UNIVERSIDADE ESTADUAL DE MARINGÁ
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APLICAÇÃO DE DIFERENTES MÉTODOS DE SECAGEM PARA OBTENÇÃO DE MACAÚBA (*Acrocomia aculeata*) EM PÓ.

DALANY MENEZES OLIVEIRA

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Tese apresentada ao Programa de Pós-Graduação em Ciência de Alimentos da Universidade Estadual de Maringá, como parte dos requisitos para obtenção do título de Doutor em Ciência de Alimentos.

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Orientador

Prof. PhD. Edmar Clemente

BIOGRAFIA

Dalany Menezes Oliveira, filha de Francisco Ibanês Rolim Oliveira e Francisca Ednólia Menezes Oliveira, nasceu em 05 de maio de 1982, na cidade de Várzea-Alegre, Ceará.

Graduou-se em 01 de setembro de 2006, em Tecnologia em Alimentos, pelo Instituto Centro de Ensino Tecnológico – CENTEC, Unidade de Juazeiro do Norte - Ceará, atual Faculdade de Tecnologia Centec - FATEC, com a defesa do trabalho intitulado “Aproveitamento de co-produtos da semente de gergelim CNPA-G4 para geração de emprego, renda e segurança alimentar”, orientada pelo professor Jonas dos Santos Sousa.

Durante o período da graduação, foi estagiária do Laboratório de Tecnologia de Alimentos do Centro Nacional de Pesquisa do Algodão – EMBRAPA- CNPA.

Em 28 de fevereiro de 2010, concluiu o mestrado no Programa de Pós-Graduação em Agronomia, Área de Concentração em Produção Vegetal, com ênfase em Pós-colheita de frutas e hortaliças na Universidade Estadual de Maringá, com a dissertação intitulada “Influência de revestimentos comestíveis e refrigeração na conservação da amora-preta”, orientação do professor PhD Edmar Clemente.

No mês de março de 2010 ingressou no doutorado no Programa de Pós-Graduação em Ciências de Alimentos com a orientação do professor PhD Edmar Clemente.

Em outubro de 2012 passou no teste seletivo para professor colaborador da Universidade Estadual de Maringá, campus Umuarama, e em fevereiro de 2013 foi contratada para ministrar aulas nos cursos de Tecnologia de Alimentos e Engenharia de Alimentos.

Tem experiência na área de Ciências e Tecnologia de alimentos, com ênfase na qualidade pós-colheita e secagem das frutas e hortaliças.

Dedico

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APRESENTAÇÃO

Esta tese de doutorado está apresentada em forma de 5 artigos científicos, redigidos de acordo com as normas das revistas listadas.

1 Dalany Menezes Oliveira, Janaina de Paula da Costa, Edmar Clemente, José Maria Correia da Costa. Characterization of Grugru Palm Pulp for Food Applications. Journal of Food Science and Engineering. v.3, n.2, p.107-112, 2013. Qualis Capes: C.

2 Dalany Menezes Oliveira, Edmar Clemente, José Maria Correia da Costa. Bioactive compounds and physicochemical parameters of grugru palm pulp and powder. Food Science and Technology Research. v.20, n.1, 2014. Fator de Impacto: 0,557. Qualis Capes: B2

3 Dalany Menezes Oliveira, Edmar Clemente, José Maria Correia da Costa. Hygroscopic behavior and degree of caking of grugru palm (Acrocomia aculeata) powder. Journal of Food Science and Technology. Publicado em: 01/09/2012. DOI 10.1007/s13197-012-0814-9. Fator de Impacto: 1,123. Qualis Capes: B2

4 Dalany Menezes Oliveira, Edmar Clemente, Marcos Rodrigues Amorim Afonso, José Maria Correia da Costa. Hygroscopic behavior of lyophilized powder of grugru palm *(Acrocomia aculeata).* American Journal of Analytical Chemistry. v.4, n.10A, 2013. Qualis Capes: C.

5 Dalany Menezes Oliveira, Cleidiane Gomes Lima, Edmar Clemente, Marcos Rodrigues Amorim Afonso, José Maria Correia da Costa. Estabilidade dos compostos bioativos e parâmetros de qualidade do pó de macaúba (*Acrocomia aculeata*) nas diferentes condições de secagem. Journal of Food Processing and Preservation. Fator de Impacto: 0,45. Qualis Capes: B2.

GENERAL ABSTRACT

INTRODUCTION. Grugru palm (*Acrocomia aculeata* (Jacq.) Lodd. Ex Mart.) is native to the savannas, open woodlands and 'cerrados' in tropical America. Several products can be obtained from such palm, especially the fruit pulp with great nutritional potential once it is a natural source of β-carotene, vitamin A and minerals. Grugru palm fruits can be consumed fresh or in powder form, they can also be used as raw material for formulations many products formulations. The powder can be obtained by different drying techniques, but some of these processes cause a large quality loss (nutritional and sensory) in the final product. For this reason, knowing the properties of the grugru palm pulp and powder is important for adding value to the regional fruits and approaching the drying processes for better conservation, development of products and preparation of various types of food.

