UNIVERSIDADE DE SÃO PAULO
FACULDADE DE CIÊNCIAS FARMACÊUTICAS
Programa de Pós-graduação em Ciência dos Alimentos
Área de Bromatologia

Obtenção de extratos secos de cagaita (*Eugenia dysenterica* DC) pelo método de atomização: avaliação das propriedades físicas e potencial funcional *in vitro*

Luis Daniel Daza Ramírez

Dissertação para obtenção do grau de
MESTRE

Orientadora:
Prof. Dr. Maria Inés Genovese

São Paulo
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Luis Daniel Daza Ramírez

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da
Dissertação para obtenção do grau de Mestrado

Prof. Dr. Maria Inés Genovese
Orientador / Presidente

1º examinador

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Dedico este trabajo a mis grandes amigos Elizabeth Murillo Perea, Pilar Valencia y Diego Leandro Castaño, por su constante apoyo y su incondicional cariño.

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RESUMO

DAZA, L. D. **Obtenção de extratos secos de cagaita (*Eugenia dysenterica* DC) pelo método de atomização: avaliação das propriedades físicas e potencial funcional *in vitro*.** 2015. 87f. (Dissertação de mestrado) – Faculdade de Ciências Farmacêuticas, Universidade de São Paulo, São Paulo, 2015.

As frutas nativas brasileiras têm grande potencial agroindustrial e farmacêutico por serem excelentes fontes de compostos bioativos fenólicos. A cagaita (*Eugenia dysenterica* DC.), típica do Cerrado, se destaca por conter compostos fenólicos com alta capacidade de inibir as enzimas envolvidas no metabolismo de carboidratos *in vitro*. Esta ação também ocorre com o consumo de suco, que causa redução da hiperglicemia pós-prandial em seres humanos, tanto saudáveis como resistentes a insulina. No entanto, devido à alta perecibilidade dos frutos, o armazenamento, transporte e ampla distribuição destes torna-se fator limitante para sua incorporação na dieta. Dessa forma, a aplicação de processos que garantam a conservação dos componentes bioativos e facilitem o transporte e o armazenamento do material pode tornar o fruto mais atrativo para seu uso industrial. Este trabalho teve como objetivos a obtenção de extratos secos a partir do fruto da cagaita através da secagem por atomização, comparando a utilização de goma arábica e inulina como agentes carreadores e avaliando o efeito dos parâmetros de processo sobre as propriedades físico-químicas e estabilidade dos compostos fenólicos nos pós produzidos, assim como seu potencial funcional *in vitro*. A secagem do extrato bruto foi realizada variando-se a temperatura do ar de entrada (120, 140 e 160°C) e o tipo e a concentração dos carreadores (goma arábica – GA ou inulina – I; 10, 20 ou 30%). Os melhores rendimentos de processo foram os dos pós obtidos usando goma arábica como agente carregador (41% a 81%). Todas as amostras apresentaram menores valores de umidade e atividade de água quando comparadas com a amostra utilizada como controle (amostra liofilizada). O processo de atomização incrementou a solubilidade e reduziu a higroscopicidade das amostras. No caso das amostras obtidas usando GA como carreador, a temperatura de transição vítrea foi principalmente influenciada pela temperatura de ar de entrada. A concentração de carreador foi o parâmetro que mais influenciou a temperatura de transição vítrea das amostras com inulina. A morfologia das partículas foi influenciada por ambos os parâmetros, temperatura do ar de entrada e concentração do carreador. Os valores de retenção de fenólicos totais dos pós com goma arábica variaram de 88% a 99%, e para os pós obtidos com inulina, de 78% a 98%. A retenção das proantocianidinas variou de 72% a 98%, para os pós obtidos com goma arábica, e de 63% a 98%, para os pós obtidos com inulina. Altos coeficientes de correlação de Pearson, entre o conteúdo de fenólicos totais e a capacidade antioxidante das amostras contendo GA e inulina, foram obtidos de 0,90 a 0,99 ($p < 0,05$). A GA foi o material mais adequado para estabilizar os extratos e evitar a perda de compostos fenólicos ao longo do armazenamento. Comparadas com a acarbose ($CI_{50} = 0,013$ mg/mL), as amostras apresentaram uma baixa inibição da enzima α -amilase, sendo que a concentração média inibitória (CI_{50}) dos extratos secos variou entre 11 a 107 mg/mL e entre 10 a 100 mg/mL, para os pós obtidos usando GA e inulina, respectivamente. Todas as amostras mostraram inibição da enzima α -glicosidase (6 mg/mL a 159 mg/mL e 13 mg/mL a 154 mg/mL, para os pós obtidos usando GA e inulina, respectivamente), mas a inibição observada foi média a baixa em comparação com a atividade do controle acarbose ($CI_{50} = 0,11$ mg/mL). No entanto, a atividade dos fenólicos dos pós comparada com a acarbose foi boa, 0,3 a 1,1 mg GAE/mL para α -amilase e de 0,3 a 1,7 mg GAE/mL para α -glicosidase. Os pós apresentaram atividade inibitória contra *Staphylococcus aureus* e *Listeria monocytogenes*, semelhante aos antibióticos utilizados como controle positivo. Os resultados obtidos sugerem que os produtos do processo de secagem por atomização utilizando goma arábica apresentam melhores propriedades físicas que os pós obtidos utilizando inulina, e que o uso de secagem por atomização para obtenção de extratos secos de cagaita pode se tornar uma alternativa viável para reduzir os custos de transporte e evitar a deterioração dos compostos bioativos que estão relacionados com algumas propriedades benéficas para a saúde.

Palavras-chave: cagaita, compostos fenólicos, atomização, atividade antioxidante.

ABSTRACT

DAZA, L. D. **Spray-dried extracts from cagaita (*Eugenia dysenterica* DC): evaluation of physical properties and functional potential *in vitro***. 2015. 87f. (Dissertação de mestrado) – Faculdade de Ciências Farmacêuticas, Universidade de São Paulo, São Paulo, 2015.

Brazilian native fruits have great agroindustrial and pharmaceutical potential for being excellent sources of phenolic bioactive compounds. The cagaita (*Eugenia dysenterica* DC.), fruit typical of the Cerrado, stands out for containing phenolic compounds with high ability to inhibit enzymes involved in carbohydrate metabolism *in vitro*. This action also occurs with the consumption of juice, which causes reduction in postprandial hyperglycemia in humans, both healthy and insulin resistant. However, due to the high perishability of the fruit, storage, transportation and broad distribution becomes a limiting factor for its incorporation in the diet. Thus, the application of processes that ensure the preservation of bioactive components and facilitate the transport and storage of the material can make the fruit more attractive for industrial use. This study aimed to obtaining dry extracts from the cagaita fruit by spray drying, comparing the use of gum arabic and inulin as carrier agents and evaluating the effect of process parameters on the physicochemical properties and stability of phenolic compounds produced in the dry extracts, as well as their functional potential *in vitro*. The drying of the crude extract was performed by varying the inlet air temperature (120, 140 and 160 °C) and the type and concentration of carrier agent (gum arabic - GA or inulin - I; 10, 20 or 30%). The best process yields were obtained using gum Arabic as the carrier agent (41% to 81%). All samples showed lower humidity values and water activity when compared with the lyophilized sample used as control. The spray drying process increased solubility and reduced hygroscopicity of the samples. In the case of the samples obtained using GA as a carrier, the glass transition temperature was mainly influenced by the inlet air temperature. The carrier concentration was the parameter that most influenced the glass transition temperature of the samples dried with inulin. The morphology of the particles was influenced by both inlet air temperature and concentration of the carrier. The retention values of total phenolics in gum arabic powders ranged from 88 to 99%, and the powders obtained with inulin, from 78 to 98%. The retention of proanthocyanidins ranged from 72 to 98% for powders obtained with gum arabic, and 63 to 98% for powders obtained with inulin. High Pearson correlation coefficients between the total phenolic content and antioxidant capacity of the samples containing GA and inulin were obtained from 0.90 to 0.99 ($p < 0.05$). GA was the most suitable material to stabilize the extracts and prevent the loss of phenolic compounds during storage. Compared with acarbose (IC₅₀ = 0.013 mg/mL), samples showed a low inhibition of α -amylase, the half maximal inhibitory concentrations (IC₅₀) of the dried extract ranged from 11 to 107 mg/mL and 10 to 100 mg/mL, for the powders obtained by using Ga and inulin, respectively. All samples showed inhibition of α -glucosidase (6 mg/mL to 159 mg/mL and 13 mg/mL to 154 mg/mL, for the powders produced using inulin and GA, respectively), but the inhibition was lower compared to the activity of acarbose used as a control (IC₅₀ = 0.11 mg/mL). However, the activity of the phenolic compounds of the powders, when compared with acarbose was good 0.3 to 1.1 mg GAE/mL for α -amylase and 0.3 to 1.7 mg GAE/mL for α -glucosidase. The powders showed inhibitory activity against *Staphylococcus aureus* and *Listeria monocytogenes*, similar to antibiotics used as positive control. The results suggest that the products of the spray drying process using gum arabic have better physical properties than powders produced using inulin, and the use of spray drying to obtain dry extracts of cagaita can become a viable alternative to reduce transportation costs and avoid the deterioration of bioactive compounds that are related to properties beneficial to health.

Key-words: cagaita, phenolic compounds, spray drying, antioxidant capacity.

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Lista de abreviaturas, siglas e símbolos

%	Porcentagem / <i>Percentage</i>
µg	Micrograma / <i>Microgram</i>
µL	Microlitros / <i>Microliter</i>
°C	Graus Celsius / <i>Celsius degree</i>
µm	Micrometros / <i>Micrometers</i>
a*	Parâmetro de cor, diferença do vermelho e do verde / <i>redness</i>
AUC	Área sob a curva / <i>Area under curve</i>
a_w	Atividade de água / <i>water activity</i>
b*	Parâmetro de cor, diferença do azul e do amarelo / <i>greenness</i>
b.s	Base seca
C	Concentração de carreador / <i>Carrier concentration</i>
CI₅₀	Concentração inibitória média
Da	Dalton
DAD	Detector de arranjo de diodos / <i>diode array detector</i>
DPPH	Radical (1,1-difenil-2-picril-hidrazila)
DSC	Varredura diferencial de calorimetria / <i>Differential scanning calorimetry</i>
DW	<i>Dry weight</i>
EAG	Equivalente de ácido gálico
EC	Equivalente de catequina
ETQ	Equivalente de tanino quebracho / <i>Tannin quebracho equivalent</i>
FRAP	<i>Ferric reducing antioxidant power</i>
g	Constante gravitacional
g	Gramas / <i>Gram</i>
GA	Goma arábica / <i>gum arabic</i>
GAE	<i>Galic acid equivalent</i>
H	Higroscopicidade / <i>Higroscopicity</i>
HPLC	Cromatografia líquida de alta eficiência / <i>High-performance liquid chromatography</i>
I	Inulina / <i>Inulin</i>
IC₅₀	<i>Half maximal inhibitory concentration</i>
k	Constante da velocidade da reação de degradação de fenólicos
kg	Quilogramas / <i>Kilogram</i>
KHz	Quilohertz

L	Litros / <i>Liters</i>
L*	Parâmetro de cor, luminosidade / <i>Lightness</i>
m	Metros / <i>Meters</i>
M	Umidade / <i>moisture</i>
m³	Metro cúbico
mg	Miligramas / <i>Miligram</i>
MIC	Concentração mínima inibitória / <i>Minimum inhibitory concentration</i>
min	Minutos / <i>Minutes</i>
mL	Mililitros / <i>Mililiters</i>
mm	Milímetros / <i>Milimeters</i>
mM	Milimolar
nm	Nanômetros / <i>Nanometers</i>
ORAC	<i>Oxygen Radical Absorbance Capacity Assay</i>
pH	Potencial hidrogeniônico / <i>Hydrogen potential</i>
RH	Umidade relativa / <i>relative humidity</i>
rpm	Rotações por minuto / <i>Revolutions per Minute</i>
S	Solubilidade / <i>Solubility</i>
SS	Sólidos solúveis / <i>soluble solids</i>
T	Temperatura / <i>Temperature</i>
t	Tempo / <i>Time</i>
t_{1/2}	Tempo de meia vida dos compostos fenólicos durante a estocagem
Tg	Temperatura de transição vítrea / <i>glass transition temperature</i>
TPC	Compostos fenólicos totais / <i>Total phenolic content</i>
TS	Açúcares totais / <i>Total sugars</i>
UFC	Unidades formadoras de colônias / <i>Colony-forming unit</i>
v/v	Proporção volume / <i>volume</i>
w/v	Proporção peso/volume / <i>weigh/volume</i>
Y	Rendimento / <i>Yield</i>

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1. INTRODUÇÃO

Dietas ricas em frutos e vegetais podem reduzir o risco de doenças cardiovasculares, diabetes e câncer (CROZIER, JAGANATH E CLIFFORD, 2009). Esse efeito protetor pode ser atribuído, em parte, aos compostos fenólicos presentes na matriz alimentícia, capazes de proteger as células do dano oxidativo (LAURA et al., 2014).

O Brasil é o país com a maior biodiversidade do mundo e é o terceiro maior produtor de frutos (FAO, 2013). Dentro desta produção de frutos têm-se as frutas nativas com grande potencial agroindustrial e farmacêutico por serem excelentes fontes de compostos bioativos, particularmente de compostos de natureza fenólica. Entre estes frutos destaca-se a cagaita (*Eugenia dysenterica* DC.), uma fruta do Cerrado utilizada na medicina popular para o tratamento de diabetes (MARTINOTTO et al., 2008). Estudos realizados pelo nosso grupo de pesquisa têm demonstrado que os compostos fenólicos presentes no fruto da cagaita possuem a capacidade de inibir as enzimas envolvidas no metabolismo de carboidratos *in vitro*, provavelmente relacionado à diminuição da hiperglicemia pós-prandial observada em seres humanos saudáveis e resistentes a insulina. No entanto, devido à alta perecibilidade dos frutos, o armazenamento e transporte destes torna-se uma limitante para seu uso. Dessa forma, a aplicação de processos na produção de extratos secos a partir de *E. dysenterica*, que garantam a conservação dos componentes bioativos e facilitem o transporte e o armazenamento do material, pode tornar o fruto mais atrativo para as indústrias farmacêutica e alimentícia.

O método de atomização é o mais comum na indústria, além de ser mais econômico. Neste método de secagem, o fluido a ser desidratado é introduzido por aspersão sob pressão e recebe uma corrente de ar quente sob temperaturas elevadas, formando, instantaneamente, partículas sólidas. Em função do curto tempo de residência do fluido na câmara de secagem e do uso de adjuvantes, o produto final reúne padrões elevados de qualidade em relação à granulometria, umidade final, homogeneidade, higroscopicidade, forma e fluidez, além de proteger os constituintes do produto inicial.

1.1 Compostos fenólicos

Estudos epidemiológicos têm mostrado uma associação positiva entre o consumo de frutos e vegetais e uma redução da incidência de doenças cardiovasculares, diabetes e câncer (JAGANATH e CROZIER, 2010). Esse efeito protetor é atribuído, em parte, aos compostos

fenólicos, já que uma ingestão de alimentos ricos nestes compostos é associada com uma diminuição de dislipidemia e aterosclerose, disfunção endotelial e hipertensão, ativação plaquetária e trombose, além de processos inflamatórios associados à indução das doenças cardiovasculares (FRAGA et al., 2010).

