaixo de 3,5 os pedaços solubilizam-se novamente no suco. No caso da polpa de umê, cujo pH natural é de aproximadamente 2,7, o pH deve ser ajustado (aumentado para 3,5), pois em valores de pH acima de 4,5 o sabor da polpa é afetada, perdendo o amargor e a acidez e ficando levemente salgada. Foram produzidas partículas esféricas, com diâmetro de aproximadamente 3 mm. A elevação do pH para, no mínimo 3,5 mostrou-se condição necessária à estabilidade física das partículas. A adição de 0,5% alginato de sódio à polpa de umê com pH corrigido, mergulhadas em solução com 1% cloreto de cálcio, por período superior a 5 minutos forneceu partículas esféricas estáveis no suco clarificado de umê.

## **1. INTRODUÇÃO**

O consumo de bebidas não alcoólicas tem apresentado forte crescimento no Brasil. Dentro deste mercado, existem as bebidas com sabor de frutas, que podem ser classificadas como: 1) sucos naturais, obtidos diretamente da fruta por prensagem ou extração física, como no caso do suco natural de laranja ou de maçã; 2) sucos desidratados, produzidos a partir da retirada de água, geralmente em *spray-drier*, que contribui para o barateamento na logística e armazenamento, pela diminuição do peso, volume e da atividade de água do produto; 3) sucos concentrados, obtidos pela retirada parcial de água, realizada geralmente em evaporadores de múltiplos efeitos, visando diminuição do peso e do volume, e preservando a maior parte das características da matéria prima original; 4) sucos reconstituídos, obtidos pela diluição de suco concentrado ou desidratado, até a concentração original do suco integral ou ao teor mínimo de sólidos solúveis estabelecidos pelo padrão de identidade e qualidade (PIQ) do suco; 5) néctares de frutas, que são sucos formulados prontos para beber, onde podem ser adicionados água, adoçantes naturais ou artificiais e ácido orgânico predominante da fruta. Neste caso, o produto deve conter no mínimo 30% da polpa de fruta, salvo os casos de frutas de sabor ou acidez muito forte; 6) polpas congeladas, caracterizadas pela ausência de qualquer adição de ingredientes, obtida da extração, seguida do congelamento para a conservação do produto (ROSA, COSENZA & LEÃO, 2006; BRASIL, 2003; WELTI-CHANES, BARBOSA-CÁNOVAS & AGUILERA, 2002).

No panorama mundial, as bebidas a base de frutas são principalmente comercializadas na forma concentrada, sendo que o suco de laranja é ainda o principal suco comercializado mundialmente. O suco de laranja responde por cerca de 40% do consumo mundial de sucos seguido pela maçã, que corresponde a aproximadamente 25% deste mercado. As principais companhias que atuam na comercialização de sucos são: Gerber Foods, Grupo Fischer (Citrosuco), Refresco Group, Stute Foods, Conserve Itália, Procter & Gamble, Riedel Drinks , PepsiCo e Coca-Cola Company (ROSA, COSENZA & LEÃO, 2006).

No Brasil, o mercado de bebidas com sabor de frutas tem apresentado crescimento vigoroso, na ordem de 11 a 13% ao ano, nos últimos 15 anos, especialmente na categoria de sucos prontos para beber, o que tem incentivado o ingresso de várias empresas neste mercado. Este aumento do consumo alterou significativamente a produção de uvas, com o aumento da produção de suco de uva em detrimento da produção de vinhos a partir de uvas americanas, não recomendadas para a elaboração de vinhos. Segundo IBRAVIN (2011), a partir de 1999 o suco de laranja deixou de ser o principal sabor no mercado nacional de sucos prontos para beber, onde atualmente ocupa a terceira posição, liderada pelo suco de uva e seguida do sabor pêssego.

No mercado nacional de bebidas com sabor de frutas, os produtos em pó ainda apresentam grande participação, devido ao seu baixo custo e facilidade de armazenamento e conservação. Porém, o consumo de néctares tem aumentado significativamente, principalmente pelo forte apelo saudável, aumento do poder aquisitivo médio da população e praticidade (CITRUSBR, 2010).

A maior parte dos sucos prontos para beber são néctares de frutas. No Brasil, o Decreto nº 6.871 de 6 de junho de 2009 regulamenta a Lei nº 8.918, de 14 de julho de 1994, para os produtos comerciais, onde os de maior importância são os néctares e as bebidas à base de soja (BRASIL, 2009). A Instrução Normativa nº 14, de 04 de setembro de 2003 estabelece os padrões de identidade e qualidade de sucos tropicais e néctares de frutas (BRASIL, 2003). Dessa forma, são estabelecidos limites mínimos de polpa, bem como a lista de aditivos permitidos.

No Brasil, os néctares e os sucos integrais são geralmente comercializados em embalagens de vidro de 500 mL, lata de 335 mL, ou em cartonados de 200 ou 1000 mL. O desenvolvimento de novos sabores e tipos diferenciados de produtos foram os principais responsáveis por este aumento do consumo. Porém, este mercado ainda encontra-se distante do seu potencial, fato que fica evidente quando se compara o consumo de sucos na ordem de 8 litros por habitante-ano no Brasil, com o consumo na Europa e Estados Unidos, na ordem de 20 e 40 L/(hab.ano), respectivamente (ROSA, COSENZA & LEÃO, 2006).

Segundo uma tendência mundial, a maior demanda por produtos saudáveis beneficia o consumo de sucos no Brasil. Com a crescente preocupação com o aumento da obesidade e doenças associadas ao aumento da massa corporal da população brasileira, os sucos *diet* e *light* adquiriram rápida e intensa aceitação no mercado nacional (ROSA, COSENZA & LEÃO, 2006).

Outra tendência atual é o desenvolvimento de sucos com partículas em suspensão. Estudos de mercado apontaram que os consumidores associam a presença de partículas ou pedaços de frutas com um produto saudável (SIG, 2012). No Brasil, o lançamento de suco de laranja com “gomas” segue este apelo. Com isso, o produto chega ao mercado a um valor muito acima do néctar comum de laranja.

No caso de frutos onde não é possível o corte ou a separação mecânica de pedaços, durante o despolpamento do fruto ocorre a perda do formato e da textura do fruto. Portanto, para a elaboração de partículas em suspensão, pode-se unir a polpa na forma de partículas e garantir a sua estabilidade durante os processos de transporte no interior de tubulações, pasteurização, envase e estocagem. Frutas reestruturadas podem ser produzidas com o uso de hidrocolóides como goma gelana, alginato ou pectina de baixo grau de metoxilação, seguidas da retirada parcial da água (GRIZOTTO, AGUIRRE & MENEZES, 2005). Partículas de menor tamanho podem ser obtidas pela alimentação da polpa adicionada de hidrocolóides a uma solução contendo cálcio, para permitir a combinação do cálcio à estrutura formada pelo hidrocolóide. A massa específica da partícula formada deve ser a mais próxima possível da massa específica do suco onde ficará mergulhado, para evitar ou retardar a separação física. A viscosidade do suco pode ser aumentada com o uso de gomas, podendo impedir ou retardar a separação física de fases, fazendo com que as partículas permaneçam igualmente distribuídas no produto (CPKELCO, 2011).