AIMS. Application of two drying processes, lyophilization and oven dehydration in order to obtain grugru palm powder, carry out physical and physicochemical evaluations, determine bioactive components and both pulp and powder hygroscopic behavior to finally evaluate the stability of quality parameters for the dehydrated product stored for 120 days in different packages.

MATERIAL AND METHODS. The grugru palm fruits were harvested in the Araripe Plateau of Cariri area, State of Ceara, Brazil, and then taken to the Laboratory of Food Quality Control and Drying of Federal University of Ceará (UFC) the fruits were selected according to both ripeness and sanity rates to be soon sanitized and peeled, the pulp was then set apart by the use of a knife and stainless steel. All the obtained pulp was homogenised, grinded and divided in plastic packages containing 100g pulp. Initially, the product characterization was conducted concerning respiratory rate, physical, chemical and mineral analysis. Later on, the analyses on the pulp were performed, handled to form two types of pulp: integral pulp (IP) and pulp with 8% maltodextrin (MP). Four grugru palm powders were prepared using the following processes: oven drying with air circulation at 65° C for 25 hours, without maltodextrin (T1) and with 8% of maltodextrin (T2); lyophilization for 25 hours, without maltodextrin (T3) and with 8% of maltodextrin (T4). Physical analyses were conducted on: water activity, color, adsorption isotherms, hygroscopicity and degree of caking; Physcochemical analyses were conducted on: moisture, pH, tritable acidity, soluble solids; Bioactive components: total phenolic, yellow flavonoids, vitamin C, β-carotene and vitamin A. While studying the stability of quality parameters, each treatment was placed in different packages: plastic - P; laminated – L; laminated and vacuum closed - LV. Subsequently, the packages were stored at room temperature (± 25° C) for 120 days and analyzed every 30 days for the following determinations: moisture, water activity, color, β-carotene, total phenolics and yellow flavonoids. Data were statistically analyzed through the analysis of variance (ANOVA), the differences between the averages were determined by the Tukey test at 5% probability.

RESULTS AND DISCUSSION. The fresh fruit evaluation revealed a respiration rate of 74.45 ml $CO_2.kg^{-1}.\ h^{-1}$. High levels of sugars, soluble solids, pH, a_w, lipids and carotenoids were found; however, the pulp showed low levels of acidity, moisture and

ashes. Potassium (K) presented the highest content, followed by magnesium (Mg) and calcium (Ca); zinc (Zn) showed the lowest content. The results also showed high contents of β-carotene and vitamin A, high levels of water activity and soluble solids, low level of moisture and acidity in the grugru palm pulp, characterizing it as a sweet product. The pulps in this study showed an orange color, and the IP showed a more intense orange color than that from MP. The T3 and T4 treatments presented the highest levels of the bioactive compounds: phenolics, vitamin C, β-carotene and vitamin A, indicating that lyophilization was better in holding these compounds. However, both T1 and T2 levels of flavonoids showed higher values than those of T3 and T4. The best treatment in reducing moisture and water activity (a_w) was T3, but all drying methods reduced moisture and a_w levels, thus providing the inhibition of microbial growth. The T2 powder showed the darkest L* parameter, and the chromaticities a* and b* of T3 presented the most saturated yellow color followed by T4, revealing more attractive colors. The adsorption isotherms results showed that all models studied were fitted to the grugru palm powder dehydrated in oven and lyophilization. The adsorption isotherms were classified as Type III. The values for hygroscopic behaviors were 6.39 and 5.17% and for degree of caking were 3.11 and 0.03% for T1 and T2, respectively. The values for hygroscopic behaviors were 7.68 and 6.86% and for degree of caking 0.33 and 0.09% for T3 and T4, respectively. The GAB model was the best at representing isotherms behavior in T1 and T3, and the Oswin model in T2 and T4. For the stability study on bioactive compounds and the quality parameters for grugru palm powder analyzed each 30 days during 120 days of storage at room temperature (± 25° C), the following results were obtained: the moisture content and water activity for all treatments and packages presented a gradual increase, except for water activity in T1 and T2 packed in LV. At the end of 120 days of storage, all treatments stored in plastic bags (P) and T1 stored in laminated packs surpassed the maximum moisture allowed by the legislation. In the color evaluation, T1 showed the most visible changes in brightness (L*) at the end of the experiment. The values for -a* coordinate were low, presenting a grayish hue for the powder; this coordinate showed no significant decrease during the storage period. The chromaticity +b* showed a yellow hue for the powder for all treatments, being T3 and T4 more saturated than T1 and T2. The dehydration method affected the content of β-carotene: T3 and T4 β-carotene concentrations were higher than those from oven dehydration (T1 and T2); another factor that influenced the β-carotene content was the addition of maltodextrin. During the storage period, the β-carotene has degraded regardless of both treatment and packaging. The phenolic contents presented a decrease at the end of the storage period. All treatments showed a slight fluctuation in the flavonoids values and a raise at the end of 120 days of storage.