Os compostos fenólicos são metabólitos secundários das plantas que estão envolvidos em diversos processos como crescimento, lignificação, pigmentação, polinização e resistência contra patógenos, predadores e estresse ambiental (DUTHIE, GARDNER e KYLE, 2003). Quimicamente, os fenóis são compostos com um ou mais grupos hidroxila ligados a um anel de benzeno e são classificados em dois grupos, o primeiro que são os compostos não flavonoides como: ácidos fenólicos, acetofenonas, ácidos hidroxicinâmicos (cumárico, caféico, ferúlico, sinápico), coumarinas, estilbenos, lignanas e taninos, e o segundo grupo que são os compostos flavonoides (CROZIER, JAGANATH e CLIFFORD, 2009).

Os flavonoides são o maior grupo de compostos fenólicos, sua estrutura básica (C6-C3-C6) está conformada por dois anéis aromáticos ligados por um anel heterocíclico oxigenado. As principais classes de flavonoides presentes em alimentos são: flavonóis (quercetina, caempferol, miricetina), flavonas (luteolina, apigenina), isoflavonas (genisteína, daidzeína, gliciteína), flavanonas (naringenina, hesperetina), antocianidinas (cianidina, delphinidina) e flavanóis (catequina, epicatequina) (Figura 1) (CROZIER, JAGANATH e CLIFFORD, 2009). Os flavonoides têm sido associados com diversas atividades biológicas e farmacológicas como atividade antioxidante (SUN et al., 2011), antitumoral, antiangiogênica, anti-inflamatória, antialérgica e propriedades antivirais (MIDDLETON, KANDASWAMI e THEOHARIDES, 2000). Além disso, alguns flavonoides mostraram capacidade de inibir a inflamação crônica em vários modelos animais (CAMUESCO et al., 2004; HENDRIKS et al., 2004; KAWAGUCHI et al., 2004).

Além dos flavonoides, os taninos também são compostos fenólicos de muito interesse e são subdivididos em duas classes: taninos condensados e hidrolisáveis. O primeiro grupo é também conhecido como proantocianidinas, que são oligômeros dos flavan-3-óis e sua estrutura química é definida pelo tipo de monômero e, também, de acordo com a forma de ligação entre os monômeros (JAGANATH e CROZIER, 2010), sendo a mais comum a ligação entre os carbonos C4-C8 das unidades monoméricas, podendo ser do tipo α ou β (HAGERMAN, 2002). Os taninos hidrolisáveis são subdivididos em galotaninos e elagitaninos, que são facilmente hidrolisados por ácidos, bases ou enzimas (tanase) (KHANBABAEI e VAN REE, 2001), liberando ácidos gálico, caféico, elágico e uma molécula de açúcar (BRAVO, 1998).

Figura 1. Estruturas básicas dos principais flavonoides presentes nos alimentos (DEL RIO et al., 2013).

1.2 Cagaita (*Eugenia dysenterica* DC.)

Cagaita ou "cagaiteira" (*Eugenia dysenterica* DC.) é uma espécie nativa frutífera pertencente à família das Mirtáceas, sendo nativa da região do Cerrado. Esta espécie é distribuída principalmente, nos estados de Bahia, Goiás, Maranhão, Mato Grosso, Mato Grosso do Sul, Minas Gerais, Pará, Piauí, São Paulo, Tocantins e Distrito Federal (OLIVEIRA et al., 2011).

A árvore é de altura mediana (4 a 10 m) de tronco e ramos tortuosos, com casca suberosa e fendada bem característica, com folhas novas membranáceas e folhas adultas coriáceas, glabras ou quase glabras nas duas faces, oposto-cruzadas, de ovaladas a elípticas,

decíduas durante o florescimento. Ocorre em maior densidade em áreas com temperaturas médias anuais variando entre 21°C e 25°C e altitudes de 380 a 1100 m. A inflorescência é do tipo ráculo axilar que surge com a folhagem nova avermelhada. Suas flores vistosas formam panículas fasciculadas e são brancas, delicadas, com quatro pétalas, com cálice de quatro lacínios ovados e ciliados. Possui flores hermafroditas. Seus frutos são bagas globosas, suculentas, de cor amarelo-clara quando maduros, com uma a quatro sementes de cor creme, de sabor agradável e levemente ácido. Suas sementes são elipsoides e achatadas (CHAVES, 2001). Os períodos de frutificação ocorrem entre outubro e dezembro (OLIVEIRA et al., 2011).

A cagaita é consumida *in natura* e é considerada uma espécie de interesse, principalmente devido ao uso de seu fruto na culinária. Este uso é difundido entre os moradores do Cerrado, onde várias preparações regionais são feitas a partir da polpa deste fruto, especialmente doces, compotas, licores, refrigerantes, sucos e sorvetes (MARTINOTTO et al., 2008). A exploração desta espécie é uma atividade econômica muito promissora, devido às excelentes qualidades de seus frutos e suas diversas utilidades (de SOUZA et al., 2007).

O fruto da cagaita é uma boa fonte de vitaminas A e C, além de rico em compostos fenólicos. Por sua vez, as folhas de *E. dysenterica* são utilizadas na medicina popular para o tratamento de diferentes doenças: diarreia, diabetes e icterícia. Os extratos da fruta e da folha apresentam atividade antioxidante *in vitro*, assim como atividade inibitória das enzimas α -amilase e α -glicosidase (GENOVESE et al., 2008; DE SOUZA SCHMIDT GONCALVES, LAJOLO e GENOVESE, 2010). Consequentemente, a cagaita é uma fonte promissora de compostos bioativos que pode ser usada em diferentes indústrias e provavelmente no campo da medicina.

1.2.1 Compostos fenólicos da *Eugenia dysenterica* DC.

Os compostos fenólicos presentes na cagaita têm sido encontrados em diferentes partes da planta. No caso das sementes, foram encontrados teores de fenólicos totais de 137 mg EAG/g b.s. para o extrato etanólico, e 38 mg EAG/g b.s. para o extrato aquoso (NEUZA, MORENO e BERTANHA, 2010; ROESLER et al., 2007). Em relação ao fruto ou à polpa congelada, foi encontrado um valor de 0,32 mg EAG/g b.s. para extrato etanólico (ABADIO, SILVA e OLIVEIRA, 2012). No entanto, (GENOVESE et al., 2008) reportaram teores mais elevados destes compostos (15 mg EAG/g b.s.), utilizando metanol aquoso 70% como

solvente extrator. Essas variações nas concentrações de compostos fenólicos estão relacionadas com diferenças nos métodos de extração, condições ambientais e fatores pós-colheita. A identificação dos compostos presentes no fruto foi realizada, e foram encontrados catequina e derivados de quercetina e kaempferol (DE SOUZA SCHMIDT GONCALVES, LAJOLO e GENOVESE, 2010).

1.2.2 Capacidade antioxidante da cagaita

Genovese et al. (2008) avaliaram a capacidade antioxidante *in vitro* de diferentes frutas nativas brasileiras e polpas comerciais congeladas, incluindo *E. dysenterica*, através de duas metodologias diferentes: capacidade de sequestro do radical DPPH e branqueamento do sistema β -caroteno/ácido linoleico. Entre as polpas congeladas comerciais, a de cagaita foi a que apresentou a maior capacidade de sequestro do radical DPPH foi à polpa mais eficiente em inibir o branqueamento do sistema β -caroteno/ácido linoleico. As amostras apresentaram uma correlação positiva entre a capacidade antioxidante frente ao radical DPPH e o conteúdo de compostos fenólicos ($r=0,738$). No entanto, o mesmo não foi observado em relação ao método do β -caroteno ($r=0,414$). A polpa congelada apresentou concentração média inibitória do radical oxigênio de $455 \pm 31 \mu\text{mol Trolox/g b.s.}$ (GONÇALVES, LAJOLO e GENOVESE, 2010). Roesler *et al.* (2007) avaliaram a atividade antioxidante frente ao radical DPPH de diferentes partes da cagaita (sementes, polpa e resíduos), utilizando na extração dois diferentes solventes (água e etanol 95%). O extrato de sementes em etanol apresentou a melhor resposta com uma CI_{50} (concentração meia inibitória) de $14,15 \pm 0,18 \mu\text{g/mL}$ e uma correlação positiva entre a resposta antioxidante e o teor de compostos fenólicos ($R^2 = 0,9966$). Neuza, Moreno e Bertanha (2010), também pesquisaram a capacidade do extrato da cagaita frente ao DPPH, encontrando uma resposta dependente da concentração do extrato ($R^2 = 0,903$), assim como uma concentração média inibitória de $40,63 \text{ mg/mL}$ de reação e o máximo de atividade de inibição do radical de 95,92%.

1.2.3 Capacidade inibitória das enzimas α -glicosidase e α -amilase e a incidência na obesidade dos compostos fenólicos da Cagaita

A inibição das enzimas que hidrolisam carboidratos, tais como a α -amilase e a α -glicosidase, que estão presentes no trato digestivo, reduz a resposta glicêmica pós-prandial. Os inibidores destas enzimas podem ser de aproveitamento terapêutico para o tratamento ou

prevenção da diabetes. Evidências apontam o potencial do consumo de cagaita em inibir a atividade da α -amilase e da α -glicosidase *in vivo*. Gonçalves et al. (2010) avaliaram o potencial antidiabético de diferentes extratos ricos em flavonoides, obtidos a partir de frutas nativas brasileiras e polpas comerciais congeladas, por meio de metodologias *in vitro*. Os autores observaram que a eficiência dos compostos fenólicos para inibir a α -amilase (CI_{50} expresso em mg equivalente de catequina) da polpa congelada de cagaita foi a melhor, juntamente com a fruta de cupuaçu (CI_{50} 0,3 mg EC/mL). No caso da α -glicosidase, também se observou que a polpa da cagaita apresentou alta eficiência para inibir esta enzima, com CI_{50} de 0,9 mg EC/mL de reação.

A inibição das enzimas do metabolismo de carboidratos também parece ocorrer *in vivo*. Um estudo sobre o efeito de diferentes sucos clarificados de frutas nativas brasileiras na glicemia pós-prandial em indivíduos saudáveis evidenciou que o suco da cagaita (copo de 300 mL) apresentou a maior redução da glicemia máxima ($p < 0,05$), quando comparado aos demais sucos e ao controle (copo de água). Os resultados sugerem que a melhora na resposta glicêmica pode estar associada com a inibição das enzimas α -amilase e a α -glicosidase por parte dos compostos fenólicos presentes no suco (BALIESTEIRO, 2013).

Um estudo realizado por nosso grupo de pesquisa avaliou o efeito de extratos de cagaita concentrados em compostos fenólicos sobre a obesidade e as complicações metabólicas de camundongos C57BL/6J, induzidas pelo consumo de uma dieta rica em gordura e sacarose. Os resultados mostraram que os grupos que consumiram os extratos de cagaita apresentaram diminuição do ganho de peso corporal e da acumulação de gordura, sem afetar a ingestão de alimentos. Os autores associaram esses resultados a atenuação da hiperglicemia, hipertrigliceridemia e hipercolesterolemia, concluindo que a presença de composto fenólicos pode desempenhar um papel importante na prevenção da obesidade e suas anormalidades associadas, e apontar o seu potencial para o desenvolvimento de novas aplicações e estratégias complementares em alternativas terapêuticas (DONADO-PESTANA, BELCHIOR e GENOVESE et al., 2015).

Em relação à folha, Souza et al. (2012), avaliaram a inibição das enzimas α -amilase e α -glicosidase, utilizando diferentes solventes para a obtenção dos extratos: etanol, hexano e água, e verificaram uma inibição da α -amilase de 98,95%, 78,01%, e 93,08%, respectivamente, e α -glicosidase, uma inibição de 100% com todos os extratos. Adicionalmente, tanto o extrato aquoso como o etanólico apresentaram uma inibição das enzimas maior do que a dos controles utilizados (Acarbose e Desoxinojirimicina), com

valores de CI_{50} de 14,93 e 20,80 $\mu\text{g/mL}$, frente à α -amilase, e 0,46 e 0,77 $\mu\text{g/mL}$, para α -glicosidase.

1.3 Extratos secos

Os extratos secos são preparações sólidas, pulverulentas ou granuladas obtidas por evaporação, apresentando diferentes vantagens frente a preparações de outros tipos, como as fluídas. Tais vantagens são o aumento da estabilidade físico-química e microbiológica, elevada capacidade de transformação em diferentes tipos de formas farmacêuticas e alimentares e facilidade de transporte e armazenamento (OLIVEIRA & PETROVICK, 2010). Por definição, os pós utilizados para o preparo de alimentos devem ser preparados com ingredientes são e limpos, de primeira qualidade, apresentando aspecto fino ou granulado e cor de acordo com os componentes do material, além de ausência de sujidades, parasitos e larvas (ANVISA, 1978).

Atualmente, a utilização de materiais vegetais como matéria prima para a obtenção de extratos secos representa um campo em crescente expansão. Porém, para que os materiais possam ser utilizados na indústria, os extratos vegetais devem estar em formas padronizadas, com a caracterização qualitativa e quantitativa dos seus princípios ativos, fornecendo os requisitos de qualidade, efetividade e segurança exigidos (BRASIL, 2014). Um dos aspectos mais relevantes na obtenção do extrato padronizado é definir quais são as substâncias responsáveis pela ação terapêutica. No entanto, há vários pontos no processo produtivo como cultivo, colheita, secagem, moagem, extração e formulação, em que poderão ocorrer alterações na composição química destas substâncias ativas, vindo a interferir na qualidade do produto final (BORELLA et al., 2010).

1.4 Atomização

O método de atomização é uma técnica que utiliza um tipo de secador de atomização e é o mais comum na indústria, além de ser mais econômico, e tem sido largamente utilizado na indústria de aromas (BARROS e STRINGHETA, 2006). Neste secador, o fluido a ser desidratado é introduzido por aspersão sob pressão, e recebe uma corrente de ar quente sob temperaturas elevadas, formando instantaneamente partículas sólidas (OSORIO et al., 2010). O produto obtido pela atomização depende principalmente do material e das condições de operação, podendo ser um pó fino (10-50 μm) ou um produto com tamanho de partícula maior

(2-3 mm) (GHARSALLAOUI et al., 2007). Além disso, os pós obtidos por atomização reúnem padrões elevados de qualidade em relação à granulometria do produto, umidade final, homogeneidade, densidade, forma, e uma alta fluidez, sendo que esta propriedade pode ser atribuída à forma esférica das partículas obtidas (DA SILVA BASTOS et al., 2012)

Um dos parâmetros de qualidade de um extrato seco é a sua higroscopicidade, a qual deve ser a mais baixa possível para garantir sua estabilidade e evitar a contaminação biológica. Para melhorar e/ou conservar as características destes extratos secos, a indústria utiliza a microencapsulação, a qual faz uso de adjuvantes de secagem, que também contribuem para aumentar o rendimento do produto final e o tamanho final da partícula (AHMED et al., 2010). Os adjuvantes de secagem mais comuns são a goma arábica, os carboidratos de baixo peso molecular como maltodextrinas e sacarose, a celulose, a gelatina, os lipídeos, as proteínas (proteína da soja) e o dióxido de silício coloidal (GHARSALLAOUI et al., 2007; HOGAN et al., 2001).