A Lei de Stokes (Equação 1) mostra que a velocidade terminal de uma partícula imersa em um fluido é inversamente proporcional à viscosidade ( $\mu$ ), ou seja, quanto maior a viscosidade do fluido, maior será o tempo necessário para a separação (decantação ou flotação) da partícula. É possível observar também, que para minimizar a velocidade de separação, é necessário que a massa específica da partícula ( $\rho_p$ ) seja a mais próxima possível da massa específica do fluido ( $\rho$ ).

$$v_t = \frac{(\rho_p - \rho)gD_p^2}{18\mu} \quad (1)$$

Onde:  $v_t$  = velocidade terminal da partícula;

$g$  = aceleração da gravidade ( $9,81 \text{ m/s}^2$ );

$D_p$  = diâmetro característico da partícula.

O desejo do consumidor brasileiro por sucos de diferentes sabores e que apresentem benefício à sua saúde pode ser suprido pelo desenvolvimento de néctares de frutas tropicais ou exóticas como o umê (*Prunus mume*). Diversos trabalhos atribuem ao consumo de frutos de umê benefícios à saúde, como a melhoria da fluidez do sangue, capaz de prevenir doenças coronárias e o combate ao câncer em diversos órgãos e em estágios diferentes, assim como o auxílio na prevenção de doenças degenerativas, pelo combate aos radicais livres formados no organismo (TSUBAKI; OZAKI and AZUMA, 2010; SHI et al., 2009; SHI and MOY, 2005; UTSUNOMIYA et.al., 2002). Além disso, apresenta grande rusticidade agrícola, o que permite vislumbrar a produção industrial de umê sem a necessidade de defensivos agrícolas.

Além disso, o sabor do umê lembra o pêssego, com um aroma mais acentuado e mais adocicado. No entanto, o suco de umê apresenta elevada acidez e grande amargor. Grande parte do amargor pode ser retirada pela clarificação do suco, por centrifugação ou com o auxílio de enzimas pectinases. Porém, o clarificado resultante apresenta um poder antioxidante significativamente inferior ao original. Dessa forma, o presente trabalho teve como objetivo estudar a micro reestruturação da polpa de umê, com elevado amargor e elevada atividade antioxidante, para posterior adição ao suco clarificado, de modo a obter um produto diferenciado com elevado poder antioxidante.

## **2. MATERIAL E MÉTODOS**

### **2.1. Preparo da polpa**

Os frutos de umê foram colhidos na região de Angatuba-SP, ainda no estágio verde de maturação, com início de coloração amarelada ou verde clara. Em seguida, foram mantidos em local protegido, mas com aberturas para permitir a respiração dos frutos.

De 6 a 8 dias após a colheita, os frutos maduros foram selecionados pela coloração amarelada dos frutos. Neste ponto, a acidez total titulável decresce significativamente e ocorre formação de perfume semelhante ao de pêssego, mas de maior intensidade.

Lotes de 5 kg de frutos maduros foram branqueados termicamente pela imersão em água em ebulação por 120 segundos. O tempo mínimo de branqueamento foi avaliado pela inativação da enzima peroxidase. Amostras de 8 frutos inteiros foram imersos em água em ebulação por períodos de 20 a 120 segundos, resfriados em água corrente logo após este período. Em seguida, foram cortados, imersos em água destilada, adicionado 1 mL solução de guaiacol-1%, seguido de 1 mL peróxido de hidrogênio 0,5%, por 120 segundos. A coloração marrom é indício de ação da enzima peroxidase.

Testes preliminares foram realizados para avaliar o rendimento do despolpamento com a instalação de um inversor de frequência no motor do despolpador. A faixa estudada foi para a frequência entre 40 e 60 Hz. Para a obtenção da polpa de umê, o despolpamento foi realizado logo após o branqueamento, ainda com os frutos quentes, em despolpador tipo escovas com tela perfurada com aberturas de 2,2 mm, com máxima velocidade de rotação, para obter máximo rendimento, conforme resultados dos testes preliminares.

Em seguida, a polpa foi desaerada por 30 minutos, 50 a 60 °C em tacho bule encamisado à vácuo, dotado de pá misturadora. A polpa desaerada, ainda no bule desaerador, foi rapidamente aquecida até 80 °C, mantida por 10 minutos e imediatamente envasada em potes de vidro com 600 mL de capacidade, com tampa metálica. Os potes foram invertidos por 5 minutos, para garantir a pasteurização da tampa.

Após a pasteurização, os produtos foram resfriados com água corrente sobre a tampa. Este preparo da polpa pasteurizada foi realizado na planta piloto de processamento de Frutas e Hortaliças da Faculdade de Engenharia de Alimentos da UNICAMP. A polpa de umê pasteurizada foi mantida ao abrigo da luz, em temperatura ambiente até o momento do uso.

## 2.2. Clarificação e obtenção das partículas micro estruturadas de polpa de umê

Em um dos tratamentos a clarificação da polpa foi realizada fisicamente em centrífuga refrigerada, modelo Hitachi HIMAC CR 21GII, com suporte R12A6 com 12.300g (11.000 rpm), por 15 minutos, resultando em uma fração clarificada e um precipitado. Na clarificação físico química, a polpa foi previamente tratada com enzima pectinolítica Pectinex 100L Plus (Novozymes®), na concentração de 0,03 mg/ L, a 25 °C por 2 horas. Após a ação das enzimas, a mistura foi centrifugada a 12.300g por 15 minutos. O pH da polpa não foi alterado, mantendo-se a 2,7.

O pH da polpa de umê foi analisado e diferentes amostras foram elaboradas, elevando-se o valor do pH de 2,7 até o valor de 4,5 com a adição de citrato de sódio, para verificar o efeito do pH sobre a estabilidade das partículas de polpa com hidrocolóide. O suco clarificado também teve o seu pH elevado para os mesmos valores utilizados para as partículas.

Para obtenção das partículas micro estruturadas de polpa de umê, concentrações de 0,5 a 2,0% de alginato de sódio foram misturadas à polpa. Em seguida, gotejou-se esta polpa a um ângulo de 45°, a uma altura que variou de 2 mm até 10 cm, entre a ponta do conta-gotas e a solução resfriada a 6 °C de cloreto de cálcio (concentração entre 1,0 e 2,0%), determinando-se as condições ideais para formação de partículas praticamente esféricas. Após o tempo pré-determinado de imersão, as micro partículas foram separadas da solução com o auxílio de uma peneira com abertura de 1 mm.

A aparência e a textura das esferas formadas foram avaliadas em diversos tempos após imersão das partículas na solução de cloreto de cálcio. As partículas foram avaliadas

visualmente e foram experimentadas sensorialmente pela equipe participante do projeto, para verificar a presença de sabor ou textura estranha.

A estabilidade das partículas formadas foi avaliada mantendo estas imersas em suco clarificado com pH ajustado aos mesmos valores de pH por 24 horas, em repouso, à temperatura ambiente. As amostras que resistiram à imersão no suco clarificado por 24 horas foram submetidas ao aquecimento até 80 °C por 5 minutos, sem agitação, avaliando-se também sua resistência térmica.