CONCLUSIONS. The grugru palm fruit has a high respiratory rate, therefore the pulp processing is recommended in order to increase the product shelf life. Grugru palm pulp proved to be a sweet fruit, presenting low acidity, orange color and excellent levels of bioactive components, particularly the levels of β-carotene. The adsorption isotherms of grugru palm powders can be classified as Type III. This powder is regarded as a non-hygroscopic and non-caking product. The best drying process in this study was lyophilization. The best conservation of grugru palm powder at room temperature (± 25° C) is lyophilization without maltodextrin (T3) and storage in laminated and vacuum closed packages (LV). This way, grugru palm can be regarded as an alternative for food enrichment and supplement.

Key words: *Acrocomia aculeata*, drying, lyophilization, β-carotene.

RESUMO GERAL

INTRODUÇÃO. A palmeira da macaúba (*Acrocomia aculeata* (Jacq.) Lodd. Ex Mart.) é nativa das savanas, cerrados e florestas abertas da América Tropical. Diversos produtos podem ser obtidos a partir desta palmeira, destacando-se a polpa do seu fruto que apresenta um grande potencial como alimento nutritivo por ser fonte natural de β-caroteno, vitamina A e minerais. A macaúba pode ser consumida *in natura* ou na forma de pó, sendo que este pode ser utilizado como matéria-prima nas formulações de diversos produtos. A obtenção do pó pode ser a partir de diferentes técnicas de secagens sendo que alguns destes processos causam grande perda na qualidade (nutricional e sensorial) do produto final. Por este motivo, o conhecimento das propriedades da polpa integral e do pó da polpa de macaúba é importante para a valorização dos frutos regionais, além de abordar os processos de secagem para melhor conservação, desenvolvimento de ingredientes e preparo de diversos tipos de alimentos.

OBJETIVOS. Aplicação de dois processos de secagem, liofilização e desidratação em estufa, para obtenção dos pós de macaúba e avaliação física, físico-química, componentes bioativos e comportamento higroscópico das polpas e pós, e avaliar a estabilidade dos parâmetros de qualidade das polpas desidratadas durante 120 dias de armazenamento em diferentes embalagens.

MATERIAL E METODOS. Os frutos de macaúba foram colhidos na Chapada do Araripe na Região do Cariri – CE nos meses de dezembro/2010 a março/2011 e encaminhados ao Laboratório de Controle de Qualidade de Alimentos e Secagem da Universidade Federal do Ceará - UFC. Os frutos foram selecionados de acordo com o grau de maturidade e sanidade e depois higienizados para o descascamento, em seguida, a polpa foi separada com auxílio de faca de aço inoxidável. Toda a polpa obtida foi homogeneizada, triturada e separadas em embalagens plásticas contendo 100 g da polpa. Inicialmente foi realizada a caracterização do fruto, no que concerne a: taxa respiratória, análises físico-químicas e mineral. Posteriormente, foram realizadas análises nas polpas, as quais foram separadas em dois lotes: polpa integral (PI) e polpa com adição de 8% de maltodextrina (PM). Foram elaboradas quatro pós da polpa de macaúba pelos seguintes processos: secagem em estufa com circulação de ar a 65°C por 25 horas: T1 (sem adição de maltodextrina) e T2 (com adição de 8% da maltodextrina); secagem em liofilizador por 25 horas: T3 (sem adição de maltodextrina) e T4 (com adição de 8% da maltodextrina). Foram realizadas analises físicas: atividade de água, cor, isotermas de adsorção, higroscopicidade e grau de *caking*; físico-químicas: umidade, pH, acidez titulável, sólidos solúveis; componentes bioativos: fenólicos totais, flavonoides amarelos, vitamina C, β-caroteno e vitamina A. Para o estudo da estabilidade dos parâmetros de qualidade, cada tratamento foi acondicionado em diferentes embalagens: Plástica - P; Laminada - L e Laminada com fechamento a vácuo - LV. Posteriormente, foram armazenadas em temperatura ambiente (±25°C) por 120 dias e analisadas a cada 30 dias, realizando-se as seguintes determinações: umidade, atividade de água, cor, β-caroteno, fenólicos totais e flavonoides amarelo. Os dados foram analisados estatisticamente por meio da análise de variância (ANOVA), e as diferenças entre as médias foram determinadas pelo teste de Tukey a 5% de probabilidade.