Devido às diversas vantagens oferecidas pela microencapsulação de compostos mediante o emprego da técnica de atomização, muitos pesquisadores tem se centrado na aplicação desta técnica para aumentar e melhorar a estabilidade e a vida útil de compostos bioativos. Couto et al. (2013), avaliaram o impacto de diferentes variáveis do processo de atomização (fluxo de ar, temperatura de entrada do ar, e a velocidade de alimentação do extrato) na qualidade do extrato etanólico de folhas de cagaita seco sem o emprego de adjuvante, concluindo que os parâmetros analisados afetaram significativamente a maioria dos indicadores de qualidade avaliados, entre eles o teor de compostos fenólicos. Por sua vez, Silva et al. (2014), utilizaram a técnica de atomização para produzir um pigmento natural a partir do encapsulamento do resíduo do despulpamento da jabuticaba empregando maltodextrina como carreador. As amostras obtidas após o processamento mostraram altos teores de antocianinas e fenólicos, assim como boa atividade antioxidante e antimicrobiana. Os extratos secos encapsulados obtidos pela atomização de resíduos de vinificação da uva Bordo com maltodextrina, quando comparados com um controle obtido por liofilização sem emprego de carreador, apresentaram maior retenção de antocianinas, assim como melhores características de higroscopicidade e solubilidade. Os autores concluíram que é possível produzir extratos secos com cor estável ao longo do tempo utilizando a metodologia de atomização (SOUZA et al., 2014).

1.5 Agentes carreadores

Uma das principais vantagens da obtenção de extratos secos por atomização é a possibilidade do uso de um agente carreador. Os principais objetivos do uso dos agentes carreadores no processo de atomização são: proteger o material a ser secado da degradação (aumento da vida útil de prateleira), e evitar a presença de deficiências tecnológicas nos produtos obtidos, como a alta higroscopicidade, baixa solubilidade e pegajosidade (VIDOVIĆ et al., 2014). Os critérios para a seleção do agente carreador dependerão da finalidade do produto e das propriedades físico-químicas do material. As principais características a considerar são a solubilidade; peso molecular; temperatura de transição vítrea; cristalinidade; difusibilidade; capacidade de formação de película e emulsões (GHARSALLAOUI et al., 2007).

1.5.1 Goma arábica (GA)

A goma arábica é um exsudado gomoso e comestível composta por polissacarídeos e glicoproteínas, extraída dos caules e ramos da *Acacia senegal* e *Acacia seyal* (PHILLIPS e WILLIAMS, 2009). Comercialmente, a goma arábica está disponível sob a forma de pó ou cristais mais ou menos arredondados de cor amarelo pálido a cinza claro. GA é usada na indústria de alimentos como estabilizador, agente espessante e agente emulsionante (VERBEKEN, DIERCKX e DEWETTINCK, 2003).

Estruturalmente, a goma arábica é um polissacarídeo altamente ramificado, neutro ou ligeiramente ácido, encontrado como uma mistura de sais de cálcio, magnésio e potássio do ácido polissacarídeo (ácido arábico). O esqueleto é composto por unidades de 1,3-β-D-galactopiranosilo. As cadeias laterais são compostas por dois a cinco unidades de 1,3-β-D-galactopiranosilo unidas à cadeia principal por ligações C-1,6. Tanto a cadeia principal como as cadeias laterais contêm unidades de α-L-arabinofuranosídeo, α-L-rhamnopiranosil, β-D-glucopiranosil e 4-O-metil-β-D-glucopiranosil, sendo que, as duas últimas, estão presentes como unidades terminais (ALI, ZIADA e BLUNDEN, 2009). A goma arábica consta de três frações principais, sendo que a maior parte da goma (88,4% do total), um arabinogalactano (AG), apresenta um baixo teor de proteína e uma massa molecular de $3,8 \times 10^5$ Da. A segunda fração (10,4% do total), um complexo arabinogalactano-proteína (AGP), apresenta 11,8% de proteína e uma massa molecular de $1,45 \times 10^6$ Da. A terceira fração (1,2% do total), chamada de glicoproteína de baixo peso molecular, apresenta um teor de proteína de 47,3% e uma massa molecular de $2,5 \times 10^5$ Da (RANDALL, PHILLIPS e WILLIAMS, 1989).

O uso de goma arábica como agente carreador no processo de secagem por atomização tem sido amplamente explorado. Yousefi, Emam-Djomeh e Mousavi (2011), ao avaliar o efeito de diferentes agentes carregadores nas propriedades físico-químicas de suco de romã, encontraram que as amostras obtidas com goma arábica apresentaram maior rendimento de processo, temperatura de transição vítrea, solubilidade e melhor densidade aparente do que as amostras obtidas usando maltodextrina e amido ceroso.

Além de melhorar as propriedades físicas das amostras, a goma arábica ajuda na conservação e estabilidade de compostos fenólicos. Khazaei et al. (2014), relataram que o uso da goma arábica como agente carreador na secagem por atomização de antocianinas de pétalas de açafrão, diminuiu a perda desses compostos em armazenamento quando comparado com o controle obtido sem agente carreador. O uso de uma mistura de maltodextrina-goma arábica na encapsulação de epigallocatequina galato conservou a capacidade antioxidante e melhorou a biodisponibilidade do composto (PERES et al., 2011).

1.5.2 Inulina

A inulina ou os frutanos de tipo inulina são carboidratos naturais de reserva não estruturais, encontrados em alimentos comumente consumidos, como banana, alho, cebola e trigo, assim como na raiz da chicória (*Cichorium intybus*), dália (*Dahlia pinuata* Cav.) e alcachofra-girassol (*Helianthus tuberosus*), de onde são extraídos em escala industrial (CAPRILES e ARÊAS, 2012).

Estruturalmente, a inulina é uma mistura polidispersa de moléculas constituída principalmente por ligações β -(2 $\rightarrow$ 1)-D-frutossil-frutose, estrutura identificada como F_m . No entanto, uma unidade de glucopirranose no final da cadeia pode estar presente, sendo esta estrutura identificada como GF_n (Figura 2) (CHI et al., 2011). Porém, De Leenheer e Hoebregs (1994), determinaram que a inulina obtida da chicória é um frutano ligeiramente ramificado, sendo que a quantidade de ramificações β -(2 $\rightarrow$ 6) representa de 1 a 2% do total de ligações. O grau de polimerização da inulina varia entre 2 a 60 (KALYANI NAIR, KHARB e THOMPSON, 2010). Devido à configuração β do carbono anomérico 2 dos monómeros de frutose, estas moléculas são resistentes à hidrólise por enzimas digestivas presentes no trato gastrointestinal (GIBSON et al., 2010). Adicionalmente, a capacidade da inulina de modificar a microbiota estimulando o crescimento de bactérias benéficas (*bifidobactérias*) para a saúde do indivíduo hospedeiro, torna este material um alimento prebiótico. Devido a isto, o número de estudos avaliando seu uso como agente carreador aumentou significativamente, principalmente na encapsulação de probióticos (PINTO et al., 2015).

Kingwatee et al. (2014) avaliaram o efeito de diferentes misturas de carreadores (maltodextrina:Inulina e goma arábica:Inulina) na secagem por atomização de suco de lichia enriquecido com *Lactobacillus casei*, e observaram que a adição da inulina aumentou a viabilidade celular, assim como a retenção de compostos fenólicos e ácido ascórbico. No entanto, a higroscopicidade dos produtos atomizados aumentou.

Figura 2. Estrutura química da inulina (GF_n) e (F_m) (Kays e Nottingham, 2007).

A inulina também foi utilizada na microencapsulação dos compostos bioativos obtidos da tuna (*Opuntia ficus-indica*), observando-se que o uso deste material como agente carreador apresentou efeito protetor das betacianinas e das indicaxantinas presentes nos produtos obtidos por atomização, similar aos resultados obtidos usando maltodextrina como agente carreador (Saénz *et al.*, 2009).

Considerando o que foi apresentado, este trabalho teve como objetivos obter extratos secos a partir do fruto da cagaita, através da secagem por atomização, utilizando goma arábica e inulina como agentes carreadores, e avaliar o efeito dos parâmetros de processo (temperatura de entrada do ar: 120°C, 140°C e 160°C; e concentração de carregador: 10, 20 e 30%) sobre algumas propriedades físico-químicas dos pós produzidos, assim como seu potencial funcional *in vitro*.

2. OBJETIVOS

O objetivo geral deste projeto é obter extratos secos do fruto da cagaita (*Eugenia dysenterica*) pelo método de secagem por atomização, avaliando a influência de diferentes parâmetros de processo sobre as propriedades físico-químicas e o potencial funcional *in vitro* dos pós obtidos.

Os objetivos específicos são:

1. Determinar o efeito da secagem por atomização de extratos da cagaita sobre as propriedades tecnológicas dos pós;
2. Quantificar o teor de compostos bioativos fenólicos presentes nos pós, sua retenção após o processo de secagem por atomização e sua estabilidade durante o armazenamento;
3. Determinar o potencial funcional *in vitro* dos extratos secos obtidos por atomização, através da determinação das capacidades antioxidante, antimicrobiana e inibitória das enzimas envolvidas no metabolismo de carboidratos.

3. RESULTADOS

Os resultados foram divididos em dois capítulos e foram apresentados na forma de artigos científicos, conforme a seguir:

1. Effect of spray drying conditions on the physical properties of Cagaita (*Eugenia dysenterica* DC.) fruit extracts (submitted in *Food and Bioproducts Processing*)
2. Stability of phenolic compounds and functional properties of encapsulated cagaita (*Eugenia dysenterica* DC.) fruit extract (submitted in *Food Chemistry*)

Effect of spray drying conditions on the physical properties, *in vitro* antioxidant capacity and anti-obesity properties of Cagaita (*Eugenia dysenterica* DC.) fruits powders

ABSTRACT AND KEYWORDS PAGE

Cagaita is a Brazilian native fruit with potential health effects associated to its high content and peculiar profile of polyphenols. The aim of this work was to produce spray dried extracts from *E. dysenterica* fruit comparing gum arabic (GA) and inulin as carrier agents and to evaluate the effect of different drying conditions on physical properties of powders. These inlet air temperature (120°C, 140°C and 160°C), and concentrations of carrier agents (10%, 20% and 30%) were tested. Samples were evaluated for moisture content, water activity, hygroscopicity, solubility, glass transition temperature, color and particle morphology, and results were subjected to response surface methodology. A freeze dried sample was used for comparison. Better yield process was observed when using GA as carrier agent. Regarding moisture content and water activity of powders, all samples showed better values than those presented by the freeze-dried sample. The spray drying process increased the solubility and reduced the hygroscopicity of the samples. Glass transition temperature was more affected by temperature in case of samples with GA; in contrast, concentration of carrier agent was the parameter that most influenced the samples spray-dried with inulin. The morphology of the particles was affected by both the carrier concentration and temperature.

Keywords: *E. dysenterica*, Powder extract, Glass transition temperature, Inlet air temperature, Carrier agents, Spray-dried.

1. INTRODUCTION

Cagaita (*Eugenia dysenterica* DC.) is an important native fruitful species belonging to the Myrtaceae family and is native to Brazilian Cerrado, occurring at higher density in areas with temperatures ranging from 21°C to 25°C and altitudes from 380 to 1100 m, with fructification period between November and January (De Souza *et al.*, 2007). *E. dysenterica* is used in Brazilian folk medicine in treatment of various diseases (Lima *et al.*, 2010) and the fruits are also used in typical preparations such as jams and jellies (Cardoso *et al.*, 2011).

Previous studies have demonstrated that fruits of *E. dysenterica* are a good source of polyphenols, among them quercetin, kaempferol and catechin derivatives as well as ellagitannins, which in the case of this fruit have been related with antioxidant activity and inhibiting key enzymes in the metabolism of carbohydrates such as α -glucosidase and α -amylase (Genovese *et al.*, 2008; De Souza Schmidt Goncalves, Lajolo e Genovese, 2010); hence, the use of this fruit should be a strategy to control hyperglycemia. However, the consumption of this fruit in Brazil is limited due to its high perishability and high transport costs, and these two limiting factors need to be overcome to provide the Brazilian and worldwide population with this source of bioactive compounds.

In this sense, production of powders from *E. dysenterica* extracts is an alternative to reduce transportation costs, protect the main bioactive components of the food matrix and ensure product availability. One of the techniques used for this purpose is spray drying, in which a fluid product is transformed into a powder by atomizing it into a hot gas stream. The low processing cost and equipment availability make this the most common technology used in food industry (Gharsallaoui *et al.*, 2007). The process involves four stages: atomization of feed, spray-air contact, evaporation of water, and separation of the dried product from the drying air (Ronald, 1997). Spray dryer can be used in both heat-sensitive and heat-resistant products, without major changes in their overall quality (Perdana *et al.*, 2014; Silva *et al.*, 2014; Wu *et al.*, 2014).

Different parameters can be controlled during the spray-drying process in order to obtain desired characteristics in the final product. However, some intrinsic drawbacks, such as stickiness, hygroscopicity and low solubility of the product may occur (Vidović *et al.*, 2014). Drying additives (or carriers) are commonly used in order to avoid these technological problems. The choice of the appropriate carrier depends on the desired physico-chemical properties and the final application of the powdered extract.

Based on the considerations regarding the potential of *E. dysenterica* to inhibit enzymes involved in carbohydrate metabolism, and also its restricted consumption due to its

limited availability, the aim of this study were to produce powdered fruit extract from *E. dysenterica* by spray drying technology using gum arabic and inulin as carrier agent, in order to evaluate the impact of drying conditions on the physical properties of spray-dried extracts.

2. MATERIAL AND METHODS

2.1 Material. Frozen commercial pulp of cagaita (*Eugenia dysenterica* DC.) was purchased from Cooperativa dos Agricultores Familiares Agroextrativistas Grande Sertão (Montes Claros, MG, Brazil). The carrier agents used in this work were food grade inulin extracted from chicory (SIBA ingredients Com. Ltd, Brazil), and gum arabic (Nexira Brazil Com. Ltd., Brazil).

2.2 Selection of solvent and method for extraction of phenolic compounds

2.2.1 Solvent type. Selection of solvent for extraction was based in the extraction efficiency of phenolic compounds from *E. dysenterica*, different solvents were assessed (water, aqueous ethanol 50%, 70%, 80%, 90 and absolute ethanol), in the ratio of 1:20 (w/v). The sample was homogenized for 3 min at 14000 rpm using an Ultra-Turrax T-25 Tissue homogenizer (Janke & Kunkel, IKA Ò-Labortechnik, Saufen, Germany). The homogenized extract was then centrifuged for 10 min at 5640 g, supernatant was keep at 4°C until subsequent analysis.

2.2.2 Extraction method. Different extraction methods were compared in order to select the one with the highest extraction yield of phenolic compounds. In all extractions methods, was used the solvent previously selected.

2.2.2.1 Ultrasound extraction. Sample (2 g) was added to 20 mL of solvent and then sonicated in an ultrasound bath (UltraCleaner 1400A), at a frequency of 40 kHz for 30 min at room temperature. After that the extract was separated from the sample residue by centrifugation for 10 min at 5640 g. The supernatant was concentrated at 40°C in a rotatory evaporator (Rotavapor® R-210, BUCHI Switzerland), and then, the volume was adjusted to final volume with the same solvent of extraction.

2.2.2.2 Percolation. For percolation, the sample (15 g) was moistened with solvent for 24 hours in the ratio of 1:20 (w/v). Then the solvent was collected at a control rate (10 drops per min), with addition of fresh solvent (150 mL). The extract was then separated from the sample residue by centrifugation (5640 g for 10 min). Extraction was repeated twice. The resultant

extract was concentrated at 40°C in a rotatory evaporator and the volume was adjusted to a final volume with the same solvent of extraction.

2.2.2.3 Maceration. Extraction was carried out with the help of stirring. Five grams of sample was placed in contact with the solvent (30 mL) for 24 h and then the extract was separated from the residue by centrifugation, extraction was repeated once. After that the two extracts were combined and concentrated. Finally, the volume was adjust with the same solvent.

2.2.2.4 Homogenization. The sample (5 g) was homogenized with solvent (100 mL), for 3 min at 14000 rpm using a homogenizer (Ultra-Turrax T25; IKA, Germany). The homogenized extract was centrifuged for 10 min at 5640 rpm, supernatant was concentrated and kept at 4°C until subsequent analysis.