Para preparo do néctar clarificado de umê, foi adicionado 0,5% de goma gelana kelcogel PS (CP Kelco®), com uso de um mixer, para preparo de lotes de 2 litros de produto.

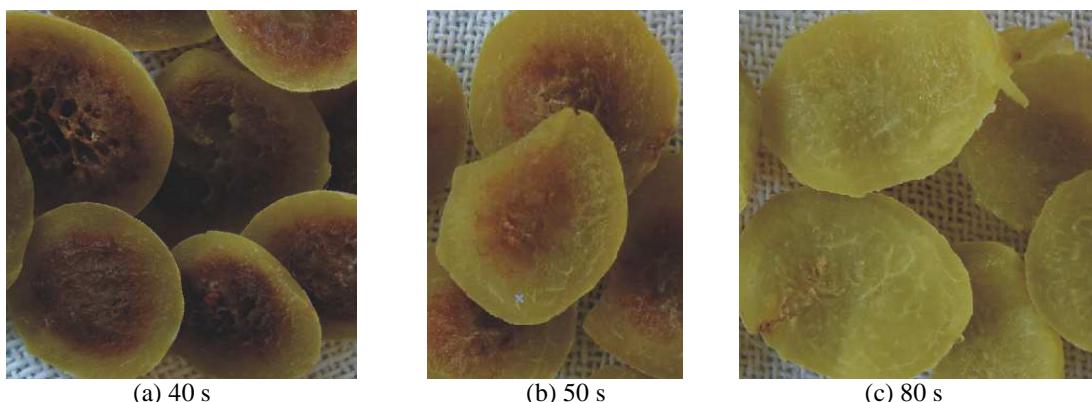
Foram pesados 10 g de polpa micro reestruturada no interior de frascos de vidro, seguido de enchimento a quente com 200 mL de néctar clarificado com 0,5% de goma gelana. Este produto foi deixado por 3 dias para avaliação visual com relação à separação das fases.

### **3. RESULTADOS E DISCUSSÃO**

#### **3.1. Preparo da polpa**

O pH original da fruta foi de  $2,69 \pm 0,2$ . O teor de sólidos solúveis totais da fruta variou de 9,5 a 10 °Brix. A acidez total titulável variou de 2,0 a 3,8 g/ 100g de fruta, expresso em ácido cítrico. A polpa final apresentou um teor médio de sólidos solúveis totais de 9,0 °Brix, acidez titulável de 2,2 g/ 100g e “ratio” de 4,09.

No branqueamento dos frutos, o tempo mínimo de imersão dos frutos em água em ebulação foi de 80 segundos. Tempos inferiores mostraram a inativação somente da parte externa dos frutos, como mostrado na Figura 1.



**Figura 1** – Branqueamento de frutos inteiros, por imersão em água em ebulação.

A partir de 100 segundos de branqueamento térmico dos frutos inteiros, observou-se o amolecimento da polpa, que aumentou o rendimento do despolpamento. No entanto, a partir de 120 segundos de imersão em água em ebulação, ocorreu a perda da consistência e rompimento da casca, com consequente perda de sólidos por escorrimento.

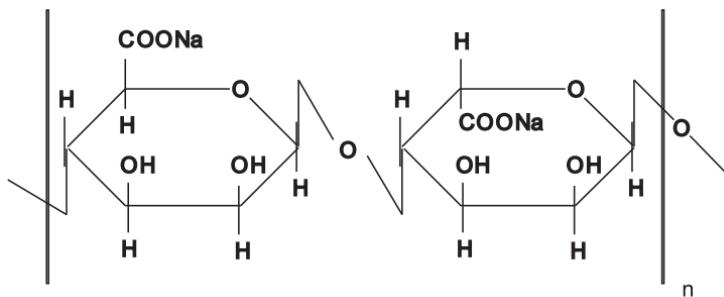
O rendimento do despolpamento aumentou com o aumento do tempo de branqueamento, devido ao amolecimento da polpa e do aumento da velocidade de rotação das escovas do despolpador.

### 3.2. Clarificação e obtenção das partículas micro estruturadas de polpa de umê

Na clarificação do suco de umê, não foi observada diferença significativa no rendimento de suco clarificado, que correspondeu a 85-88% do volume inicial de polpa. Isto ocorreu provavelmente à elevada aceleração centrífuga à qual as amostras foram submetidas.

Não foi observada a formação de partículas sólidas na solução de cloreto de cálcio nas amostras com pH 2,7 e 3,0. Partículas formadas com pH 3,5; 4,0 e 4,5 apresentaram estabilidade física somente a partir do tempo de 5 minutos após a imersão na solução de cloreto de cálcio. O íon bivalente presente em solução difunde-se para o interior da partícula e estabiliza a cadeia polimérica de alginato, constituída por várias unidades de sais de ácidos  $\beta$ -D-manurônico e  $\alpha$ -L-gulurônico, unidas por ligações glicosídicas, como

mostrada na Figura 2. O íon  $\text{Ca}^{2+}$  promove a ligação entre as cadeias lineares, formando uma estrutura tridimensional gelatinosa insolúvel na forma de esferas (Garcia-Cruz, Foggetti & Silva, 2008; Souza, Peralta-Zamora & Zawadzki, 2008).



**Figura 2** – Estrutura do alginato de sódio (Souza, Peralta-Zamora & Zawadzki, 2008).

Em valores de pH inferiores a 3,0 o ácido algínico assume a forma iônica, não permitindo a ligação com o íon cálcio, o que impede a estabilização da cadeia e formação de gel. Por isso, a polpa com alginato de sódio, sem modificação do pH não resultou na formação de micro partículas esféricas.

No experimento com a adição de 0,5% alginato de sódio à polpa de umê, temos 469 mg de alginato ( $\text{PM} = 349 \text{ g/mol}$ ) para cada 100 g de polpa e se todas as ligações iônicas da molécula de alginato forem ligadas ao íon cálcio, forneceria aproximadamente 96,5 mg de cálcio por 100 g de micro partícula. A adição de 5 g de polpa micro reestruturada para cada 100 mL de suco, fornece 4,8 mg de cálcio, o que é insuficiente para o “claim” fonte de cálcio, onde 100 mL de líquido deve fornecer no mínimo 7,5% da IDR.

Dentro da faixa de trabalho utilizada, a concentração de cloreto de cálcio não influenciou na textura das partículas. Porém, em escala industrial o teor de íons cálcio na solução deverá ser monitorado e controlado, para garantir a estabilidade das partículas formadas. Para cada 100 L de suco clarificado com micro partículas em suspensão, são necessários 5 kg de micro partículas formadas, onde são consumidos aproximadamente 1,3 L de solução de cloreto de cálcio 1%.

A altura de alimentação da mistura polposa exerceu importante influência sobre o formato da partícula formada. Distâncias entre a alimentação da mistura polposa e a superfície da solução de cloreto de cálcio superiores a 2 cm resultaram no achatamento da partícula devido ao aumento da velocidade e impacto com a solução. Alturas inferiores a 2 cm não apresentaram diferença significativa.