RESULTADOS E DISCUSSÃO. A avaliação da fruta fresca demonstrou uma taxa de respiração de 74,45 mL CO_2 Kg^{-1} h^{-1}. Teores elevados foram encontrados para os açúcares, sólidos solúveis, pH, a_w, lipídios e carotenóides totais, no entanto a polpa apresentou baixa acidez, umidade e cinzas. O potássio (K) foi o mineral de maior valor encontrado, seguido do magnésio (Mg) e do cálcio (Ca), sendo o zinco (Zn) o mineral de menor teor. Os resultados obtidos para as polpas da macaúba apresentaram elevados teores nas concentrações de β-caroteno e vitamina A, demonstrando baixa umidade e acidez, um elevado a_w e teor de sólidos solúveis, caracterizando-se como um produto adocicado. As polpas deste estudo apresentaram coloração laranja, sendo que a PI apresentou uma coloração laranja mais intensa que a PM. Os compostos bioativos dos pós dos tratamentos T3 e T4 obtiveram os maiores teores dos fenólicos, vitamina C, β-caroteno e vitamina A, mostrando que a liofilização foi melhor na retenção destes bioativos. Entretanto os teores de flavonóides os T1 e T2 apresentaram valores superiores aos T3 e T4. O tratamento que melhor reduziu a umidade e a_w foi o T3, entretanto para todas as secagens realizadas foram obtidos teores de umidade e a_w reduzidos, proporcionando a inibição do crescimento microbiano. O pó que apresentou o parâmetro L* mais escuro foi o T2, e as cromaticidades a* e b* o T3 foi o que apresentou a cor amarelo mais saturada seguido do T4, demonstrando assim uma coloração mais atrativa. Os resultados das isotermas de adsorção demonstraram que todos os modelos estudados se ajustaram aos pós da macaúba desidratados na estufa e no liofilizador. As isotermas de adsorção foram classificadas como do Tipo III. Os valores obtidos para higroscopicidade foram: 6,39 e 5,17% e grau de *caking* 3,11 e 0,03% para o T1 e T2, respectivamente. Os valores obtidos para higroscopicidade foram 7,68 e 6,86% e grau de *caking* 0,33 e 0,09% para o T3 e T4, respectivamente. O modelo GAB melhor representou o comportamento das isotermas para os pós T1 e T3, e o modelo Oswin para o T2 e T4. No estudo da estabilidade dos compostos bioativos e parâmetros de qualidade dos pós da macaúba analisados durante 120 dias de armazenamento em temperatura ambiente (± 25°C) foram obtidos os seguintes resultados: A umidade e atividade de água durante o tempo de armazenamento, para todos os tratamentos e embalagens, obtiveram um aumento gradativo, exceto para a atividade de água nos T1 e T2 acondicionados nas embalagens LV. Ao final dos 120 dias de armazenamento, todos os tratamentos armazenados na embalagem plástica - P e o tratamento T1 embalagem - L extrapolaram o máximo permitido pela legislação para a umidade. Na avaliação da cor, o T1 foi o tratamento que apresentou alterações mais visíveis em sua luminosidade (L*) das amostras até o final do experimento. Os valores encontrados para a coordenada -a* foram baixos, demonstrando uma tonalidade acinzentada no pó, essa coordenada sofreu uma redução não significativa durante o tempo de armazenamento. A cromaticidade +b* do pó da macaúba em todos os tratamentos apresentaram tonalidade amarela, sendo os T3 e T4 sua cor mais saturada em relação ao tratamento T1 e T2. O tipo de desidratação influenciou na quantidade do β-caroteno, os tratamentos T3 e T4 apresentaram teores maiores em relação aos pós secos em estufa T1 e T2, outro fator que influenciou nesta determinação foi a adição da maltodextrina. Durante o tempo de armazenamento o β-caroteno sofreu degradação independente do tratamento e embalagem utilizada. O tempo de armazenamento em cada tratamento demonstrou uma redução dos teores dos fenólicos ao final dos 120 dias. Todos os tratamentos apresentaram uma leve oscilação nos valores dos flavonóides e aumento ao final dos 120 dias de armazenamento.

CONCLUSÕES. O fruto possui alta taxa respiratória e, desta forma, recomenda-se processamento das polpa para aumentar o tempo de vida útil do produto da macaúba. A polpa da macaúba demonstrou ser adocicado, de baixa acidez, de coloração laranja e com excelentes teores dos componentes bioativos, com destaque para os teores de β-caroteno. As isotermas de adsorção dos pós da macaúba podem ser classificados como do Tipo III. A macaúba em pó é classificada como um produto não higroscópico e não formador de *caking*. O melhor processo de secagem deste estudo é a liofilização. A melhor condição de conservação do pó da macaúba durante o tempo de armazenamento na temperatura ambiente (±25°C) é a liofilização sem adição de maltodextrina (T3) e armazenamento na embalagem laminada a vácuo (LV). Desta forma, a macaúba pode ser considerada uma alternativa para o enriquecimento da dieta e suplementação alimentar.

Palavras chaves: *Acrocomia aculeata*, secagem, liofilização, β-caroteno.

Characterization of Grugru Palm Pulp for Food Applications

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Abstract

Studies are needed to know the grugru palm (*Acrocomia aculeata*) potential for food industry. This study aimed to characterize physicochemical quality and minerals in the mesocarp (pulp) of grugru palm. Grugru palm fruits from NE Brazil production area were collected and analyzed. The determinations included: respiratory rate, sugars, soluble solids, pH, titratable acidity, humidity, water activity (a_w), ashes, protein, lipids, total phenolics, total carotenoids, vitamin C, color and minerals. The fruit respiration rate was 74.45 mL CO_2 kg^{-1} h^{-1}. High levels were found for sugars, soluble solids, pH, aw, lipids and carotenoids; however, the pulp showed low acidity, moisture and ashes. The values found for lipids, minerals and vitamin C are equivalent to those observed in the literature. The grugru palm fruit has a high rate of respiration, which turns necessary the use of conservation techniques and methods. The grugru palm fruit pulp shows physical quality parameters attractive to the consumer, physicochemical quality and nutritive composition which can be considered for food ingredients and alternative source of ingredient for supplementation of diet food for needy population.