2.2 Production of liquid extract. The pulp samples were homogenized with aqueous ethanol (79%, v/v) in a ratio of 1:20 (w/v) for 3 min at 14000 rpm using a homogenizer (Ultra-Turrax T25; IKA, Germany). The extract was then separated from the sample residue by centrifugation (5640 g for 10 min). Extraction of residue was repeated twice. The resultant extracts were pooled and concentrated at 40 °C in a rotatory evaporator (BUCHI, B-491) until complete elimination of ethanol, and kept at 4 °C until subsequent analysis.

The concentrations of total solids, ashes, protein and titrable acidity of the concentrated extract were determined according to AOAC (AOAC, 1990). Total sugars (TS) were determined using the method of Dubois et al. (Dubois et al., 1956); pH was measured with a potentiometer (Hanna pH21) and soluble solids (SS, °Brix) with a refractometer (Reichert r2 mini).

2.2 Spray drying. The feed liquid was prepared by mixing the concentrated extract with different concentrations (10%, 20% and 30% w/v) of a carrier agent (inulin or gum arabic). Afterwards, the carrier agent was dispersed in the concentrated extract using a homogenizer (Ultra-Turrax T25; IKA, Germany) for 2 min at 20000 rpm. Then, the prepared feed liquid was atomized with a spray dryer (model MSD 1.0, Labmaq, Brazil) at different temperatures (120 °C, 140 °C and 160 °C) by a peristaltic pump at a fixed rate of 0.60 m³/min. The production yield was expressed as the ratio between the total solids content in the resulting powder and the total solids content in the feeding mixture. The formulation was mixed continuously by a magnetic stirrer during the entire process of spray drying.

2.2 Freeze-dried sample. A control sample was obtained by lyophilization of the extract in a Pironi 501 freeze-dryer (Thermo Electron Corporation, New York, USA) at -80°C and 100 mTorr for 96 h.

2.3 Analytical methods

2.3.1 Moisture content (*M*) and water activity (*a_w*)

Moisture of samples was determined by using a moisture analyzer (MB35 Halogen, Ohaus, Switzerland). The *a_w* was measured using a dew point hygrometer (Aqualab, Decagon Devices, USA).

2.3.2 Higroscopicity (*H*)

Higroscopicity of powders was determined according to the methodology described by Cai and Corke (Cai e Corke, 2000), with some modifications. Samples (about 0.5 g) of each powder were placed in a desiccator containing saturated solution of NaCl (RH 75.3%). The results were expressed as the mass of water absorbed per 100 g of sample after 7 days of storage.

2.3.3 Solubility (*S*)

Solubility of spray dried samples was determined according to the methodology reported by Eastman and Moore (Eastman e Moore, 1984), with some modifications. The sample (0.5 g) was added to recipients containing 50 mL of distilled water, stirred at 110 rpm for 30 min using a TE-420 shaker (Tecnal, Piracicaba, Brazil), and then centrifuged at 4000 rpm for 5 min. An aliquot (25 mL) of each supernatant was removed, transferred to porcelain dishes, and dried to constant weight in an oven at 105 °C. Solubility was expressed as a percentage (%).

2.3.4 Glass transition temperature (*T_g*)

Glass transition temperature was determined by differential scanning calorimetry in a DSC (DSC 4000, Perkin-Elmer Corp., Norwalk, CT, USA) equipped with an intracooler SP (CA – Cooling accessory) according to Grabowsky et al. (Grabowski, Truong e Daubert, 2006), with some modifications. About 5-10 mg of *E.dysenterica* powder samples were placed into 50 uL aluminum pans and hermetically sealed. Samples were cooled to -60 °C at 10 °C/min, kept isothermally at -60 °C for 4 min, then heated from -60 °C to 120 °C at a

constant rate of 10 °C/min, and finally cooled to 30 °C at 10 °C/ min, under a dynamic atmosphere of Helium (20 mL/min). An empty pan was used as reference. Results were expressed as the midpoint value for glass transition range. Thermograms were examined using Pyris thermal analysis software (Perkin Elmer co., São Paulo., Brazil). The temperature and heat of melting were calibrated with indium (initial temperature of 156.6 °C). All analyses were done in triplicate.

2.3.5 Morphological characterization of the particles - Scanning Electron Microscopy (SEM)

The morphology of the powdered extracts was assessed using scanning electron microscopy (Hitachi TM- 3000, Hiscope – New Jersey, USA) with the TM 3000 program. The microcapsules were placed on strips of double-faced carbon tape (Ted Pella, Inc., Redding, CA). Afterwards, they were fixed in aluminium stubs. The images were captured with voltage acceleration of 5 kV and a current of 1750 mA.

2.3.6 Instrumental color

Color parameters were assessed using a reflectance spectrophotometer (Color Quest XE, HunterLab, Fairfax, VA, USA). Calibration was done using black and white calibration plates, provided with the instrument. Results were expressed according to the CIELAB color system (L^* =lightness, a^* =redness, and b^* =greenness).

2.4 Statistical analysis. A 3^2 full-factorial design coupled with response surface methodology (RSM) was applied to evaluate the effects of T and C totaling 9 trials for each carrier agent. Data were expressed as means followed by the standard deviation of three independent measurements. Prior to one-way ANOVA application, experimental results were formally checked for normality (Anderson-Darling test) and homoscedasticity (Brown-Forsythe test). The means among the different treatments were compared using the Tukey test ($p < 0.05$). Correlation analysis based on the Pearson's correlation coefficient was calculated to analyze the degree of association between pairwise variables and the probability level (p -value) of these correlations was also provided. P-values below 5% were used to establish differences (Granato, De Araújo Calado e Jarvis, 2014).

When statistical differences were obtained using one-way ANOVA, linear multiple regression analysis based on the response surface methodology was applied. For this purpose,

linear, quadratic, and interactions between factors (concentration of carrier agent and inlet air temperature) were calculated and results $[E(y)]$ were expressed using Equation x:

$$E(y) = B_0 + B_1x_1 + B_2x_2 + B_{11}x_1^2 + B_{22}x_2^2 + B_{12}x_1x_2$$

Where $E(y)$ is the predicted response, B_0 is a constant, B_1 , B_2 , B_{11} , and B_{12} are the regression coefficients and x_1 , x_2 are the factors under investigation. After modeling each response variable, the quality of the proposed model was assessed: firstly, the statistical significant was tested by ANOVA and terms that presented p-value higher than 0.05 were deleted and data were refitted to the parameters that showed significance on the response. Then, the explained variance (R^2) and the adjusted R^2 (adj R^2) were also calculated, while the residuals were obtained and tested for obvious patterns and also for normality (Anderson-Darling test) (Granato *et al.*, 2014). Surface response plots of the fitted RSM models were generated using Statistica v. 7.0 software in order to visualize the relationships between the responses and the independent variables.

3. RESULTS AND DISCUSSION

The physicochemical characteristics of the cagaita fruit extract are presented in table 1. According to the results obtained *E. dysenterica* extract present high content of solids, total soluble solids (Brix), ashes, proteins and high titrable acidity, but low content of carbohydrates and similar value of pH, in comparison to the results obtained for the whole fruit (Cardoso *et al.*, 2011).

After ethanol removal by rotaevaporation, the spray drying technique was employed to obtain the powders. Two different parameters were assessed, namely drying temperature (T), and concentration (C) of the carrier agent (gum arabic, GA; or inulin). The choice of the temperatures was based on preliminary tests (results not shown), and the carrier agents were selected considering that both inulin and gum arabic are not hydrolyzed in the stomach and the small intestine, being fermented in the large intestine by microflora (Stevens, Meriggi e Booten, 2001; Ali, Ziada e Blunden, 2009). Therefore, there is no increase in the glycemic index, becoming potential ingredients for foods destined to diabetics.

Table 1. Physicochemical characterization of *E. dysenterica* extract.

Parameter	Mean $\pm$ SD
Total solids (%)	19.30 $\pm$ 0.47
Ashes (%)	0.81 $\pm$ 0.02
Total soluble solids ($^{\circ}$ Brix)	18.20 $\pm$ 0.39
pH	2.93 $\pm$ 0.00
Titrate acidity (g acid citric/100 g)	1.77 $\pm$ 0.03
Carbohydrates (g glucose/100 g)	1.46 $\pm$ 0.07
Proteins (%)	1.69 $\pm$ 0.13

Values are expressed as means $\pm$ SD (n=3).

The values of process yield for spray dried powders with gum arabic (41.1 to 81.20%, $p<0.05$) were slightly higher than those obtained using inulin (37.0 to 75.6%, $p<0.05$) (**Tables 2 and 3**). Fang and Bhandari (Fang e Bhandari, 2012) reported similar results for spray drying of bayberry juice using whey protein isolate in different concentrations as carrier agent (recovery of powders varied between 45.6 to 56.2%). Lower values of recovery, between 5.15 and 48.13%, were reported by Krishnaiah et al. (Krishnaiah, Sarbatly e Nithyanandam, 2012) who assessed two different carrier agents, κ -carrageenan and maltodextrin, and different temperatures (90 to 140°C) in the microencapsulation of *Morinda citrifolia* L. extract by spray drying. Increases in drying temperature and concentration of carrier agents led to increases in the yield process, which can be attributed to better water removal at higher temperatures, and to protective effect of the carrier at higher concentrations, avoiding stickiness of material in the dry chamber wall. Similar results for the influence of the temperature on recovery of black mulberry juice powder were reported (Fazaeli *et al.*, 2012). However, Tonon et al. (Tonon, Brabet e Hubinger, 2008) reported a decrease in yield with an increase in maltodextrin concentration, attributed to the higher viscosity of the mixture.

Table 2. Experimental design with coded and real values, production yield (Y), water activity (a_w), moisture content (M), solubility (S), higroscopicity (H), glass transition temperature (Tg) and color values of spray-dried *E. dysenterica* powders using arabic gum.

Test	Drying conditions		Y	a_w	M	S	H	Tg	Color		
	T	C							L*	a*	b*
1	120 (-1)	10 (-1)	41.13	0.26±0.0 ^f	5.01±0.0 ^g	94.4±0.9 ^d	15.7±0.4 ^b	39.2±3.8 ^{cd}	90.3±0.1 ^a	0.2±0.0 ^b	11.4±0.0 ^{ab}
2	120 (-1)	20 (0)	64.05	0.19±0.0 ^d	3.81±0.3 ^{ef}	95.1±0.2 ^{cd}	14.9±0.2 ^a	47.2±0.8 ^{bc}	88.2±0.6 ^c	0.1±0.0 ^{bc}	10.9±0.2 ^a
3	120 (-1)	30 (1)	75.51	0.16±0.0 ^c	4.75±0.2 ^g	95.6±0.4 ^{bcd}	14.8±0.0 ^a	68.1±1.7 ^a	89.0±0.2 ^{abc}	-0.1±0.0 ^b	11.3±0.4 ^{ab}
4	140 (0)	10 (-1)	55.78	0.21±0.0 ^e	3.45±0.3 ^{cd}	95.2±0.3 ^{bcd}	17.7±0.5 ^d	34.5±0.7 ^d	86.2±0.5 ^d	1.8±0.2 ^e	15.2±0.3 ^e
5	140 (0)	20 (0)	71.26	0.17±0.0 ^{cd}	3.64±0.1 ^{de}	96.4±0.4 ^{abc}	17.1±0.3 ^c	47.8±4.1 ^{bc}	88.1±0.4 ^{abc}	0.1±0.0 ^{ab}	11.7±0.0 ^b
6	140 (0)	30 (1)	79.50	0.15±0.0 ^b	4.06±0.1 ^f	96.6±1.4 ^{ab}	16.9±0.1 ^c	62.8±4.8 ^a	88.6±1.8 ^{bc}	0.1±0.0 ^{bc}	12.7±0.2 ^c
7	160 (1)	10 (-1)	68.63	0.17±0.0 ^c	2.55±0.2 ^a	96.4±0.7 ^{abc}	18.8±0.2 ^e	46.7±0.7 ^{bc}	83.3±0.3 ^e	2.7±0.1 ^f	16.5±0.1 ^f
8	160 (1)	20 (0)	75.13	0.12±0.0 ^a	2.9±0.1 ^{ab}	97.4±0.6 ^a	18.2±0.3 ^{de}	51.6±2.3 ^b	87.5±0.3 ^{cd}	0.3±0.0 ^c	14.0±0.5 ^d
9	160 (1)	30 (1)	81.20	0.13±0.0 ^a	3.17±0.1 ^{bc}	97.8±0.7 ^a	16.6±0.2 ^c	64.5±4.8 ^a	84.2±0.3 ^e	1.2±0.0 ^d	15.6±0.3 ^e
Freeze-dried	---	---	---	0.75 ± 0.0 ^g	9.55 ± 0.1 ^h	66.5±1.9 ^e	34.42±0.6 ^f	---	57.8±0.5 ^f	11.3±0.1 ^g	25.3±0.1 ^g
Arabic gum	---	---	---	---	---	---	---	---	90.1±0.5 ^{ab}	-0.4±0.0 ^a	11.1±0.2 ^a

C, concentration of carrier agents (%); T, inlet air temperature (°C); Yield (%); a_w , water activity; M, moisture content (%); S, solubility (g DW/100 g H₂O); Higroscopicity (g H₂O/100 g DW); Tg, glass transition temperature (°C); L, lightness; a and b, color-opponent dimensions. Values are expressed as means ± SD (n=3). Different superscripts letters in the same column mean significantly differences ($p<0.05$).

Table 3. Experimental design with coded and real values, production yield (Y), water activity(a_w), moisture content (M), solubility (S), higroscopicity (H), glass transition temperature (Tg) and color values of spray-dried *E. dysenterica* powders using Inulin.

Test	Drying conditions		Y	a_w	M	S	H	Tg	Color		
	T	C							L*	a*	b*
1	120 (-1)	10 (-1)	37.00	0.26±0.0 ^f	3.03±0.2 ^{cd}	88.2±0.7 ^c	17.0±0.3 ^d	30.0±3.4 ^e	80.7±0.2 ^{cd}	2.7±0.0 ^f	22.4±0.9 ^e
2	120 (-1)	20 (0)	62.48	0.20±0.0 ^e	3.13±0.4 ^{de}	91.9±0.5 ^b	15.3±0.2 ^{bc}	45.2±0.1 ^{cd}	88.2±1.6 ^b	0.6±0.0 ^d	14.1±0.2 ^c
3	120 (-1)	30 (1)	72.55	0.18±0.0 ^{de}	3.72±0.2 ^f	95.6±1.2 ^a	13.8±0.0 ^a	48.6±0.4 ^c	88.1±3.8 ^b	0.2±0.0 ^c	11.3±1.0 ^b
4	140 (0)	10 (-1)	41.49	0.19±0.0 ^{de}	2.64±0.3 ^{bc}	86.8±4.7 ^c	17.8±0.6 ^e	44.6±0.5 ^d	77.7±0.2 ^d	4.9±0.2 ^h	21.4±0.3 ^{de}
5	140 (0)	20 (0)	70.81	0.17±0.0 ^{cd}	2.81±0.1 ^{cd}	92.0±0.8 ^b	15.9±0.2 ^c	49.0±0.7 ^c	88.6±1.4 ^b	0.9±0.0 ^{de}	15.12±0.1 ^c
6	140 (0)	30 (1)	74.65	0.16±0.0 ^{bc}	3.40±0.2 ^{ef}	95.9±0.3 ^a	14.8±0.2 ^b	52.8±0.9 ^b	89.0±0.7 ^b	0.37±0.0 ^{bc}	12.6±0.4 ^b
7	160 (1)	10 (-1)	54.45	0.31±0.0 ^g	1.93±0.3 ^a	87.7±0.5 ^c	19.4±0.4 ^f	55.2±0.6 ^b	76.6±0.5 ^e	5.1±0.1 ⁱ	25.3±0.1 ^f
8	160 (1)	20 (0)	72.43	0.14±0.0 ^a	2.26±0.1 ^{ab}	91.9±0.5 ^b	16.9±0.3 ^d	69.7±0.4 ^a	82.7±0.2 ^c	3.3±0.2 ^g	20.8±0.4 ^d
9	160 (1)	30 (1)	75.64	0.15±0.0 ^{ab}	2.63±0.1 ^{bc}	95.6±0.1 ^a	14.8±0.3 ^b	73.2±1.1 ^a	89.0±0.2 ^b	1.2±0.0 ^e	14.8±0.1 ^c
Freeze-dried	---	---	---	0.75 ± 0.0 ^h	9.55 ± 0.1 ^g	66.5±1.9 ^d	34.4±0.6 ^g	---	57.8±0.5 ^e	11.3±0.1 ^j	25.3±0.1 ^f
Inulin	---	---	---	---	---	---	---	---	94.6±0.1 ^a	-0.2±0.0 ^a	2.4±0.0 ^a

T, inlet air temperature (°C); C, concentration of carrier agents (%); Yield (%); a_w , water activity; M, moisture content (%); S, solubility (g DW/100 g H₂O); Higroscopicity (g H₂O/100 g DW); Tg, glass transition temperature (°C); L*, lightness; a* and b*, color-opponent dimensions. Values are expressed as means ± SD (n=3). Different superscripts letters in the same column mean significantly differences ($p<0.05$).