Com o aumento do pH pela adição do citrato de sódio, o sabor da polpa alterou-se significativamente, diminuindo a sensação de amargor e acidez e ficando salgado. Com a tendência de redução do teor de sódio nos alimentos, aliado à alteração do sabor original da fruta, é recomendável a mínima elevação do pH, neste caso até 3,5, que permita o processamento desejado.

O aumento da concentração de alginato à polpa de umê resultou em micro partículas mais consistentes, mas notou-se sensorialmente uma consistência estranha e alteração do sabor.

Todas as micro esferas formadas e imersas por pelo menos 5 minutos em solução de pelo menos 1% cloreto de cálcio apresentaram estabilidade em suco em repouso por 24 horas à temperatura ambiente em pH de no mínimo 3,5, bem como após o aquecimento até 80 °C por 5 minutos, em repouso.

Portanto, o melhor preparo da polpa foi a mistura com 0,5% de alginato de sódio e ajuste do pH para 3,5. Esta mistura gotejada a 2 cm de altura em solução de cloreto de cálcio 1,0% forneceu partículas esféricas de aproximadamente 3 mm de diâmetro, estáveis em pH 3,5 por 24 horas e a 80 °C por 5 minutos, sem agitação.

Com a adição de 0,5% de goma gelana ao suco clarificado, foram necessários 2 dias para se observar a decantação da polpa micro reestruturada. No entanto, na elaboração de néctares sem adição de açúcar, a diferença de massa específica deve aumentar, necessitando utilizar maior concentração de goma, para aumento da viscosidade da fração líquida.

## **4. CONCLUSÕES**

A mistura de polpa de umê com 0,5% de alginato de sódio teve que ter o seu pH aumentado para 3,5 de modo a formar partículas esféricas estáveis, após permanecer um tempo mínimo de 5 minutos em solução resfriada de cloreto de cálcio 1%. As partículas formadas não se dissolveram após 24 horas em suco clarificado com pH 3,5. Além disso, mostraram-se estáveis após o aquecimento a 80 °C por 5 minutos. A adição de diferentes teores de partículas de umê com alginato ao suco clarificado de umê pode fornecer um produto completamente novo, com forte apelo de saudável, além de muito prazeroso.

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## **Conclusão geral**

Os frutos de umê na Região Sudeste do Brasil apresentam menores dimensões e massa (6 a 16 g), quando comparado com valores relatados em literatura dos países asiáticos (15 a 30 g), grandes consumidores e apreciadores destes frutos. A relação polpa:caroço também se mostrou inferior nos frutos coletados no Estado de São Paulo. Observou-se que a casca apresenta maior teor de compostos fenólicos, comparado com a polpa. No entanto, durante o despolpamento, grande parte da casca é misturada à polpa, o que ajuda a conferir sabor amargo e propriedades nutracêuticas, como o combate aos radicais livres no organismo ou em produtos processados.

O teor de compostos fenólicos e a atividade antioxidante dos frutos não apresentou variação significativa durante a maturação dos frutos, a partir dos frutos fisiologicamente maduros. Dessa forma, é possível vislumbrar a possibilidade de produtos processados a partir de frutos maduros apresentarem os mesmos benefícios relacionados ao consumo de alimentos produzidos a partir de frutos verdes de umê, amplamente divulgados na literatura científica.

O comportamento reológico da polpa de umê mostrou que a consistência do produto é fortemente influenciada pela concentração da polpa, sendo não dependente do tempo e pseudoplástico (Herschel-Bulkley). Os modelos de Herschel-Bulkley, Casson, Bingham e Lei da Potência forneceram boa correlação de valores. A equação de Arrhenius permitiu o cálculo da energia de ativação para diferentes concentrações de polpa de umê. Estes resultados são importantes no dimensionamento de tubulações industriais, equipamentos e parâmetros de processo.

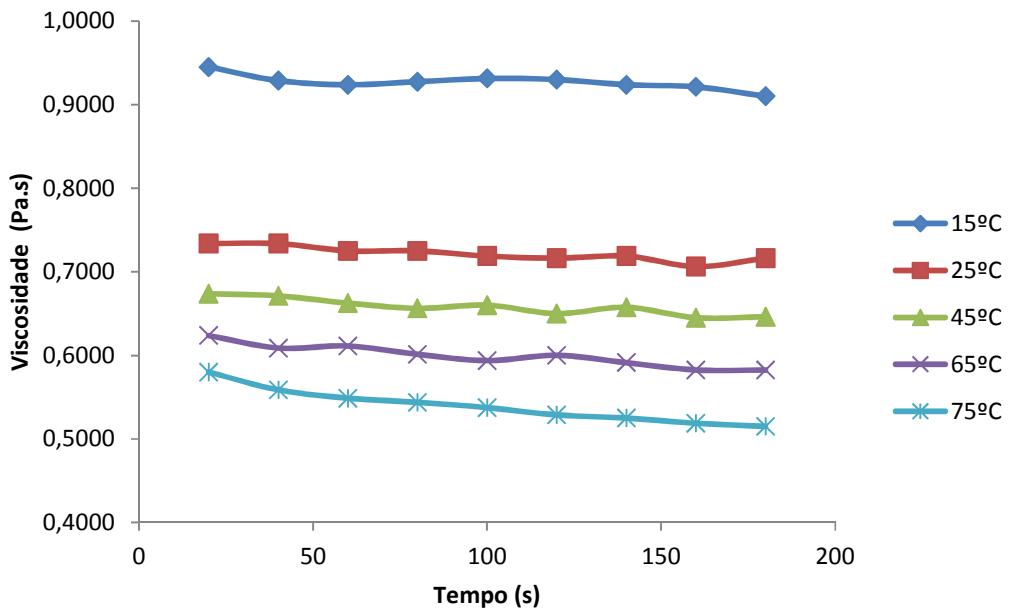
Para a micro reestruturação da polpa de umê, o pH teve que ser elevado para 3,5 e foram necessários 0,5% de alginato de sódio e a imersão em solução resfriada de cloreto de cálcio 1% por pelo menos 5 minutos. Estas partículas formadas mostraram-se estáveis ao aquecimento e podem ser adicionadas ao suco de umê, para a elaboração de um produto inovador de grande aceitação e apelo de saudável.