Key words: Grugru palm (*Acrocomia aculeata*), mesocarp, minerals, carotenoids, flavonoids.

1. Introduction

The grugru palm (*Acrocomia aculeata* (Jacq.) Lodd. Ex Mart.) is native to the grasslands, savannahs and open forests of tropical America, and in some areas occurs as dense populations [1]. According to Henderson et al. [2], the *Acrocomia aculeata* (Jacq.) Lodd. ex Mart. has several synonyms, among them are *Acrocomia intumescens* Drude and *Acrocomia sclerocarpa* Mart.

In Brazil, these palms can be found abundantly in the states of Ceará, Minas Gerais, Mato Grosso, Mato Grosso do Sul, São Paulo, Paraná, Santa Catarina and Rio Grande do Sul [3]. The common name of this palm depends on the region that is found as: Mbocayá (Argentina); totaí (Bolivia); corozo (Colombia, Venezuela); tamaco (Colombia); Coyol (Costa Rica, Honduras, Mexico). In Brazil, it is known as bocaiúva, chiclete-de-baiano, coco-baboso, coco-de-catarro, coco-de-espinho, macacaúba, macaíba, macaibeira, macajuba, macaúba, macaúva, mucaia, mucaja e mucajaba [4].

The family of palm trees has great importance in the tropical region due to the large variety of products obtained, especially those related to its fruits and seeds, being a major source of resources for food, homemade medicines, fuel, house rooftop, cooking utensils, household decorations and also as raw material for local industries [5].

The fresh pulp is directly consumed or used to produce flour that can be used within drinks, candies, ice creams, custards, cakes, jams and porridges; it has a sweet and mucilaginous taste. It can be consumed fresh or cooked in soft drinks. It can also be used for oil extraction with characteristics similar to that of oil palm, besides its almond which produces a thin oil [6].

Ramos et al. [7] demonstrated in their research the potential of grugru palm pulp to enrich diet regional programs as a supplemental food, being a natural source of β-carotene and vitamin A as well as the minerals copper, potassium and zinc.

Thus, the utilization of grugru palm pulp in preparing various foods can be an income alternative for rural communities. Therefore, in facing the need to value foods prepared with palm tree fruits as grugru palm and seeking for a better understanding of their nutritional potential and quality, the current study aimed to characterize qualitatively the grugru palm mesocarp (pulp).

2. Materials and Methods

Grugru palm fruits were harvested in the Araripe Plateau, Cariri region, Ceará State, Brazil, and taken to the Laboratory of Food Quality Control and Drying for selection and sanitization; they were subsequently peeled and pulped for analysis of the mesocarp. About five kilograms of ripe fruits were reserved after selection and sanitization for determining the respiration rate.

The determination of respiration rate was performed as described by Brackmann et al. [8], where in the fruits were packed in hermetically sealed glasses with three liters capacity, and after two hours approximately the concentration of CO_2 was determined through a gas analyzer (OXYBABY® WITT, SUNNYVALLE, Witten, Germany).

The determination of lipids was performed by direct extraction with petroleum ether as solvent in a Soxhlet extractor for six hours, according to the method 963.15 [9]. The protein concentration was determined by using the total nitrogen conversion factor of 6.25, in Kjeldahl technique, according to the method 991.20 [9]. The ashes (stable mineral residue) were determined by gravimetric method (930.22), in a calcination oven at 550 °C [9]. The determination of sugars was made through the calorimetric method of 3,5-dinitrosalicilic (DNS), as by described Miller [10], using a 0.2 mL samples, which were diluted in 1.3 mL water and added the DNS reagent; then, it was taken to warming for 100 °C for analysis of the compound absorbance at 540 nm.

The moisture content was determined gravimetrically at 105 °C in an oven until constant weight according to the technique described by Instituto Adolfo Lutz [11]. The water activity (a_w) was determined by a portable measurer (AquaLab®, BrasEq, Washington, EUA). The pH level was performed by a potentiometer, following the method described by AOAC [9], number 31.1.07. The titratable acidity was determined through NaOH (1 mol L^{-1}) until pH 8.1 was reached. The soluble solids were measured by a digital refractometer (Atago, Pocket-pal model, BrasEq, Tokyo, Japan) with scale from 0 to 35° Brix [12].

The total phenolics were extracted and determined by spectrophotometric Folin-Ciocalteau method according to Buci-Kojic et al. [13] with some modifications, 0.2 mL of the extract was mixed with 1.8 mL of distilled water and 10 mL of Folin-Ciocalteau reagent. After 30 s to 8 min, 8 mL of 7.5% of sodium carbonate solution was added and put in dark place for two hours. After this period, the absorbance was measured at 765nm (UV/VIS). Blank sample was prepared with water instead of the extract.