3.1 Selection of solvent and methodology for phenolic extraction from fruit pulps.

The extraction process was carried out at room temperature using water, ethanol and aqueous ethanol at different concentrations (50%, 70%, 80% and 90%). The efficiency on extraction of phenolic compounds is shown in **Figure 1a**. It can be observed a decrease of phenolic compounds extraction with an increase in ethanol concentration above 50% of aqueous ethanol, a phenomenon also observed by (Liu, Wei e Liao, 2013) for the extraction of phenolic compounds from *Euryale ferox* seed shells. Absolute ethanol showed the poorest extraction of phenolic compounds, 54% less compared to 50% aqueous ethanol. Water extracted 76.28% of the amount obtained with 50% ethanol, which could be explained due to the lower viscosity of ethanol, which enables alteration of the plant structure through matrix swelling, allowing the solvent to penetrate completely the sample and subsequently disrupting bonding between solutes and sample matrices (Şahin-Nadeem *et al.*, 2013b). Moreover, the addition of water to other solvent can increase the dielectric constant and consequently polarity indices, increasing the extraction capacity of the mixture (Spigno e De Faveri, 2009). Although there was no significant difference among extraction efficiency of 50% and 70% aqueous ethanol, 50% aqueous ethanol was selected as the solvent for extraction of phenolic compounds from *E. dysenterica* in this work because of the lower cost.

Figure 1. Effect of extraction condition on total phenolic content by a) water, ethanol and aqueous ethanol, and b) different methodologies.

Next, different methods of extraction using 50% aqueous ethanol were compared (**Figure 1b**). The efficiency was Ultra-Turrax > Percolation > Maceration > Sonication. Ultra-Turrax was 34.62% more efficient than sonication, and 12.28% than maceration. Homogenization effectiveness is probably due to cell disruption of the food matrix. In this way, due to the higher efficiency and lower time of extraction, Ultra-Turrax homogenization with 50% aqueous ethanol was selected as the technique for phenolic extraction in the present work.

3.1 Moisture content (M) and water activity (a_w)

For moisture content, water activity, solubility, hygroscopicity and glass transition temperature the fitted equations were obtained (**Table 4**), and the statistical adequacy of these models was checked calculating the R^2 , adjusted R^2 , and also by the distribution of the residues.

All spray dried samples presented values of water activity lower than 0.6, indicating that they are microbiologically stable, as there is no microbial growth below this value of a_w (Damodaran, Parkin e Fennema, 2010). Carrier concentration had a negative effect on water activity of powders obtained with inulin and gum arabic; the higher the concentration, the lower the water activity. Additionally, temperature had a negative effect in powders treated with gum arabic. However, the interaction of C and T had a positive influence in gum arabic samples (**Table 4**).

Powder moisture contents were generally lower than 5% (**Tables 2 and 3**), consistent with those reported for spray dried byproducts of bordo grapes (De Souza *et al.*), and spray dried beetroot juice (Pitalua *et al.*, 2010). Low moisture prevents the agglomeration of particles avoiding the caking of powders, which could reduce retention of active components and other properties such as flow and dispersion of powders (Da Silva *et al.*, 2013). Higher moisture contents were observed using higher carrier concentrations ($p < 0.05$) (**Tables 2 and 3**). Similar behavior was observed for powder of *Satureja montana* (Vidović *et al.*, 2014). This may be explained due to the difficulty of water to diffuse through the carrier agent, which forms a crust on the surface of the particle (Goula e Adamopoulos, 2010).

Table 4. Regression coefficients and coefficients of determination (R and R² adjusted) for physical properties of *E. dysenterica* spray dried powders.

Parameter	Water activity				Higroscopicity				Solubility				Moisture				Tg			
	AG	<i>p</i>	I	<i>p</i>	AG	<i>p</i>	I	<i>P</i>	AG	<i>p</i>	I	<i>p</i>	AG	<i>p</i>	I	<i>p</i>	AG	<i>p</i>	I	<i>p</i>
Mean	0.18	<0.01	0.17	<0.01	17.2	<0.01	16.2	<0.01	96.26	0	91.88	<0.01	3.70	<0.01	2.84	<0.01	51.38	<0.01	52.05	<0.01
T	-0.03	<0.01		ns	1.66	<0.01	0.82	0.01	1.09	0.02		ns	-0.82	<0.01	-0.51	<0.01		ns	12.41	<0.01
T ²	-0.02	<0.01		ns	-0.7	0.01		Ns		ns		ns		ns	-0.16	0.03		ns		ns
C	-0.03	<0.01	-0.04	0.01	-0.37	0.03	-1.8	<0.01	0.68	0.04	4.08	<0.01		ns	0.36	<0.01	12.50	<0.01	7.47	0.01
C ²		ns	0.04	0.05		ns		Ns		ns		ns		ns	0.15	0.04		ns		ns
T X C	0.01	<0.01		ns	-0.32	0.02		Ns		ns		ns		ns				ns		ns
T X C ²		ns		ns	-0.45	0.03		Ns		ns		ns		ns				ns		ns
T ² X C		ns		ns	-0.39	0.04		Ns		ns		ns		ns				ns		ns
T ² X C ²	0.03	<0.01		ns		ns		Ns		ns		ns		ns				ns		ns
R ²	0.995		0.603		0.994		0.962		0.855		0.977		0.774		0.991		0.878		0.910	
R ² adj	0.991		0.504		0.984		0.952		0.819		0.975		0.742		0.983		0.861		0.880	
<i>p</i> (model)	<0.001		0.022		<0.001		0.012		<0.001		0.001		0.04		<0.01		<0.01		<0.01	
<i>p</i> (residuals)	0.43		0.62		0.25		0.22		0.04		0.82		0.571		0.465		0.332		0.767	

AG, Arabic gum; I, inulin; T, temperature (coded values); C, carrier concentration (coded values); Tg, Glass transition temperature; ns, not significant ($p < 0.05$).

Increases in inlet air temperature led to decreases in moisture contents of the samples (**Table 4**), due to higher heat transfer to the particle, resulting in a greater driving force for water evaporation, and thus producing powders with lower moisture content. Fazeli et al. (Fazaeli *et al.*, 2012), Frascareli et al. (Frascareli *et al.*, 2012), and Tonon et al. (Tonon, Brabet e Hubinger, 2008) also observed a decrease in moisture content with an increase in temperature of air inlet in the spray drying of black mulberry juice, coffee oil and acai juice, respectively.

Moreover, freeze-dried sample showed higher values of water activity (0.75) and moisture content (9.55%), in comparison to spray dried samples ($p < 0.05$), indicating lower microbiological stability and higher difficult in handling.

3.2 Higoscopycity

Hygroscopticity is the ability of a material to absorb moisture from the surrounding environment, and it is an important property to be considered in food processing due to the influence in food stability. Hygroscopticity of powders obtained using gum arabic as carrier ranged between 14.8 and 18.8 g of water absorbed per 100 g of sample, and for inulin as carrier between 13.8 and 19.4 g of water absorbed per 100 g of sample (**Tables 2 and 3**). Similar results were reported for spray dried of *Satureja montana* using maltodextrin as carrier agent (Vidović *et al.*, 2014). Addition of the carrier agents in any concentration leads to a significant decrease of the hygroscopticity compared to freeze drying ($p < 0.05$). This demonstrated the ability of carrier agents to improve the hygroscopticity values of fruit extract powders obtained from *E. dysenterica* and make them more stable.

Moreover, both temperature and concentration of carrier agent affected the hygroscopticity values of the powders obtained using gum arabic and inulin (**Table 4**). However, temperature was the variable that most affected these values in the samples obtained with gum arabic, consequently with an increase in drying temperature, an increase of the resulting powder hygroscopticity was observed (**Fig. 2a**). This can be explained because higher temperatures may form smaller particles, increasing the exposure area of the particles to the medium. These results are in accordance with the ones reported by Goula et al. (Goula, Adamopoulos e Kazakis, 2004). Meanwhile, the variable that most affected the hygroscopticity of samples obtained using inulin as wall material was the concentration of carrier, with the lowest values obtained for the highest concentration of inulin (**Fig. 2b**). These results are in agreement with those observed for powders of *Rhamnus purshiana*

obtained by spray drying, which showed a decrease in hygroscopicity due to an increase in concentration of carrier agent (Gallo *et al.*, 2011). It is important to note that for samples with low moisture contents the hygroscopicity values were higher. This relationship can be explained due to the fact that the capacity to adsorb ambient moisture is related to water concentration gradient between the product and the surrounding air (Tonon, Brabet e Hubinger, 2008).

In the present study, the powder with the lowest hygroscopicity was obtained at 120 °C and 30% of inulin. All samples showed low values of hygroscopicity compared to those reported for spray dried betacyanin extracts (45.4 to 49.2 g/100 g) using different temperatures and concentration of maltodextrin (Cai e Corke, 2000), and those obtained for spray-dried amla juices (46.03 to 53.01 g/100 g of sample) with maltodextrin as carrier agent (Mishra, Mishra e Mahanta, 2013).

Fig. 2. Contour plots for hygroscopicity response of powders obtaining with GA (a) and inulin (b).

3.3 Solubility

Solubility is an important feature of the powders that can potentially affect some of their properties, such as the reconstitution of the dry extract or the availability of the encapsulated compounds in a food system. Solubility values of spray dried *E. dysenterica* powders obtained at different temperatures and different concentrations of gum arabic and inulin are presented in **Tables 2** and **3**. Solubility values ranged from 94.4 to 97.8% for extracts obtained using gum arabic, and 87.7 to 95.9% for those obtained with inulin. Both carrier agents are highly soluble in water, therefore, as expected, solubility increased with the

addition of carrier agent in comparison to freeze dried sample (66.5%), thus indicating that the addition of carrier agents in any concentration significantly improved values of solubility ($p < 0.05$). The results for GA were expected, since this material is largely used as carrier in spray drying process due to its high solubility. However, the results for inulin proved that this is also a good carrier for spray drying process.

These results are similar to those reported for spray drying of sage using β -cyclodextrin, gum arabic and maltodextrin as carrier agents (Şahin-Nadeem *et al.*, 2013a), for guava powders using maltodextrin as carrier agent (Patil, Chauhan e Singh, 2014), and for spray-dried chlorophyllide with gum arabic, maltodextrin and soy protein isolate-based carrier systems (Comunian *et al.*, 2011). Solubility of powders from *E. dysenterica* were influenced by carrier concentration, an increase in the concentration of inulin resulted in higher solubility of samples. Moreover, the extracts obtained with gum arabic were also positively influenced by the inlet temperature (**Table 4**). Yousefi *et al.* (Yousefi, Emam-Djomeh e Mousavi, 2011) assessed the solubility of pomegranate juice powders obtained using different carrier agents (maltodextrin, gum arabic and waxy starch) and observed that solubility was strongly affected by carrier type and, in some cases, by concentration.

3.4 Glass transition temperature

A glass transition temperature can be defined as the temperature at which an amorphous system changes from a high viscosity glassy state to a lower viscosity rubbery state. Glass transition temperature is associated to powder stability because of its relation with molecular mobility. Hence, products stored below its T_g have their viscosity increased, hindering the mobility of molecules, while storage above T_g results in an increase in the molecular mobility, leading to inconveniences such as stickiness and caking (Palzer, 2005). Values of glass transition temperature of powders from *E. dysenterica* are summarized in **Tables 2** and **3**. A typical DSC thermogram obtained for a sample containing 20% of GA atomized at 120 °C is presented in **Figure 3**.

In general, a direct correlation between a_w and T_g of the powdered samples was observed for each inlet air temperature. **Tables 2** and **3** show that increases in a_w led to a decrease in T_g values of the powders. A similar behavior was reported for spray-dried casein using gum arabic as carrier agent, correlating glass transition temperatures with different water activities (Subtil *et al.*, 2012).

Fig. 3. DSC thermogram for spray dried *E. dysenterica* fruit extract with 20% of GA and inlet air temperature of 120°C.

Table 4 shows that the concentration of GA and inulin had a positive influence in the glass transition temperature of the samples. Moreover, the parameter that most influenced this response in treatments using inulin was temperature, as an increase in the inlet air temperature led to an increase in T_g of *E. dysenterica* powders (**Figure 4**). Similar results were reported for spray dried sweet potato using different concentrations of maltodextrin and different inlet air temperatures (Grabowski, Truong e Daubert, 2006). The increment of T_g values with increases in carrier concentration can be explained due to the fact that carriers have components with high molecular weight, which can decrease the molecular mobility.

Fig. 4. Contour plots for glass transition temperature response of powder obtaining with inulin.

3.5 Color

Lightness (L^*) of samples was influenced by both concentration of carrier agent and inlet air temperature ($p < 0.05$). In general, an increase in concentration of carrier agent resulted in an increase of lightness (**Tables 2 and 3**). According to Comunian *et al.* (Comunian *et al.*, 2011), these results are due to the dilution effect of the carriers which are all white powders.

The lowest values of lightness were observed in treatments at 160°C. This can be explained in two ways, first due to the fact that at a higher temperature the samples have lower moisture contents and therefore are more concentrated (Tonon, Brabet e Hubinger, 2009), and second, due to the Maillard reaction or caramelization of sugars, which is more likely at higher temperatures. The effect of different inlet air temperatures on color of spray-dried watermelon powders was previously assessed and a reduction in the value of L^* with an increase in inlet air temperature was observed (Quek, Chok e Swedlund, 2007).

Samples showed low positives values for parameter a^* , indicating a slight tendency to reddish. In the case of drying at 120 °C with 30% of gum arabic the value was negative, indicating a slight tendency to green. The carrier agents also presented negative values (**Tables 2 and 3**). Similar results were obtained for watermelon powders using maltodextrin (Quek, Chok e Swedlund, 2007). In general, the lowest values of parameter a^* were found at higher concentrations of carrier agent, because an increase in the ratio carrier-sample led to a dilution of material. Regarding parameter b^* all samples displayed positive values, indicating a tendency to yellow color. There were also differences in b^* values between the different temperatures ($p < 0.05$). The concentration of wall material only had effect on samples obtained with inulin: higher concentrations of the carrier resulted in a decrease in the values of b^* , associated to the lower values of b^* of the inulin. In general, an increase in temperature led to an increase in parameters a^* and b^* .