## **Sugestões para trabalhos futuros:**

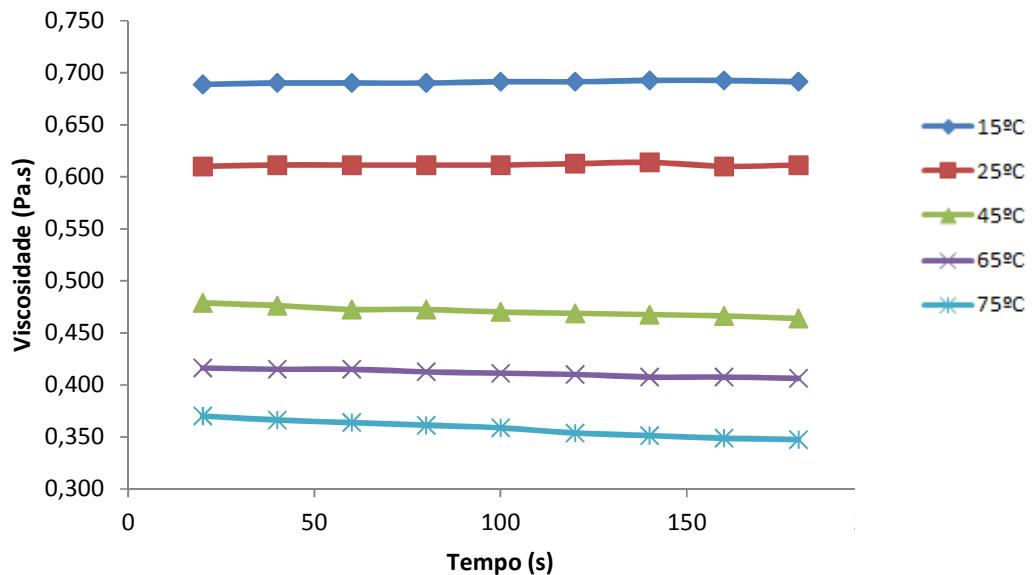
- 1) Realizar análise sensorial do néctar de umê integral. Verificar diferentes diluições, teores de açúcar e viscosidade. Avaliar a aceitação do néctar de umê em paralelo à aceitação de néctar comercial de pêssego.  
Identificar com uma sequência aleatória de três números e a ordem de apresentação das amostras para diferentes provadores;  
Realizar análises afetivas utilizando escala hedônica estruturada de 9 pontos, onde: 9) Gostei muitíssimo; 8) Gostei muito; 7) Gostei moderadamente; 6) Gostei ligeiramente; 5) Não gostei/ nem desgostei; 4) Desgostei ligeiramente; 3) Desgostei moderadamente; 2) Desgostei muito; 1) Desgostei muitíssimo.  
Analizar os resultados pelo teste das médias de Tukey para  $p<0,05$  utilizando o software Statistica, versão 5.5 (Statsoft ®).
- 2) Realizar análise sensorial de *blends* néctar de umê integral e néctar de pêssego comercial. Verificar diferentes teores de mistura, para avaliação da diferença e aceitação.  
Teste triangular, seguido de preferência da amostra diferente.
- 3) Realizar análise sensorial do néctar de umê clarificado com adição de partículas de polpa em suspensão. Verificar o teor ideal de partículas em suspensão, bem como o tamanho ideal. Avaliar a aceitação do néctar clarificado com partículas em suspensão e comparar com a aceitação do néctar comercial de pêssego, realizado em paralelo.

## Anexos

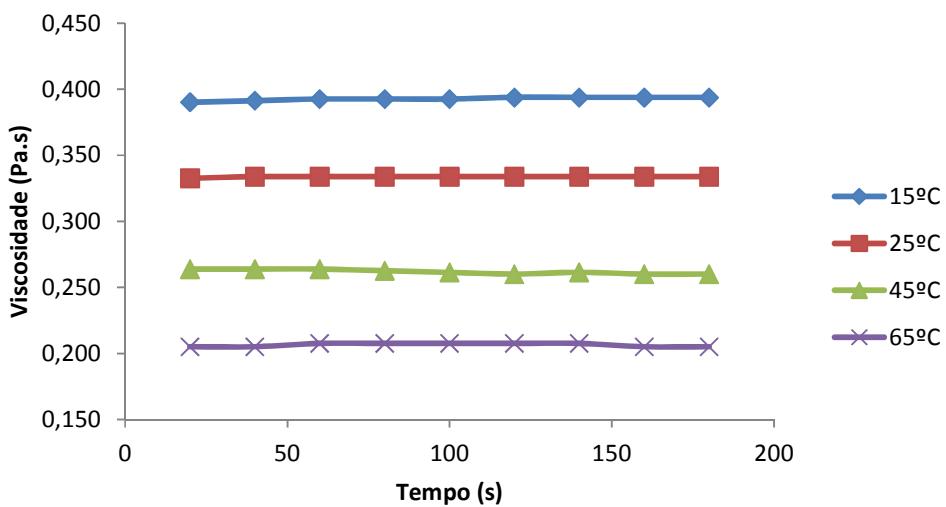
### Anexo A.1: Gráficos de reología



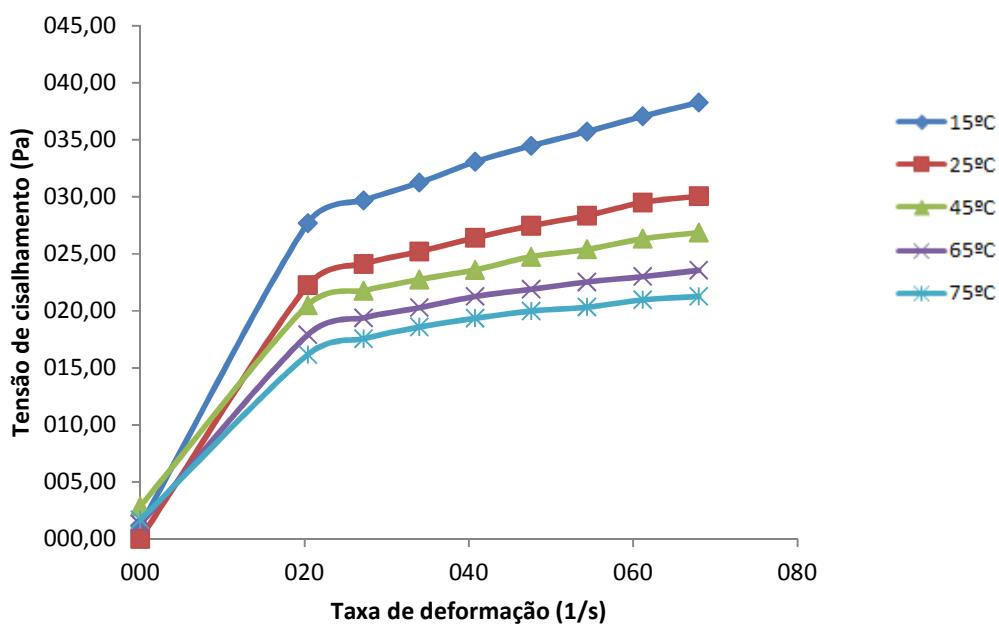
**Figura A.1** – Viscosidade aparente versus tempo para temperaturas de 15 a 75°C, a 8 °Brix