Determination of total phenolic compounds was calculated from the calibration curve obtained with gallic acid (GAE), which was used as a standard and results were expressed as mg GAE 100 g^{-1}. The yellow flavonoids were extracted in accordance to Francis [14], through a solution of ethanol:HCl 1.5 mol L^{-1} (85:15). The total carotenoids were determined by weighing about 2 g sample, grinding up and adding 18 mL of acetone 80%; then the mixture was filtered in a light-free environment and the reading performed with a spectrophotometer at 663 nm (chlorophyll a), at 646 nm (chlorophyll b) and at 470 nm (carotenoids); their contents were expressed in mg g-1, according to the formula described by Lichtenthaler [15]. The ascorbic acid (vitamin C) was titrated by a solution of 2,6-dichloro-phenol-indophenol as described by Strohecker and Henning [16].

The pulp colors were determined by a colorimeter (Minolta, model CR410, Konica Minolta Sensing, Inc., Japan) in the mode CIE L*a*b* and parameters D65 (Y = 93.9; x = 0.3167; y = 0.3336).

The minerals content was determined by nitro-perchloric digestion using the method of Malavolta et al. [17], and the determination was done by atomic absorption spectrometry (Varian-mod. Spectra A 10 Plus). Phosphorus (P) was determined using a UV/VIS spectrophotometer according to the method described by Pavan et al. [18].

3. Results and Discussion

The respiration rate of grugru palm fruits was 74.45 mL CO_2 kg^{-1} h^{-1}, which demonstrates to be post-harvest short-lived fruits with rapid senescence. Chitarra and Chitarra [19] stated that the storage period for plant products is related to their respiratory rate intensity. Generally, fruits and other vegetables with high respiratory rates tend to age faster [20]. Therefore, as to fruits such as grugru palm, more studies on technologies for improving shelf-life are recommended. Gomes et al. [21] reported that a method for fruit pulp conservation available for application in the industry is drying, which concentrates the product nutritional compounds and enables to storage them at room conditions for extended periods.

Table 1 shows the values for the physical, physicochemical and bioactive compounds of grugru palm mesocarp from the Araripe Plateau, Cariri region, Ceará State (NE Brazil).

Table 1 Physical and physicochemical characteristics and bioactive compounds of grugru palm mesocarp.

Analysis[1]		Quantification ± δ
Total sugars (g.100g^{-1})		21.67 ± 0.57
Reducing sugar (g.100g^{-1})		4.17 ± 0.05
Non-reducing sugars		17.50 ± 0.51
Soluble solids (ºBrix)		29.66 ± 0.57
pH		5.50 ± 0.02
Titratable acidity (g.100g^{-1} oleic acid)		1.47 ± 0.07
Moisture (g.100g^{-1})		33.93 ± 0.33
a_w		0.9250 ± 0.00
Ashes (g.100g^{-1})		1.15 ± 0.02
Proteins (g 100g^{-1})		3.03 ± 0.72
Lipids (g 100g^{-1})		18.70 ± 0.77
Total phenolics (mg GAE.100g^{-1})		50.89 ± 9.76
Yellow flovonoids (µg g^{-1})		350.00 ± 70.43
Total carotenoids (mg g^{-1})		17.69 ± 0.61
Vitamin C (mg 100g^{-1} ascorbic acid)		135.45 ± 9.05
Color	*L	42.52 ± 0.37
	*a	5.37 ± 0.29
	*b	17.75 ± 1.06

[1]All analysis were determined on a fresh weight basis.

High levels of total sugars were observed in grugru palm fruits, featuring the pulp as a sweet taste product; it was also observed that non-reducing sugars values were higher than that of reducing sugars. Sanjinez-Argandoña and Chuba [22] found levels of total sugars in fruits harvested in the states of São Paulo and Mato Grosso do Sul, respectively, of 11.58 and 14.53 g 100 g^{-1}, below the levels observed in this study; however, the reducing sugar levels herein were similar to those observed in the fruits harvested in São Paulo and Mato Grosso do Sul, respectively, 5.06 and 5.59 g 100 g^{-1}.

Supporting the fruit sweetish characteristic, in the grugru palm pulp a high content of soluble solids, high pH and low acidity were observed, and these factors favor the fruit sensory characteristics, making it more palatable [22].

The pulp moisture content in this study was relatively low when compared to other palm tree pulps, as shown by Canuto et al. [23], who reported values of 83.30 and 84.80 g 100 g^{-1}, in fruits of açai and bacuri, respectively. The low moisture content found in this study could represent an important feature to prevent microbial growth; however, according to Ribeiro and Seravalli [24], knowing the amount of water (water activity) available in the food will provide stability and safety, but the grugru palm pulp presents a very high a_w. Sanjinez-Argandoña and Chuba [22] found a_w values of 0.95 and 0.90 for fruits collected in São Paulo e Mato Grosso do Sul, similar values found in this study.