The freeze dried powder exhibited values for L^* , a^* and b^* of 57.88, 11.33 and 25.32 respectively, showing that this sample presented different color from spray-dried ones, with lower lightness, and tendency to green and yellow colors. This difference is due to the higher concentration of pigments and the absence of carrier in the freeze dried sample. These results are in agreement with those observed for powdered pigment obtained from vinification byproducts of the Bordo grape (*Vitis labrusca*) obtained by spray drying and freeze drying (De Souza *et al.* 2015).

3.6 Morphological characterization of the particles

Scanning electron micrographs of samples obtained using gum arabic displayed irregularly spherical shaped particles, with many shrinkages and dents on its surface (**Fig. 5a**). These results are consistent with those observed for instant soluble sage obtained by spray-dried using gum arabic as carrier agent (Şahin-Nadeem *et al.*, 2013). In the case of samples obtained with inulin, the micrographs showed particles with corrugated surface and a more spherical shape than that of those prepared with gum arabic (**Fig. 5b**). These differences in shape can be explained due to the different characteristics of the carrier material used in the spray drying of *E. dysenterica* powders (**Figs. 5c and 5d**). Another difference observed in the spray-dried samples is that some particles obtained at 140 °C and 160 °C were smooth and presented a hole in the surface, phenomenon caused because at higher temperatures the vapor pressure of water increases, leading to a breakage of the film or crust. For treatments at lower temperatures most of the water can escape through the cortex by diffusion, thus avoiding the breakage of the crust (Maas *et al.*, 2011).

Fig. 4. Micrographs of *E. dysenterica* powders (2000x) except for C (3000x); A, 160°C and 30% of GA; B, 140°C and 30% of Inulin; C, GA; D, Inulin.

4. CONCLUSION

The results of this study indicated that the use of gum arabic and inulin on obtaining dry extracts from *E. dysenterica* by the spray drying technique had high process yield and high stability of powders due to the low moisture content and low water activity, as well as high glass transition temperatures and low hygroscopicity. Additionally, the powders obtained had a high solubility in water, characteristic associated with a good reconstitution of the product. In addition, the process temperatures and concentration of carrier agents evaluated in this study influenced the physical properties of the powders. Therefore, the use of spray drying to obtain dry extracts from cagaita can be an alternative to reduce transport costs, prevent deterioration and ensure the quality and availability of the fruit.

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**Stability of phenolic compounds and functional properties of encapsulated cagaita
(*Eugenia dysenterica* DC.) fruit extract**

ABSTRACT AND KEYWORDS PAGE

Cagaita (*Eugenia dysenterica* DC., Myrtaceae) is an important Brazilian native fruitful species with potential health effects associated to unique profile and elevated concentration of polyphenols. The aim of this work was to evaluate the total phenolic (TPC), and proanthocyanidins contents, and the functional (antioxidant, antimicrobial, and anti-obesity) potential of cagaita encapsulated powders. Spray-dried extracts were produced with different carrier agents, namely gum arabic and inulin, at different drying conditions (concentration of carrier agent and inlet air temperature). *E. dysenterica* powder extracts presented values of TPC between 9.9 and 31.2 mg GAE/g DW, keeping a high correlation with the antioxidant capacity (between $r = 0.90$ and $r = 0.98$). Moreover, powders presented inhibitory activity against *Staphylococcus aureus* and *Listeria monocytogenes*, similar to the antibiotics used as positive control, and also against α -amylase and α -glucosidase. Gum arabic was the more suitable material to stabilize the extracts and prevent the loss of TPC. The use of spray-drying to dry and encapsulate extracts from cagaita can be an alternative to prevent deterioration of the bioactive compounds present in the fruit, ensuring quality and availability of the fruit during off-season.

Keywords: Myrtaceae, encapsulation, functional foods, phenolic compounds, antioxidant capacity.

1. INTRODUCTION

Brazil is the country with the highest biodiversity in the world and is the third largest producer of fruits (FAO, 2013). Concerning the fruit production, there are various native fruits with interesting potential for food and pharmaceutical industries for being excellent sources of phenolic bioactive compounds. Cagaita or “cagaiteira” (*Eugenia dysenterica* DC.) is an important native fruitful species belonging to the family of Myrtaceae and is native of Brazilian Cerrado region. The fruit is consumed “in natura” and used in various typical preparations, mainly jellies, ice-cream and juices. Despite the alleged medicinal properties of the fruits, the use is limited to the local population due to the high perishability and high transport costs.

Fruits of *E. dysenterica* are considered a good source of polyphenols, mainly flavonoids (Genovese, Pinto, Gonçalves, & Lajolo, 2008). Different activities have been associated with the presence of polyphenolic compounds in foods, such as antioxidant (Zhang et al., 2015), antimicrobial (Kołodziejczyk et al., 2013), and anti-obesity due to inhibition of key enzymes in the metabolism of carbohydrates such as α -glucosidase and α -amylase (De Souza Schmidt Gonçalves, Lajolo, & Genovese, 2010). Thus, the presence of phenolic compounds on cagaita makes this fruit attractive for potential therapeutic use. However, phenolic compounds are susceptible to degradation and can be affected by several factors, such pH, temperature, exposure to light, oxygen, and storage conditions (Grace et al., 2014; Selcuk & Erkan, 2015).

As a consequence of the limited use of the cagaita fruit and the susceptibility of the phenolic compound to degradation, production of powders from extracts obtained from *E. dysenterica* is an alternative to overcome these limiting factors. Spray-drying is a technique by which a liquid product is atomized in a hot gas current to instantaneously obtain a powder (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). Spray-drying has been successfully used for encapsulation of different components that are susceptible to degradation (Souza et al., 2014). The selection of the drying additives depends on the desired properties and the final application of the product. Gum arabic is one of the most common material used for microencapsulation due to its high solubility, low viscosity and film forming properties (Sarkar, Gupta, Variyar, Sharma, & Singhal, 2013). However, limited information is available on the use of inulin as carrier agent. A common feature between these two carrier agents is that both are not hydrolyzed in the stomach and the small intestine (Ali, Ziada, & Blunden, 2009; Stevens, Meriggi, & Booten, 2001), being fermented in the large intestine by

microflora, which avoids the increase in the glycemic index, and makes them potential ingredients for foods destined to diabetics.

The aim of this study was to evaluate and compare spray-dried cagaita extracts, using gum arabic and inulin as carrier agents, in relation to functional properties (TPC, stability of phenolic compounds during storage, “in vitro” antioxidant capacity, antimicrobial activity and the capacity to inhibit the key enzymes of carbohydrates metabolism).

2. MATERIALS AND METHODS

2.1 Materials

Frozen commercial pulp of cagaita (*Eugenia dysenterica* DC.) was purchased from Cooperativa dos Agricultores Familiares Agroextrativistas Grande Sertão (Montes Claros, MG, Brazil). The carrier agents used in this work were food grade inulin extracted from chicory (SIBA ingredients Com. Ltd, Brazil) and gum arabic (Nexira Brazil Com. Ltd., Brazil).

2.2 Preparation and encapsulation of cagaita extract

The extract of cagaita pulp was prepared using aqueous ethanol (79%, v/v) in a ratio of 1:20 (w/v) for 3 min at 14000 rpm using a homogenizer (Ultra-Turrax T25; IKA, Germany). The extract was then separated from the sample residue by centrifugation (5,640 g for 10 min). Extraction of residue was repeated twice. The resultant extracts were pooled and concentrated at 40 °C in a rotatory evaporator (BUCHI, B-491) until complete elimination of ethanol. After that, the extract was mixed with different concentrations (10%, 20% and 30% w/v) of the carrier agent (inulin or gum arabic), and then atomized with a spray-dryer (model MSD 1.0, Labmaq, Brazil) at different temperatures (120 °C, 140 °C and 160 °C) by a peristaltic pump at a fixed rate of 0.60 m³/min. The formulation was mixed continuously by a magnetic stirrer during the entire process of spray-drying.

2.3 Phenolic compounds and antioxidant capacity

2.3.1 Sample extraction

The spray-dried powders (0.5 g) were extracted with 70% of aqueous methanol (20 mL) using a homogenizer (Ultra-Turrax T25; IKA, Germany) for 3 min at 8,000 rpm. The homogenized extract was centrifuged for 10 min at 5,640 g, and the supernatant was

concentrated and kept at -18°C until subsequent analysis. All extractions were performed in duplicate, and the subsequent assays were run in triplicate.

2.3.2 Total phenolics content

The determination was carried out according to (Singleton, Orthofer, & Lamuela-Raventos, 1999), with some modifications. A 0.25 mL aliquot of the extract was mixed with 0.25 mL of the Folin-Ciocalteu reagent and 2 mL of distilled water. After 3 min at room temperature, 0.25 mL of sodium carbonate (Na_2CO_3) solution were added, and the mixture placed in a water bath for 30 min at 37°C. The absorbance was read at 750 nm in a U1100 UV/Visible spectrophotometer (Hitachi, Japan). The results were expressed as mg of gallic acid equivalents per gram of sample in dry weight basis (mg GAE/g DW).

2.3.3 Total proanthocyanidins content.

The total content of proanthocyanidins was determined according to (Porter, Hrstich, & Chan, 1985), using a reagent comprised of 154 mg of $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ per liter of *n*-butanol:hydrochloric acid (3:2). A total of 250 μL of each sample extract and 2.5 mL of the described reagent were incubated at 90 °C for 15 min. The blank consisted of 2.5 mL of the reagent and 250 μL of methanol:acetic acid (99.5:0.5). The absorbance was measured at 540 nm using a U1100 UV/Visible spectrophotometer (Hitachi, Japan) and results were expressed as mg equivalent of quebracho tannin/g DW.

2.3.4 HPLC-DAD analysis of flavonoids

Extraction was performed according to (Arabbi, Genovese, & Lajolo, 2004). The identification and quantification of the main flavonoids were conducted using a Prodigy ODS3 reversed phase silica column (5 μm , 250 $\times$ 4.6 mm, Phenomenex Ltd, Torrance, CA), in an analytical reversed-phase HPLC HewlettPackard 1100 system (automatic sample injector, quaternary pump and diode array detector (DAD) controlled by the ChemStation software (Hewlett-Packard, Palo Alto, CA, USA) as described previously by Arabbi, Genovese, and Lajolo (2004). The samples were injected in triplicate and the phenolic compounds were identified by comparing the retention time and spectra with appropriate standards.

2.3.5 DPPH radical-scavenging ability.

The antioxidant capacity was determined by the DPPH• (2,2-diphenyl-1-picrylhydrazyl) radical-scavenging method according to (Brand-Williams, Cuvelier, & Berset, 1995), with some modifications. An aliquot (50 µL) of the extract, previously diluted, was added to 250 µL of a methanolic solution of DPPH• (0.5 mM) and shaken. After 25 minutes at 25 °C, the absorbance was measured at 517 nm using a microplate spectrophotometer (Synergy H1 hybrid reader, Biotek, IL, U.S). A calibration curve was prepared using Trolox (Sigma-Aldrich) at different concentrations (20 - 80 µM). Results were expressed as µmol of trolox equivalent TE/g DW.

2.3.6 Ferric reducing antioxidant power (FRAP).

The analysis was performed at pH 3.6 according to (Benzie & Strain, 1996). The absorbance was measured at 593 nm using a microplate spectrophotometer (Synergy H1 hybrid reader, Biotek, IL, U.S), using an aqueous solution of Trolox (0.25 mg/mL), at different concentrations (0.1 - 0.8 mM), as control. The ferric reducing power was expressed as µmol of TE/g DW.

2.3.7 Oxygen Radical Absorbance Capacity Assay (ORAC).

The determination was carried out according to the method modified by (Dávalos, Gómez-Cordovés, & Bartolomé, 2004). The extract (20 µL) and a solution of fluorescein (FL; 120 µL; 70 nM, final concentration) in 75 mM phosphate buffer (pH 7.4), were mixed in the well of a microplate. The mixture was pre-incubated for 15 min at 37 °C, and then 2,2'-azobis(2-methylpropionamidine) dihydrochloride (AAPH) solution (60 µL; 12 mM, final concentration) was added. The fluorescence was recorded every minute for 80 min, and the microplate shaken before each read. A blank (FL + AAPH), using phosphate buffer instead of extract, and a standard curve of Trolox at different concentrations (1-8 µM, final concentration), were also carried out in each assay. The activity was determined using the net area under curve of sample with respect to the blank, calculated by the Gen5 Data Analysis Software (Biotek, IL, U.S). Results were expressed as Trolox equivalents by using the standard curve calculated for each assay (µg TE/g DW).

2.4 TPC stability during storage.

Cagaita fruit powders were stored at 30 °C in desiccators containing a MgCl₂ saturated solution (32.8% relative humidity) and samples were analyzed each 20 days until the total

storage time of 120 days. The TPC stability was determined by the degradation of the compounds during storage according to the method modified by (Souza et al., 2014). The first-order reaction rate constants (k) and half-life time ($t_{1/2}$) were calculated as described in the Equations (1) and (2):

$$\ln(C_t/C_0) = -kt \quad (1)$$

$$t_{1/2} = \ln 2/k \quad (2)$$

Where C_0 is the initial TPC and C_t is the TPC at the reaction time t . The total loss of the total phenolic content was calculated using Equation 3:

$$\text{Total loss \%} = [1 - (C_{120}/C_0)] \times 100 \quad (3)$$

2.5 Enzymes assays

2.5.1 α -Amylase inhibition assay.

The inhibitory activity of α -amylase was determined according to (Worthington, 1993a). A 500 μ L aliquot of the extract at different concentration was mixed with 500 μ L of 0.02 M sodium phosphate buffer solution at (pH 6.9 with 0.006 M NaCl) containing the enzyme (0.5 mg/mL of α -Amylase from porcine pancreas, Type VI-B, Sigma-Aldrich) and pre-incubated for 10 min at 25 °C. After pre-incubation, 500 μ L of the starch solution were added (1% in 0.02 M sodium phosphate buffer; pH 6.9 containing 0.006 M of NaCl). The mixture was then incubated for 10 min at 25 °C. A 1 mL aliquot of dinitrosalicylic acid was added in each tube to stop the reaction. The mixture was then incubated at 100 °C for 5 min. The reaction mixture was then cooled and diluted with distilled water, and the absorbance was measured at 540 nm. A sample blank (buffer instead of enzyme solution) and control (buffer in place of sample extract) were analyzed in parallel to the test samples. The α -glucosidase inhibitory activity was calculated as percent (eq. 4) and expressed as IC_{50} in mg of powder per milliliter of reaction, and compared to acarbose (Sigma-Aldrich).

$$\% \text{ Inhibition} = [(A_{540\text{control}} - A_{540\text{extract}}) / A_{540\text{control}}] \times 100 \quad (4)$$

2.5.2 α -glucosidase inhibition assay.