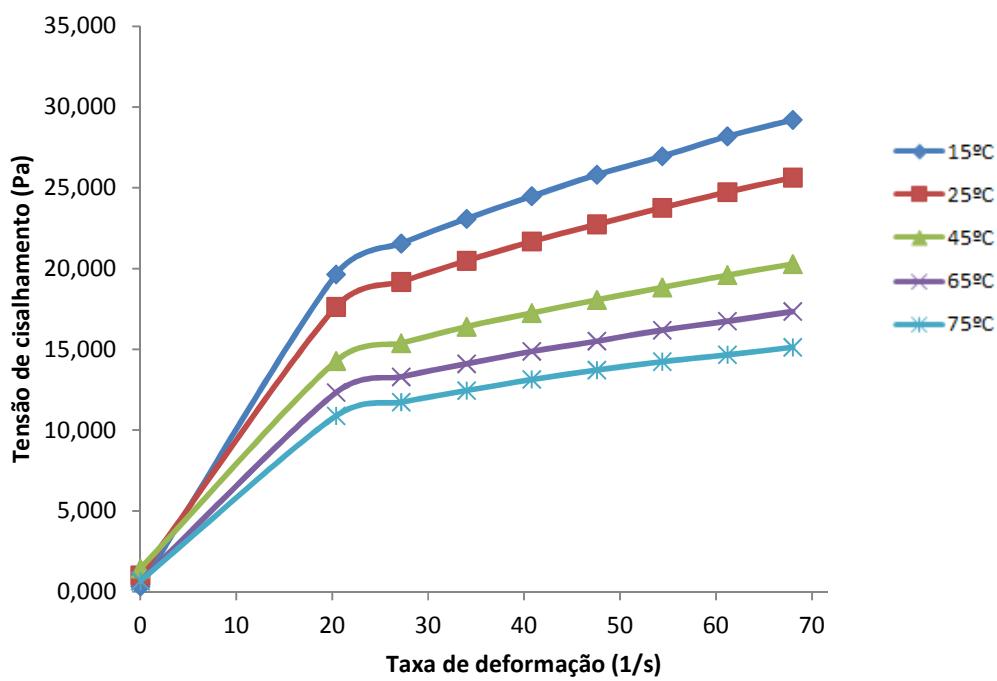
**Figura A.2** – Viscosidade aparente versus tempo para temperaturas de 15 a 75°C, a 7 °Brix



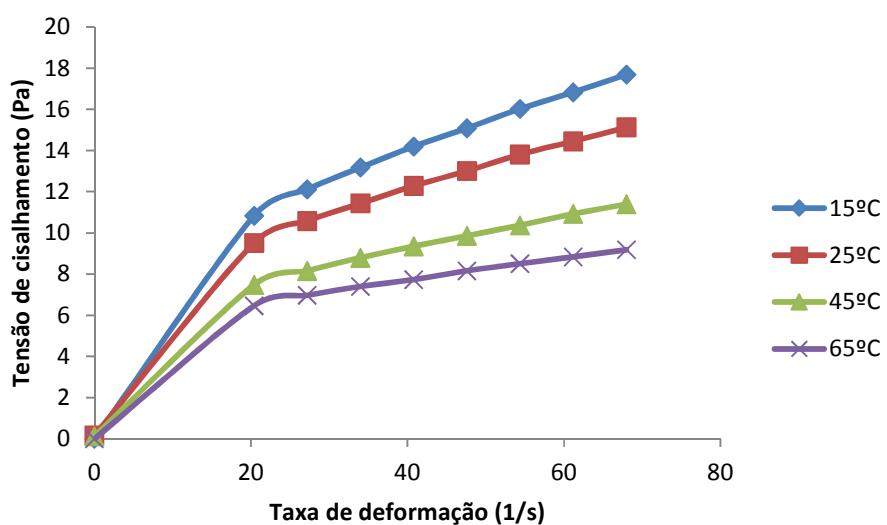
**Figura A.3** – Viscosidade aparente versus tempo para temperaturas de 15 a 65°C, a 6 °Brix



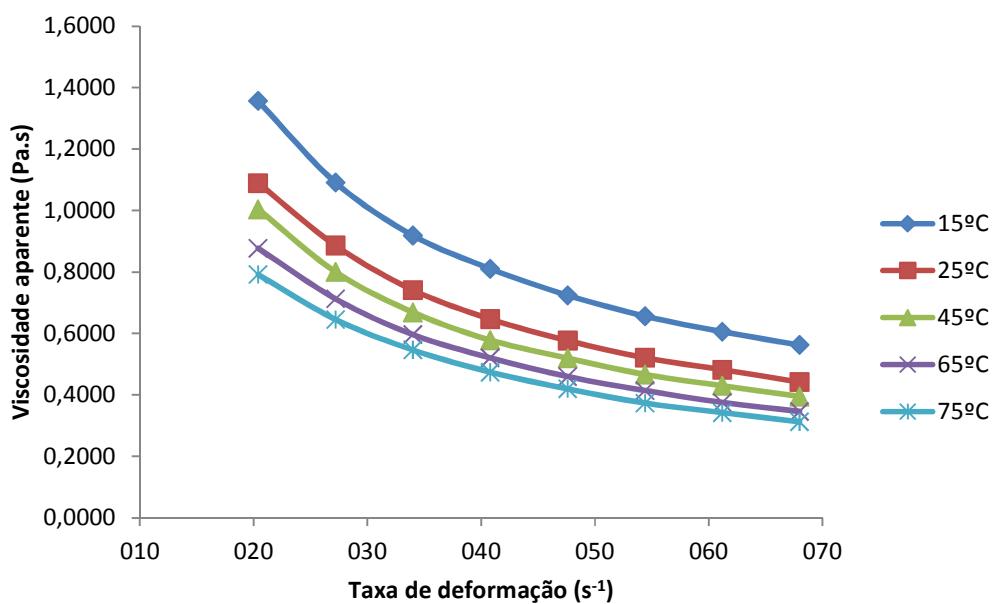
**Figura A.4** – Tensão de cisalhamento versus taxa de deformação para temperaturas de 15 a 75°C, a 8 °Brix



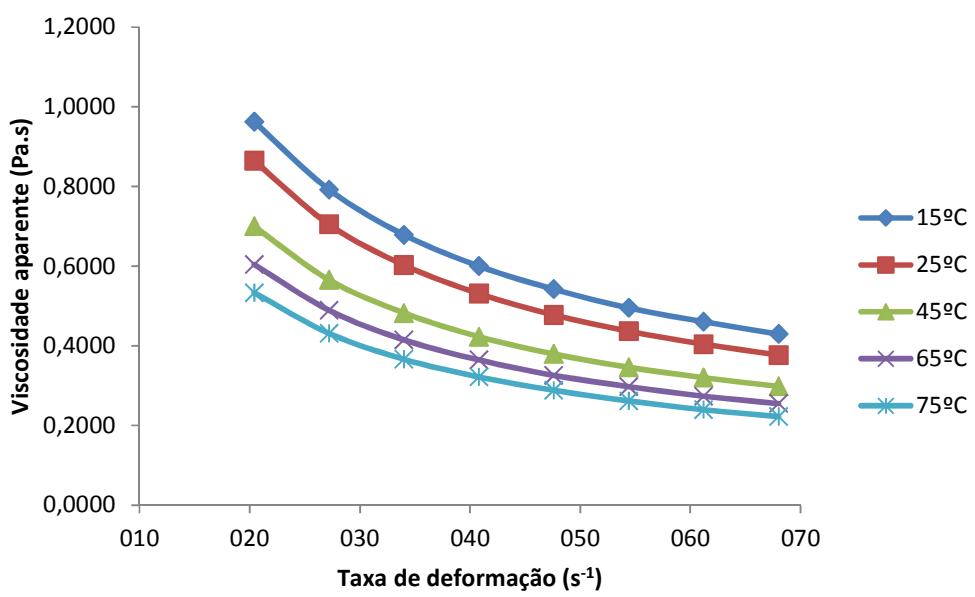
**Figura A.5** – Tensão de cisalhamento versus taxa de deformação para temperaturas de 15 a 75°C, a 7 °Brix