The ashes, or stable mineral residue, showed values close to those found in the literature, where their concentrations ranged from 2.22 to 1.51 g 100 g^{-1} [7,22, 25]. However, differences between the stable amount of minerals in the fruits from different regions and localities may occur, probably due to soil type, climate and bioavailability of minerals in each region [26].

The protein values found in this study were greater than that observed in the literature, which ranged from 2.76 to 1.5 g 100 g^{-1} [7, 25, 27]. The RDC Resolution No. 269 [28] indicates the values of Recommended Daily Intake (RDI) for proteins, vitamins and minerals; so according to these recommendations, the intake of 100 g of grugru palm represents a RDI of 6% protein for an adult and 8.8% for a child (7-10 years).

The lipid content found in grugru palm pulp in this study was similar to that found in Brasil [6] and Silva et al. [25], 13.80 and 14.93 g 100 g^{-1}, respectively, being a higher value than that found by Ramos et al. [7] of 8.14 g 100 g^{-1}; and lower than that reported by NEPA-UNICAMP [26] of 40.70g 100g^{-1}.

The total phenolic content is above the values found by Kuskoski et al. [29] for cupuassu pulp 20.50 mg GAE 100 g^{-1}, being lower than those of fruits such as grapes (117 mg GAE 100 g^{-1}) and açai (136 mg GAE 100 g^{-1}) which has great phenologic potential.

The flavonoids range in similar values to those found in kale, 266-399 μg g^{-1} [30], cv. “Pera” orange pulp, 348 μg g^{-1}, and cv. “Gala” apple, 277 μg g^{-1} [31]. No other studies on flavonoids in grugru palm were found.

The grugru palm fruit has excellent levels of bioavailable carotenoids, such as β-carotene, becoming a natural source of vitamin A [7]. The value found in grugru palm pulp was greater than that found in fruits such as pequi, 1.13 mg g^{-1}, studied by Oliveira et al. [32].

In the literature, there is a wide range of vitamin C content for grugru palm. In this study, the content was higher than those found in samples from the Midwest region, 28.00 to 34.57 mg 100 g^{-1} and 11.46 to 13 mg 100 g^{-1} in samples from São Paulo State [6, 22, 26]; however, the contents of vitamin C (ascorbic acid) in this study are close to that observed in samples taken from Piauí State by Rocha [33], 185.10 mg 100 g^{-1}. Therefore, this component behavior in different Brazilian regions supports the statement by Rufino et al. [34], in which the content can vary among regions, depending on factors such as temperature, light intensity and moisture content.

The color instrumental evaluation was specified numerically in a spherical three-dimensional space, defined by: L*, ranging from black (0) to white (100); a*, from green (-a) to red (+a) and b*, from blue (-b) to yellow (+b) [35]. Based on this definition, one can characterize the grugru palm pulp color as a dark yellow-orange color (L* tends to black). Grugru palm samples from São Paulo SP-Sanjinez Argandoña and Chuba [22] showed a less dark (light) yellow-orange color than that in this study, L* 70.66.

Table 2 presents the mineral characteristics of grugru palm pulp (from NE Brazil).

It is noted that potassium (K) presents the highest mineral value, followed by magnesium (Mg) and calcium (Ca), and zinc (Zn) presents the least mineral content. Three studies on grugru palm minerals were found, which were carried out by NEPA-UNICAMP [26], Ramos et al. [7] and Silva et al. [25]. The amount of Ca ranges within those observed in the literature, from 61.96 to 130.00 mg 100 g^{-1}; K and P contents did not differ from those in the literature, from 36.70 to 766.37 mg 100 g^{-1}, respectively. In this study, Mg, Fe, Cu and Mn were higher than those observed in other studies. No studies were found quantifying B (boron).

Table 2 Mineral characteristics of grugru palm mesocarp.

Minerals[1]	Quantification ± δ
Calcium - Ca (mg $100g^{-1}$)	101.33 ± 1.15
Potassium - K (mg $100g^{-1}$)	649.33 ± 19.34
Magnesium - Mg (mg $100g^{-1}$)	122.00 ± 8.00
Phosphorus - P (mg $100g^{-1}$)	53.33 ± 5.77
Iron - Fe (µg g^{-1})	120.66 ± 10.35
Copper - Cu (µg g^{-1})	7.33 ± 0.57
Manganese - Mn (µg g^{-1})	6.00 ± 1.00
Zinc - Zn (µg g^{-1})	4.56 ± 1.50
Boron - B (µg g^{-1})	25.77 ± 6.31

[1]All minerals were determined on a fresh weight basis.

According to a Ministry Act No. 31/1998-SVS/MS [36] a food is characterized as rich or source of mineral when it provides 30% or 15% of the Recommended Daily Intake (RDI) per 100 g pulp, respectively. Ramos et al. [7] stated that grugru palm pulp can be classified as rich in copper for children and source for adults; it is also considered as a source of zinc and potassium.

The grugru palm mesocarp (pulp) is the fruit part of higher yield, which corresponds to 48% of the total fruit weight [7]; therefore, the amount of pulp is an important feature, reflecting on the importance of fruit extractivism [22]. This fruit pulp is up to technological utilization and supplying of nutritional components used in various foods (vitamins, juice, cake, jelly, ice cream, etc).