Inhibition of the α -glucosidase was determined according to (Worthington, 1993b) with some modifications (Mccue, KWON, & Shetty, 2005). A 50 μ L aliquot of the extracts, at different concentrations, was diluted with 50 μ L of buffer phosphate (0.1 M; pH 6.9) and 100 μ L α -glucosidase solution (1.0 U/mL, of 0.1 M buffer phosphate, pH 6.9, α -Glucosidase from *Saccharomyces cerevisiae*, Type I, Sigma-Aldrich), were mixed in a 96-well microplate, and

then pre-incubated at 25°C. After pre-incubation, 50 µL of 5 mM of *p*-nitrophenyl- α -D-glucopyranoside (Sigma-Aldrich) solution, prepared in the same buffer, were added. The reaction mixtures were done at 25°C for 5 min. The absorbance was measured before and after the incubation at 405 nm using a microplate spectrophotometer (Synergy H1 hybrid reader, Biotek, IL, U.S). Absorbance was compared to the control which contained buffer instead of the sample. The capacity of inhibition of the extracts was calculated as percent (eq. 5) and expressed as IC₅₀ in mg of powder per milliliter of reaction, and compared to acarbose.

$$\% \text{ Inhibition} = \frac{[(\text{Abs}^{\text{control5min}} - \text{Abs}^{\text{control0min}}) - (\text{Abs}^{\text{extract5min}} - \text{Abs}^{\text{extract0min}})]}{(\text{Abs}^{\text{control5min}} - \text{Abs}^{\text{control0min}})} \times 100 \quad (5)$$

2.6 Antimicrobial activity and determination of Minimum Inhibitory Concentration (MIC)

The powder extracts were tested for activity against *Staphylococcus aureus* ATCC 29213, *Escherichia coli* ATCC 8739, *Enterobacter aerogenes* ATCC 13048, *Listeria monocytogenes* ATCC 7644, *Salmonella typhimurium* ATCC 14028 and *Salmonella enteritidis* ATCC 13076, according to CLSI (2009). The cultures were grown in Tryptic Soy Agar (Oxoid, Basingstoke, UK) for 18–24 h at 37 °C, and the colonies were suspended in sterile saline solution (0.85%) to reach a turbidity correspondent to 0.5 of the McFarland scale (10⁸ UFC mL⁻¹). The suspensions (0.1 mL) were applied to the surface of Muller–Hinton agar plates (Oxoid, Basingstoke, UK). An aliquot of powder extracts (30 µL) was pipetted onto sterile paper discs (6 mm diameter, Oxoid) and placed onto the surface of inoculated agar plates, which were incubated at 37 °C for 24 h. The diameters of the inhibition zones were measured using a caliper rule. Erythromycin (15 µg), Vancomycin (5 µg) and Ceftriaxone (X µg) (Oxoid, Basingstoke, UK) were used as a positive control.

For determination of the MIC, the micro dilution method was used (CLSI, 2010). Except for the first row, the wells of sterile 96-well microplates were filled with 50 µL of Muller–Hinton broth (Oxoid, Basingstoke, UK). The wells of the first row of were filled with 100 µL of the extracts, homogenized, and then 50 µL was serially transferred to the subsequent wells. The microplates were incubated at 37 °C for 24 h and the MIC, correspondent to the lowest concentration that inhibited the visible growth of the microorganism after 24 h.

2.7 Statistical analysis

The results were presented as mean $\pm$ standard deviation (SD). Differences between means were first analyzed by one-way ANOVA test and the Tukey test was used to compare the means and p values below 0.05 were considered significant. Prior to one-way ANOVA application, experimental results were formally checked for normality (Anderson-Darling test) and homoscedasticity (Brown-Forsythe test). Correlation analysis based on the Pearson's correlation coefficient was calculated between responses (Granato, Calado, & Jarvis, 2014). All statistical procedures were performed using the Statistica software package v. 11 (StatSoft, Inc., Tulsa, OK, USA).

3. RESULTS AND DISCUSSION

3.1 Phenolic contents and antioxidant capacity of cagaita powders

The TPC ranged between 11 and 31 mg GAE/g DW for samples spray-dried using gum arabic, and between 10 and 29 mg GAE/g DW for samples with inulin as carrier agent. Proanthocyanidins content was 6 – 16 mg ETQ/g DW for GA powders, and 7 – 14 mg ETQ/g DW for powders with inulin, the highest content of proanthocyanidins was observed in the powder obtained using 10% of GA and inlet air temperature of 160°C (**Tables 1 and 2**). As expected, the freeze dried sample presented the highest content of phenolic compounds (98 mg GAE/g DW) and proanthocyanidins (42 mgTQE/g DW) (**Tables 1 and 2**), as there was no dilution effect due to the absence of carrier agent, the loss of extractability was less intense (Fujita, Borges, Correia, de Melo Franco, & Genovese, 2013). Total phenolics and proanthocyanidins content was affect by the dilution effect of carrier agent, and all powders obtained using 30% of GA or inulin presented the lowest concentrations of bioactive compounds.

In comparison to other processed fruits and food materials, powders of cagaita presented much higher content of phenolic compounds than powders of bayberry juice (3 mg GAE/g DW) (Fang & Bhandari, 2011), and spray-dried sumac extract (1 mg GAE/g DW) (Caliskan & Dirim, 2013), but lower compared to powders of pomegranate peel (95 to 105 mg GAE/g) (Çam, İçyer, & Erdoğan, 2014).

Fig. 1 shows the chromatographic profile (270 nm) of phenolic compounds extracted from *E. dysenterica* powder extract. Three peaks corresponding to quercetin derivatives were common to all samples and were the most abundant in the powders. As expected, the content

of quercetin derivatives showed an inverse correlation with the carrier agent concentration. Freeze dried sample presented the highest content of quercetin derivatives (**Tables 1 and 2**).

Powders showed a high retention of bioactive compounds, in the range of 77.9 to 99.6% in the case of total phenolic content, and between of 62.7 to 98.7% for proanthocyanidins. The highest retentions were observed in samples with the highest concentrations of carrier agents, and these results are similar to those obtained for the freeze dried sample (**Table 2 and 3**). Similar results were reported for retention of phenolic compounds and anthocyanidins in spray dried bayberry juice (96% and 94%, respectively), using maltodextrin as carrier agent and inlet air temperature of 150°C (Fang & Bhandari, 2012). In general, inlet air temperature did not affect the retention of bioactive compounds in the powders, which can be explained due to the low residence time of the powder in the drying chamber.

Other authors have also evaluated the impact of the spray drying on phenolic compounds. (Peng, Li, Guan, & Zhao, 2013) obtained higher retention of anthocyanins, flavonoids and total phenolics, and a protective effect for these compounds using maltodextrin as a carrier agent in spray drying of purple sweet potato flours. Anthocyanin degradation in the spray dried açai juice obtained using different carrier agents was reported by (Tonon, Brabet, & Hubinger, 2010), who observed that the temperature influenced negatively the stability of these compounds, due to high sensitivity of the pigments to heat, and that maltodextrin 10DE and gum arabic were the carrier agents that produced particles with the better half-lives of anthocyanins.

Table 1. Total phenolics, proanthocyanidins, and quercetin contents and *in vitro* antioxidant activity of spray-dried powders of cagaita obtained using gum arabic as carrier agent.

Sample	Total phenolic content (GAE mg/g DW)	TPC retention (%)	Total proanthocyanidins (TP/TQE mg/g DW)	TP retention (%)	Quercetin (µg/g)	DPPH (µmol Trolox/g DW)	FRAP (µmol Trolox/g DW)	ORAC (µmol Trolox/g DW)
1	27.9±1.9 ^c	88.0±10.9 ^a	12.7±3.0 ^c	87.9±19.4 ^{ab}	49.8±3.3 ^b	36.5±1.2 ^b	159.9±3.4 ^b	83.7±0.8 ^{b,c}
2	16.3±0.9 ^d	98.4±5.4 ^a	9.0±0.6 ^{d,e,f}	93.7±6.8 ^c	27.8±1.6 ^c	17.3±0.4 ^c	79.4±0.4 ^d	54.9±0.8 ^{d,e}
3	10.8±0.8 ^e	99.2±7.7 ^a	6.2±0.5 ^g	93.1±8.2 ^c	15.0±0.8 ^d	11.1±0.3 ^d	50.9±4.5 ^e	44.9±3.4 ^{e,f}
4	28.3±1.3 ^c	92.3±9.9 ^a	12.9±0.6 ^{b,c}	72.4±3.5 ^a	49.9±2.6 ^b	35.5±1.9 ^b	159.3±3.9 ^b	85.4±2.6 ^b
5	16.8±0.8 ^d	98.9±4.7 ^a	9.9±0.7 ^{d,e}	96.6±7.7 ^c	28.6±1.8 ^c	18.4±0.6 ^c	82.4±1.8 ^d	60.5±1.2 ^{d,e}
6	11.6±0.7 ^e	99.1±5.8 ^a	7.0±0.8 ^{f,g}	96.7±11.1 ^c	16.9±0.8 ^d	12.4±0.3 ^d	53.7±2.6 ^e	37.2±0.8 ^g
7	31.2±2.8 ^b	91.8±8.3 ^a	15.6±0.7 ^b	89.2±4.4 ^b	47.7±1.8 ^b	34.0±1.1 ^b	158.1±4.6 ^b	99.5±2.9 ^b
8	16.8±1.6 ^d	99.6±12.1 ^a	10.8±0.2 ^{c,d}	95.6±15.2 ^c	27.7±1.8 ^c	18.5±1.1 ^c	99.2±4.6 ^c	67.3±1.4 ^{c,d}
9	11.0±0.7 ^e	98.5±6.0 ^a	7.8±0.5 ^{e,f,g}	98.2±6.6 ^c	17.6±1.7 ^d	11.3±0.4 ^d	53.6±1.2 ^e	47.5±3.2 ^{e,f}
Freeze-dried	95.9±1.1 ^a	94.0±2.3	41.7±5.4 ^a	96.8±12.7	144.1±7.1 ^a	138.3±4.7 ^a	357.8±3.9 ^a	272.8±13.6 ^a

GAE, gallic acid equivalent; DW, dry weigh; TQE, tannin quebracho equivalent; CA, carrier agent; Samples: 1, 120 °C/10% CA; 2, 120 °C/20% CA; 3, 120 °C/30% CA; 4, 140 °C/10% CA; 5, 140 °C/20% CA; 6, 140 °C/30% CA; 7, 160 °C/10% CA; 8, 160 °C/20% CA; 9, 160 °C/30% CA. Values are expressed as means ± SD (n=3). Different superscripts letters in the same column mean significantly differences ($p < 0.05$).

Table 2. Total phenolics, proanthocyanidins and quercetin contents and *in vitro* antioxidant activity of spray-dried powders of cagaita obtained using inulin as carrier agent.

Sample	Total phenolic content (GAE mg/g DW)	TPC retention (%)	Total proanthocyanidins (TQE mg/g DW)	TP retention (%)	Quercetin ($\mu\text{g/g}$)	DPPH (mmol Trolox/g DW)	FRAP ($\mu\text{mol Trolox/g DW}$)	ORAC (mmol Trolox/g DW)
1	24.2 $\pm$ 3.1 ^c	77.9 $\pm$ 9.9 ^a	11.4 $\pm$ 0.3 ^c	62.7 $\pm$ 2.1 ^a	37.6 $\pm$ 2.0 ^b	30.7 $\pm$ 1.2 ^b	140.3 $\pm$ 2.4 ^c	85.1 $\pm$ 5.6 ^b
2	15.4 $\pm$ 0.8 ^d	90.7 $\pm$ 5.1 ^b	9.9 $\pm$ 0.4 ^d	90.9 $\pm$ 4.1 ^c	18.4 $\pm$ 1.2 ^c	16.7 $\pm$ 1.0 ^{e,f}	81.3 $\pm$ 0.9 ^f	55.3 $\pm$ 2.9 ^{c,d}
3	10.1 $\pm$ 0.4 ^e	94.9 $\pm$ 3.6	6.7 $\pm$ 0.3 ^e	98.7 $\pm$ 4.6 ^d	10.8 $\pm$ 0.6 ^d	10.6 $\pm$ 0.8 ^{g,h}	62.1 $\pm$ 4.0 ^g	43.8 $\pm$ 1.6 ^{c,d}
4	27.7 $\pm$ 0.7 ^b	88.4 $\pm$ 2.2 ^a	12.0 $\pm$ 0.5 ^c	65.6 $\pm$ 2.5 ^a	41.9 $\pm$ 2.9 ^b	30.9 $\pm$ 1.1 ^b	108.7 $\pm$ 1.9 ^d	89.3 $\pm$ 4.7 ^b
5	16.7 $\pm$ 0.5 ^d	93.3 $\pm$ 2.7 ^b	9.3 $\pm$ 0.2 ^d	88.2 $\pm$ 1.7 ^c	23.7 $\pm$ 1.3 ^c	19.9 $\pm$ 0.8 ^{d,e}	84.7 $\pm$ 5.9 ^{e,f}	60.5 $\pm$ 6.0 ^d
6	10.4 $\pm$ 0.4 ^e	97.7 $\pm$ 4.4 ^b	7.3 $\pm$ 0.1 ^e	93.3 $\pm$ 1.1 ^d	11.8 $\pm$ 0.5 ^d	8.5 $\pm$ 0.5 ^h	59.4 $\pm$ 1.1 ^g	39.5 $\pm$ 3.7 ^a
7	29.1 $\pm$ 2.9 ^b	93.2 $\pm$ 2.9 ^b	13.6 $\pm$ 0.4 ^b	74.4 $\pm$ 2.2 ^b	42.0 $\pm$ 1.9 ^b	27.2 $\pm$ 0.8 ^c	152.6 $\pm$ 4.3 ^b	94.0 $\pm$ 1.2 ^b
8	17.7 $\pm$ 2.4 ^d	96.9 $\pm$ 14.1 ^b	11.7 $\pm$ 0.5 ^c	87.3 $\pm$ 3.7 ^c	23.7 $\pm$ 1.6 ^c	21.2 $\pm$ 0.8 ^d	92.7 $\pm$ 1.7 ^e	62.1 $\pm$ 3.6 ^c
9	9.9 $\pm$ 0.6 ^e	98.1 $\pm$ 6.2 ^b	7.0 $\pm$ 0.2 ^e	96.4 $\pm$ 2.9 ^d	13.8 $\pm$ 0.5 ^d	13.9 $\pm$ 0.7 ^{f,g}	77.4 $\pm$ 1.6 ^f	42.9 $\pm$ 2.9 ^{c,d}
Freeze-dried	95.9 $\pm$ 1.1 ^a	94.0 $\pm$ 2.3	41.7 $\pm$ 5.4 ^a	96.8 $\pm$ 12.7	144.1 $\pm$ 7.1 ^a	138.3 $\pm$ 4.7 ^a	357.8 $\pm$ 3.9 ^a	272.8 $\pm$ 13.6 ^a

GAE, gallic acid equivalent; DW, dry weigh; TQE, tannin quebracho equivalent; CA, carrier agent; Samples: 1, 120 °C/10% CA; 2, 120 °C/20% CA; 3, 120 °C/30% CA; 4, 140 °C/10% CA; 5, 140 °C/20% CA; 6, 140 °C/30% CA; 7, 160 °C/10% CA; 8, 160 °C/20% CA; 9, 160 °C/30% CA. Values are expressed as means $\pm$ SD (n=3). Different superscripts letters in the same column mean significantly differences ($p < 0.05$).

Antioxidant capacities of the spray-dried extracts of *E. dysenterica* were assessed by DPPH, FRAP and ORAC assays (**Tables 1** and **2**). A decrease in the antioxidant capacities was observed with an increase in concentration of carrier agent, similar to previous reports (Silva et al., 2014; Souza et al., 2014; Vidović, Vladić, Vaštag, Zeković, & Popović, 2014). Interesting to note that inlet air temperature did not show a significant influence on the antioxidant activity.

Fig. 1. HPLC chromatogram (270 nm) of flavonoid glycosides from spray-dried cagita powder obtained at 120 °C with 10% of gum arabic as carrier agent.