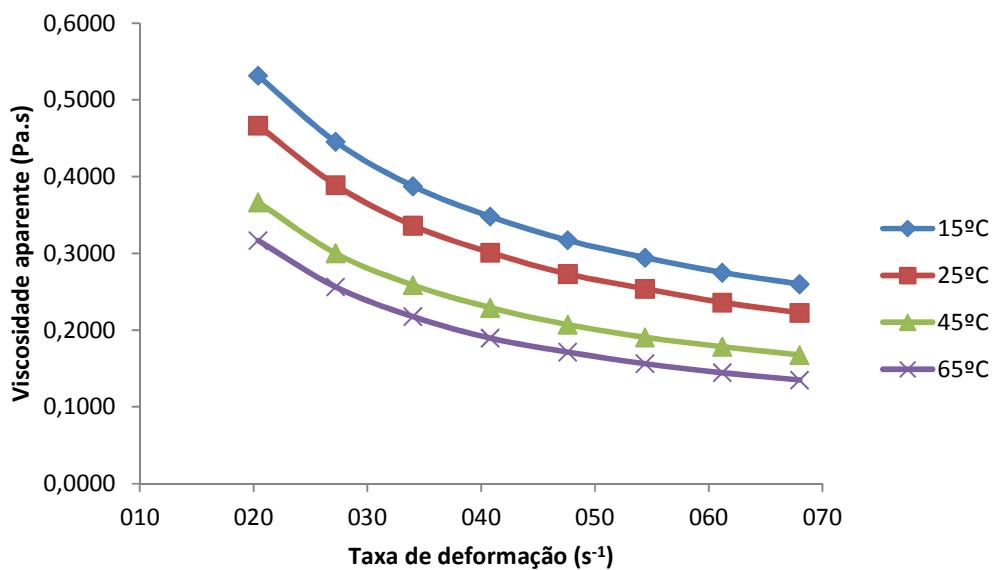
**Figura A.6** – Tensão de cisalhamento versus taxa de deformação para temperaturas de 15 a 65°C, a 6 °Brix



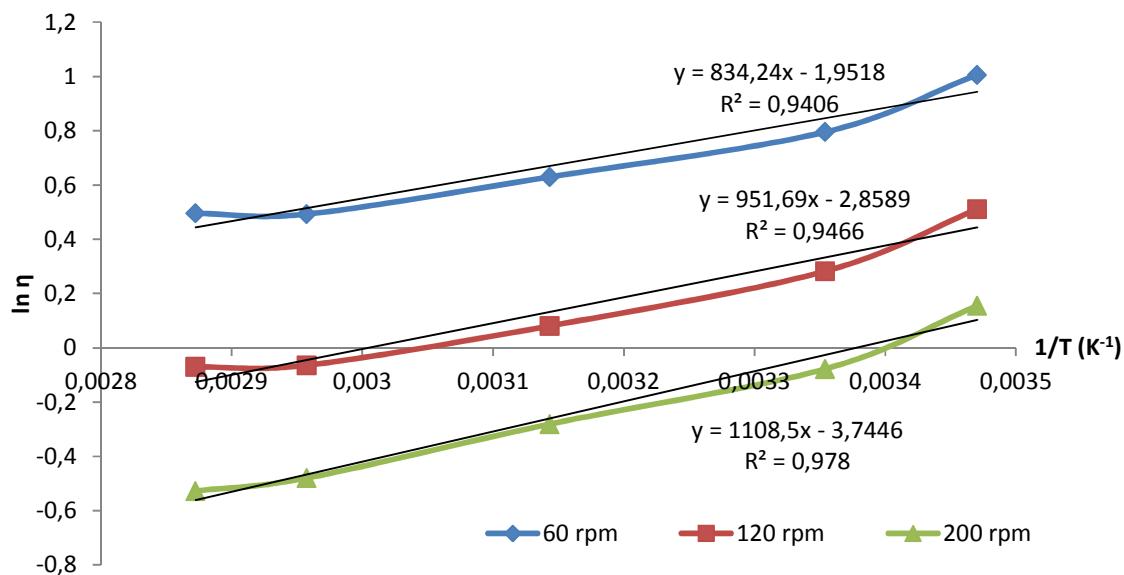
**Figura A.7** – Viscosidade aparente versus taxa de deformação para temperaturas de 15 a 75°C e 8°Brix



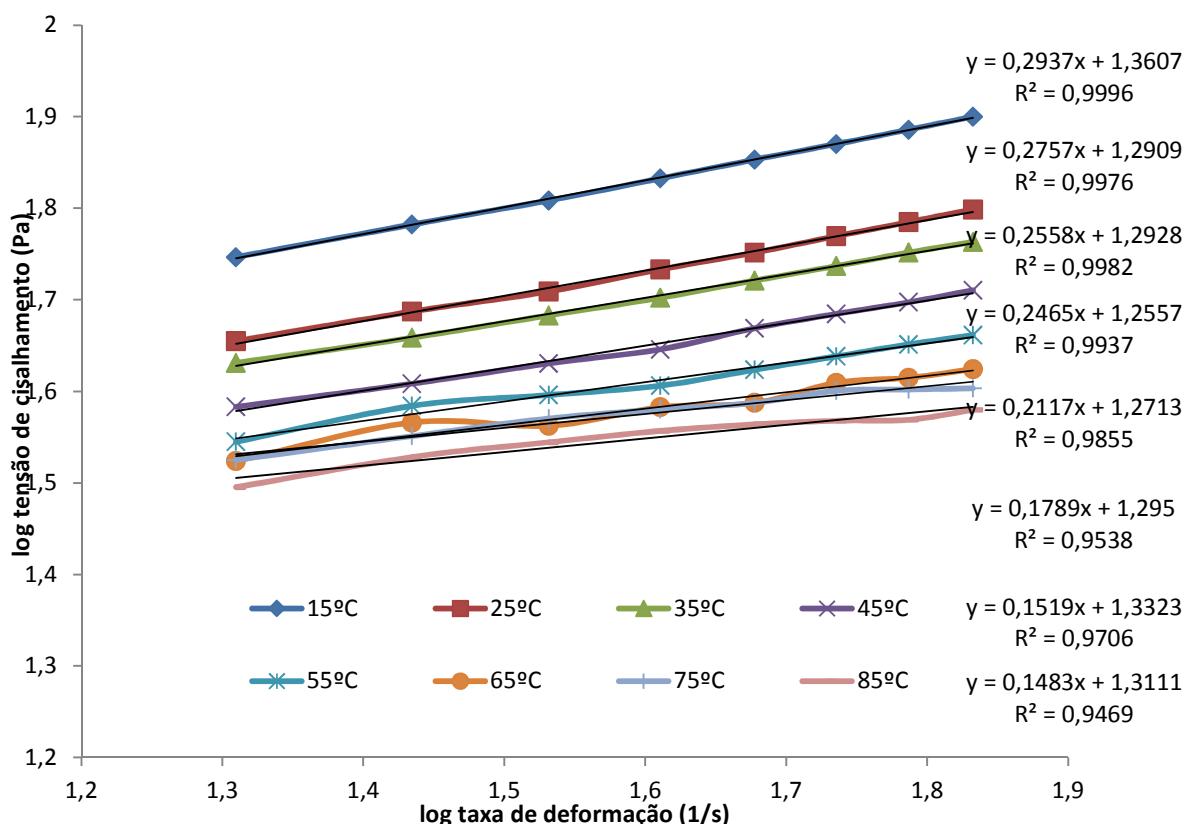
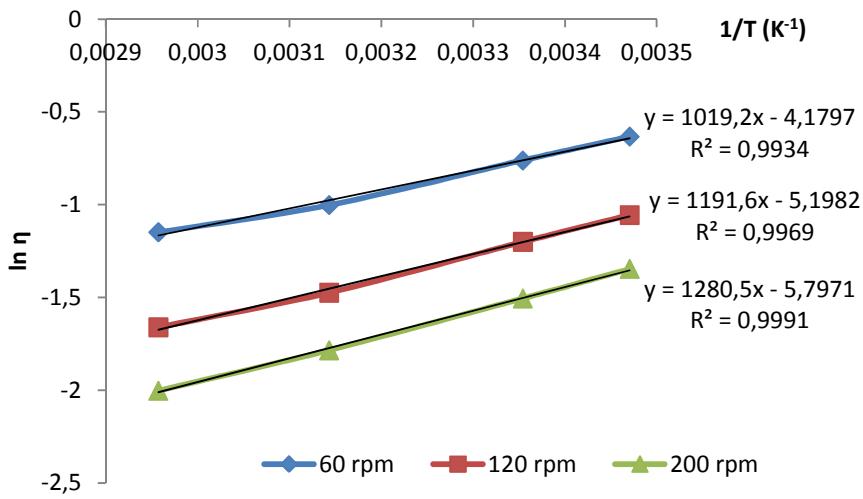
**Figura A.8** – Viscosidade aparente versus taxa de deformação para temperaturas de 15 a 75°C e 7°Brix

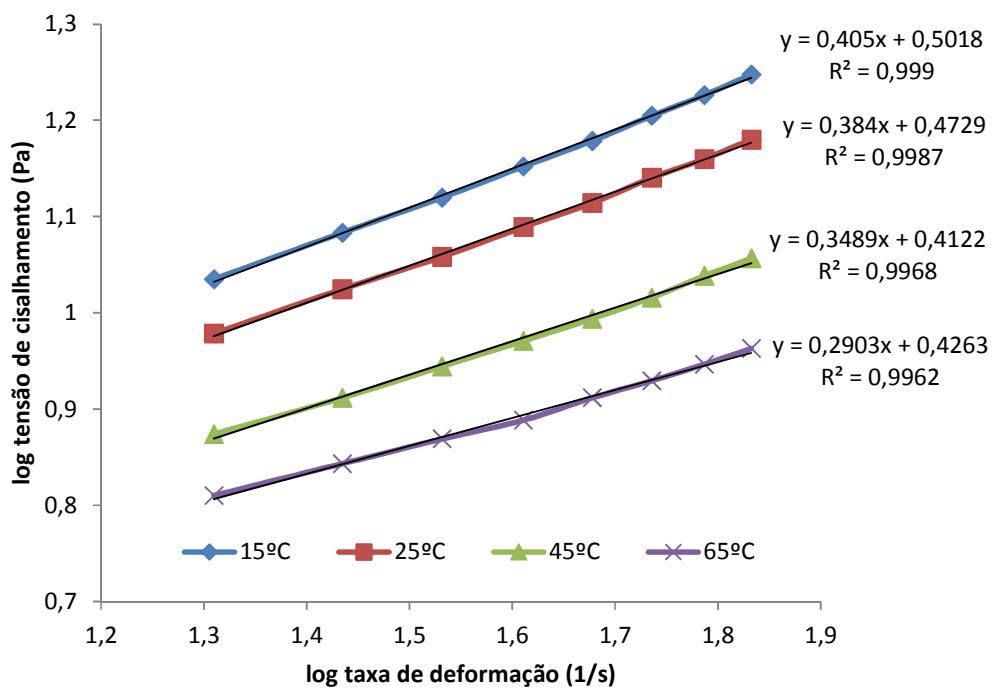


**Figura A.9** – Viscosidade aparente versus taxa de deformação para temperaturas de 15 a 75°C e 6°Brix



**Figura A.10** – Linearização da Equação de Arrhenius, para polpa de umê com 9°Brix





**Figura A.13** – Linearização da Equação de Ostwald-Waele, para polpa de umê com 6°Brix

**Anexo A.2:** Observations about Chapter 3: *Prunus mume* – fruit characteristics and phenolic content capacity

Figure 3: Whole and cut fruit collected in São Paulo, Brazil.

Mume fruits were collected in Valinhos, SP, Brazil, Latitude 22°59'28"S; Longitude 47°02'38"W; Elevation 640 m; Koeppen classification Cwa.

Fruits had their color evaluated every day after harvest. On the 6<sup>th</sup> day, fruits presented yellow color and characteristic odor of ripe mume fruits, similar to peach with floral notes. After the 7<sup>th</sup> day the fruits showed advanced signs of senescence.

Total phenol content of the ethanol extracts was determined with Folin-Ciocalteu colorimetric method (SINGLETON and ROSSI, 1965) with some modifications. Briefly, 0.1 ml extract was diluted with 8.4 ml distilled water and mixed with 0.5 ml Folin-Ciocalteu reagent. The contents were mixed by manual shaking for 15-20 s.

After 3 min, 1.0 ml of 20% sodium carbonate solution was added. The reaction mixture was incubated at room temperature for 1 h and its absorbance was measured at 720 nm using a dual beam UV-Vis spectrophotometer (Shimadzu, Mod. UV-Mini 1240). Catechin (SIGMA, C-1251) was used as an analytical standard for total phenol quantification and it was expressed as milligrams of catechin equivalents (CE) per gram wet basis (g-WB) of fruit.

TPC analyses were performed in triplicate.

Pulping was performed in pilot-scale brush pulper with 2.2 mm orifice apertures. Vacuum was applied in 200g of pulp poured into a deep plate with 20x20 cm and submitted to 0.7 bar for 60 seconds. Then 150g of deaerated pulp was weight inside co-extruded polyethylene and polyamide flexible bags of 20x20 cm submitted to -0.7 bar for 20 seconds and hot sealed.

Statistical comparison of means were performed using Tukey ( $p<0.05$ ).