Spray-dried powders displayed low to middle antioxidant activity, in the range of 5.8% to 26.5% for DPPH, 13.9% to 43.1% for FRAP, and 13.6% to 36.5% for ORAC, compared to the freeze dried sample, following the same behavior observed for phenolic content. Compared to other reports, the antioxidant capacity of *E. dysenterica* powder extracts are similar to those of spray-dried Bordô grapes residues, assessed by DPPH and FRAP assays (Souza et al., 2014), but lower than that reported for depulping residue of jabuticaba assessed by DPPH and ORAC assays (Silva et al., 2014).

It was observed a high and significant correlation ($p < 0.05$) between antioxidant capacity and total phenolic content of powders. Correlation coefficients values for total phenolic content with respect to the antioxidant capacity of samples obtained using gum arabic and inulin were $r = 0.98$ and $r = 0.92$; $r = 0.99$ and $r = 0.90$; $r = 0.97$ and $r = 0.99$ ($p < 0.05$) for DPPH, FRAP and ORAC assays, respectively. Correlation coefficients for antioxidant capacity for samples dried with gum arabic and inulin were $r = 0.91$ and $r = 0.84$; $r = 0.94$ and $r = 0.86$; $r = 0.98$ and $r = 0.92$ ($p < 0.05$) for DPPH, FRAP and ORAC assays respectively with respect to proanthocyanidins content. These results are in accordance to those reported by (Peng et al., 2013) who found a high positive correlation among

flavonoids/total phenolics content and the antioxidant capacity of purple sweet potato flours obtained by spray-drying.

3.2 Phenolic stability during storage of powders

There was a significant loss of phenolic compounds during storage; the decrease in total phenolic content was greater than 25% in all samples at the end of the storage at 30 °C, 32.8% relative humidity, for 120 days. The powders obtained with inulin showed the highest losses, with values up to 50% (**Table 3**). It is important to note that it was not possible to measure the loss of TPC in the freeze-dried control either in powders with 10% of inulin. The samples presented agglomeration, which can be associated to the rapid adsorption of water in the first days of storage.

Regarding spray-drying conditions, losses in TPC exhibited an inverse correlation with the concentration of carrier agent. An increase in the proportion of carrier agent protected phenolics during storage due to a restriction of the molecular mobility as the result of reduced hygroscopicity and increased viscosity of the powders. Similar results were reported for anthocyanins in spray-dried bordo grape powders (Souza et al., 2014). Meanwhile, spray-drying temperature did not influence TPC losses.

Phenolics degradation in all samples presented a first-order kinetic behavior. Lower rates constant values (k) were observed in samples obtained with gum arabic as carrier agent, indicating that the rate of conversion or degradation of phenolic compound was less intense than that for powders obtained using inulin as carrier agent. As expected, the half-life of phenolic compounds was directly proportional to the carrier agent concentration.

3.3 α -amylase and α -glucosidase inhibitory activity

α -amylase and α -glucosidase are key enzymes in the metabolism of carbohydrates. Therefore, products with the capacity to inhibit these enzymes can be attractive for the control of postprandial hyperglycemia by reducing carbohydrate metabolism (Hanhineva et al., 2010). In this work, the capacity to inhibit these enzymes by spray-dried powders from cagaita fruit was measured.

Table 3. Total loss of total phenolic content (TPC) and kinetics parameter of spray-dried cagaita powders during storage at 25 °C during 120 days.

Simple	Gum arabic				Inulin			
	Total loss of TPC (%)	k (days ⁻¹)	$t_{1/2}$ (days)	R^2	Total loss of TPC (%)	k (days ⁻¹)	$t_{1/2}$ (days)	R^2
1	29.4±2.9 ^{a,b}	0.00290	238.6	0.904	ne	ne	Ne	ne
2	31.3±3.4 ^{a,b}	0.00313	221.4	0.952	50.9±0.4 ^{a,b}	0.00593	116.9	0.972
3	26.2±2.3 ^b	0.00253	274.3	0.897	35.9±2.6 ^d	0.00371	186.6	0.939
4	31.1±2.0 ^{a,b}	0.00311	222.8	0.949	ne	ne	ne	ne
5	30.5±1.2 ^{a,b}	0.00303	228.9	0.947	55.7±3.6 ^a	0.00679	102.1	0.947
6	26.5±3.1 ^b	0.00257	270.0	0.970	47.1±1.2 ^b	0.00530	130.7	0.998
7	35.3±2.3 ^a	0.00363	190.9	0.949	ne	ne	ne	ne
8	26.7±1.2 ^b	0.00259	267.4	0.937	44.9±0.4 ^{b,c}	0.00497	139.3	0.958
9	28.7±2.0 ^{a,b}	0.00282	246.1	0.860	39.9±1.2 ^{c,d}	0.00423	163.5	0.961

TPC, total phenolic content; k , Reaction rate constant; $t_{1/2}$, half-life time; CA, carrier agent; ne, not estimated; Samples: 1, 120 °C / 10% CA; 2, 120 °C / 20% CA; 3, 120 °C / 30% CA; 4, 140 °C / 10% CA; 5, 140 °C / 20% CA; 6, 140 °C / 30% CA; 7, 160 °C / 10% CA; 8, 160 °C / 20% CA; 9, 160 °C / 30% CA. Values are expressed as means ± SD (n=3). Different superscripts letters in the same column mean significantly differences ($p < 0.05$).

The IC_{50} values for the inhibition of α -amylase were in the range of 10.6 to 106.6 mg/mL and 9.8 to 99.5 mg/mL, for powders obtained with gum arabic and inulin respectively (**Fig. 2**). The powders presented lower inhibition capacity when compared to the control acarbose (IC_{50} 0.013 mg/mL). The samples dried with 10% of carrier agent were the most active, however, the freeze-dried sample was more active (IC_{50} 2.5 mg/mL). As shown Fig. 2, higher concentration of carrier agent decreased the effectiveness of the powder to inhibit the enzyme ($p < 0.05$). Nevertheless, the influence of the temperature in the activity of the powders was no significant.

All the powder extracts were active against α -glucosidase with IC_{50} values between 5.7 mg/mL to 159.1 mg/mL and 13.2 mg/mL to 154.5 mg/mL for powders with gum arabic and inulin as carrier agents, respectively (**Fig. 2**). Likewise the antioxidant activity, the effect of carrier agent influenced the inhibition response of the powders: more active extracts were, in both cases, those obtained with 10% of carrier agent and therefore those with higher content of phenolic compounds. In comparison to the positive control acarbose (IC_{50} 0.11 mg/mL), the powders presented much lower activity against α -glucosidase. However, the freeze-dried extract sample presented a higher inhibitory activity than acarbose (IC_{50} 0.07 mg/mL), which was expected due to the lack of carrier agent.

The IC_{50} for α -amylase and α -glucosidase inhibition showed an inverse correlation to the total phenolic content (values for α -amylase $r = -0.92$ and $r = -0.90$; α -glucosidase $r = -0.87$ and $r = -0.86$, for powders obtained with gum arabic and inulin, respectively), suggesting that the presence of phenolic compounds was responsible for the activity (IC_{50} expressed as mgGAE/mL ranged between 0.27 to 1.15 mgGAE/mL, for α -amylase, and 0.17 to 1.71 mgGAE/mL, for α -glucosidase). The potential to inhibit key enzymes of carbohydrate metabolism by phenolic compounds has been described by different authors (Ademiluyi & Oboh, 2013; Gonçalves, Mateus, & De Freitas, 2011; Mojica, Meyer, Berhow, & de Mejía, 2015)

In general, the powders had similar activity against α -amylase and α -glucosidase, however, phenolic extracts from *Citrus maxima* were more active against α -glucosidase than α -amylase (Oboh & Ademosun, 2011). These differences suggest that the inhibition of these enzymes depends on the nature of the phenolic compounds present in the extracts. (Tadera, Minami, Takamatsu, & MATSUOKA, 2006) evaluated the activity of quercetin against these enzymes and showed more efficiency against α -glucosidase (7 μ M) than α -amylase (0.50 mM). The above suggests that the activity of the powders extract is primarily related to the

presence of phenolic compounds other than quercetin, such as proanthocyanidins and ellagitanin (De Souza Schmidt Goncalves et al., 2010).

Fig. 2. Half maximal inhibitory concentration (IC₅₀) of the α -amylase and α -glucosidase by the spray-dried *E. dysenterica* powder extracts. A, using gum arabic as wall material; B, using inulin as carrier agent.

3.5 Antimicrobial activity

Antimicrobial activity of powders from cagaita fruit against *S. aureus* and *L. monocytogenes* is presented in Table 4. Worth to note that the extracts were active only against Gram positive bacteria. The absence of activity against gram negative bacteria can be explained due to the presence of an outer membrane and a periplasmatic space, which can act as a barrier to penetration of molecules able to serve as antibiotics (Duffy & Power, 2001).

All samples obtained with gum arabic as carrier agent showed inhibition of *S. aureus* and *L. monocytogenes*; meanwhile, only the samples with 10% and 20% of inulin showed activity. As expected, increased carrier agent concentrations led to decreased of the inhibition (**Table 4**). Similar results were reported for antimicrobial activity of spray-dried pigments from Bordo grapes (Souza et al., 2014). In addition, the activity against *S. aureus* and *L. monocytogenes* presented by some of the powder extracts was similar to the antibiotics Vancomycin and Cetriaxone used as positive controls; however it was lower compared with Erythromycin (**Table 4**).

Similar to the zone of inhibition, the MIC values were influenced by the concentration of carrier agent (**Table 4**). The lowest value of MIC was presented by the power obtained with 10% of GA at 160 °C, for both microorganisms. As expected, freeze-dried sample presented the highest activity against the two microorganisms, due to the lack of dilution effect.

The inhibitory effect of the extracts can be linked to the presence of phenolic compounds in the powder. According to (Borrás-Linares et al., 2015) this activity is caused due to the disturbance of the function of bacterial cell membranes which retards bacterial growth or multiplication, explained by the presence of hydroxyl groups in the molecule (Friedman, Henika, & Mandrell, 2003). Several studies have shown the relation between the presence of phenolic compound and antimicrobial activity (Barbosa-Pereira et al., 2014; Megdiche-Ksouri et al., 2015), and the antimicrobial activity of pure quercetin (Rauha et al., 2000).

4. CONCLUSIONS

The use of the carrier agents increased the stability of the powders and reduced the loss of phenolic compounds during storage. Gum arabic was the more suitable material to stabilize the extracts as they presented lower phenolic losses after 120 days of storage. Meanwhile, powders obtained using inulin had reduction of about 50% of the total phenolic content; due to its high hygroscopicity. In this sense, the use of spray-drying to obtain dry

extracts from cagaita can be an alternative to prevent deterioration of the bioactive compounds present in the fruit, ensuring the quality and availability of the fruit. Moreover powders can be used as functional ingredient in the preparation of other foods.

Table 4. Antimicrobial activity and minimum inhibitory concentration (MIC) against the microorganisms tested of spray-dried cagaita fruit extract produce by spray drying using different temperatures and carrier agent concentration.

Samples	<i>Staphylococcus aureus</i>				<i>Listeria monocytogenes</i>			
	GA		Inulin		GA		Inulin	
	Inhibition (mm)	MIC (mg/mL)	Inhibition (mm)	MIC (mg/mL)	Inhibition (mm)	MIC (mg/mL)	Inhibition (mm)	MIC (mg/mL)
1	15±0.0 ^c	0.48	12±0.0 ^d	0.48	12±0.0 ^b	0.48	10.5±0.7 ^{c,d}	0.48
2	13±0.0 ^e	1.48	10±0.0 ^f	1.48	11±0.0 ^c	1.48	10±0.0 ^d	1.48
3	11±0.0 ^f	4.44	nd	nd	10±0.0 ^d	1.48	nd	nd
4	14±0.0 ^d	0.48	12±0.0 ^d	0.48	12±0.0 ^b	0.48	11±0.0 ^c	0.48
5	12.5±0.7 ^e	1.48	10±0.0 ^f	1.48	10±0.0 ^d	1.48	nd	Nd
6	11±0.0 ^f	4.44	nd	nd	10±0.0 ^d	1.48	nd	Nd
7	15±0.0 ^c	0.16	14±0.0 ^c	1.48	12±0.0 ^b	0.16	12.5±0.7 ^b	0.48
8	11.5±0.7 ^f	1.48	11±0.0 ^e	1.48	11±0.0 ^c	0.48	11±0.0 ^c	1.48
9	10±0.0 ^g	4.44	nd	nd	10.5±0.0 ^{c,d}	1.48	nd	nd
Freeze-dried	17.0±0.0 ^b	0.024	17.0±0.0 ^b	0.024	14.6±0.0 ^a	0.012	14.6±0.0 ^a	0.012
Vancomycin	14±0.0 ^d	ne	14±0.0 ^c	ne	9±0.0 ^e	ne	9±0.0 ^e	ne
Erythromycin	30±0.0 ^a	ne	30±0.0 ^a	ne	ne	ne	ne	ne
Ceftriaxone	ne	ne	ne	ne	10±0.0 ^d	ne	10±0.0 ^d	ne

Samples: 1, 120 °C / 10% CA; 2, 120 °C / 20% CA; 3, 120 °C / 30% CA; 4, 140 °C / 10% CA; 5, 140 °C / 20% CA; 6, 140 °C / 30% CA; 7, 160 °C / 10% CA; 8, 160 °C / 20% CA; 9, 160 °C / 30% CA; MIC, minimum inhibitory concentration; nd, not detected; ne, not estimated. Different superscripts letters in the same column mean significantly differences ($p < 0.05$).

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4. CONCLUSÕES

O uso de goma arábica e inulina como carreadores na produção de extratos secos de cagaita, usando a técnica de atomização, resultou em elevados rendimentos de processo, assim como em alta estabilidade dos pós relacionada aos baixos conteúdos de umidade, à baixa atividade de água e às altas temperaturas de transição vítrea.

Os parâmetros de processo, temperatura de ar de entrada e concentração de carreador, influenciaram as propriedades físicas dos extratos secos obtidos, principalmente umidade, atividade de água, higroscopicidade, solubilidade e temperatura de transição vítrea.

Todos os extratos secos apresentaram boas propriedades tecnológicas quando comparados com a amostra liofilizada, sendo que a higroscopicidade e a solubilidade foram as principais características melhoradas com o uso dos carreadores, proporcionando assim uma maior estabilidade e uma alta capacidade de reconstituição dos pós.

Os pós produzidos por atomização usando goma arábica como carreador apresentaram compostos fenólicos totais com alta estabilidade ao longo do tempo de armazenamento, superior à das amostras obtidas com inulina, as quais apresentaram pegajosidade e perda de compostos fenólicos.

Os extratos secos obtidos apresentaram elevados teores de compostos bioativos fenólicos, os quais foram relacionados à boa capacidade antioxidante, inibição das enzimas envolvidas no metabolismo de carboidratos (α -amilase e α -glicosidase) e ao potencial antimicrobiano frente à *Staphylococcus aureus* e *Listeria monocytogenes*.

Em relação às propriedades tecnológicas, os melhores parâmetros de processo para a obtenção de extratos secos de cagaita foram maior concentração de carreador (30%) e maior temperatura de entrada do ar (160°C). Porém, em relação às propriedades funcionais dos pós, as melhores condições de processo foram aquelas que utilizaram uma menor concentração de carreador (10%), evitando o efeito de diluição dos fenólicos por parte do carreador, e maior temperatura (160°C).

O uso da atomização para obter extratos secos de cagaita pode ser uma alternativa para reduzir os custos de transporte, evitar a deterioração e garantir a qualidade e disponibilidade da fruta.

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