4. Conclusions

The grugru palm fruit presents high respiration rate. The use of conservation techniques and methods for extending the fruit storage period is recommended.

The grugru palm mesocarp has physicochemical characteristics and mineral nutrients that attractive to the consumption; the physicochemical characteristics turn it

palatable; and the desirable nutritional composition can be considered diet enrichment and supplemental food for needy population.

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Bioactive Compounds and Physicochemical Parameters of Grugru Palm (*Acrocomia aculeata*) From Brazil: Pulp And Powder

Characterization of Grugru Palm: Pulp and Powder

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LIST OF ABBREVIATIONS

IP: integral pulp;
MP: pulp with 8% maltodextrin;
T1: oven drying without maltodextrin;
T2: oven drying with 8% maltodextrin;
T3: lyophilized without maltodextrin;
T4: lyophilized with 8% maltodextrin;
PTFE: polytetrafluoroethylene;
RE: retinol equivalent;
δ: standard deviation;
GAE: gallic acid equivalent;

ABSTRACT

As there is little study on grugru palm, the knowledge of the properties of the fruit pulp and powder is important in the fruit production recovering and in the enhancement of the processes of dehydration for better conservation, development and preparation of diverse foods. The objective of this study was to characterize the bioactive compounds and the physicochemical features of grugru palm pulp and powder. Grugru palm fruits were harvested in the Araripe Plateau, Ceará State, Brazil, and dehydrated by two drying methods (in oven with air circulation and lyophilization) with and without addition of drying adjuvant (maltodextrin). After analysis, results revealed that pulp and powder of grugru palm are products with high content of soluble solids and low acidity, highlighting the high levels of β-carotene and vitamin A. The lyophilized powder T3 have showed the best results in relation to the physicochemical and bioactive parameters.

KEYWORDS: *Acrocomia aculeata*, drying, lyophilization, maltodextrin, β-carotene, vitamin A.

INTRODUCTION

Grugru palm (*Acrocomia aculeata* (Jacq.) Lodd. Ex Mart.) is native to the grasslands and savannas of Tropical America (Clement *et al*., 2005). This palm tree is of great regional importance due to the wide diversity of products, especially those related to its fruits and seeds (Lorenzi, 2006).

The pulp is eaten fresh or used in the production of powders that can be used in drinks, candies, ice creams, custards, cakes, jellies and juices (Brasil, 2002). The grugru palm fruits show great potential in the enrichment of the regional diet and in supplemental feeding programs, being a natural source of β-carotene and vitamin A, as well as the minerals copper, potassium and zinc (Ramos *et al*., 2008).

Dehydration of fruits and vegetables is utilized in food preservation, which maintains their nutritional and commercial values and also provides a new product in the market, encouraging investments in agricultural production and processing (Soares *et al*., 2001).

Many types of dryers utilized in dehydrating food are found in literature and in the drying process of thermo-sensitive foods, additives are required and have to be added in the appropriate level in order to maintain the product acceptability and to follow the limits set by legislation (Oliveira *et al*., 2007). One of the most common additive applied in drying fruits is maltodextrin, which has low cost and low hygroscopicity thereby preventing agglomeration and retaining the volatile substances in the range of 65-80% (Oliveira *et al.*, 2006).

In the literature, the drying processes applied to grugru palm, namely conventional forced air dryers at temperatures ranging from 45 to 65° C and/or drying by direct sunlight can be found. However, other drying methods such as lyophilization have not been studied, as well as a comparison of the processes of dehydration on the quality of grugru palm powder. Therefore, the objective of this study was to characterize the bioactive compounds and the physicochemical features of grugru palm pulp and powder.

MATERIAL AND METHODS

Raw material

Grugru palm fruits were harvested in the Araripe Plateau, Cariri region, Ceará State, Brazil, and taken to the Laboratory of Food Quality Control and Drying for selection and sanitization; subsequently they were peeled and the pulp stored in a freezer at -20° C until drying and analyses.

The pulps were analyzed in the following forms: integral pulp (IP) and with 8% maltodextrin (MP).

Grugru palm pulps were dried by two processes:

T1: oven drying without maltodextrin.

T2: oven drying with 8% maltodextrin.

T3: lyophilized without maltodextrin.

T4: lyophilized with 8% maltodextrin.

Chemical

All chemicals and solvents used for determination β-carotene and Vitamin A were of reagent or HPLC grade. The standard of β-carotene was obtained from Sigma Chemical Co. (St. Louis, MO).

Oven drying

The pulps (T1 and T2) were unfrozen, smashed and distributed in stainless steel trays with 25cm diameter; then the trays were taken to a forced air oven (TECNAL, model TE-394/l) at 65° C for 25 hours (Oliveira *et al*., 2012). Immediately after drying, the product was taken to a rotatory blade mill (MA 048, Marconi) in order to obtain a homogeneous powder.

Lyophilization

The pulps (T3 and T4) were unfrozen, smashed and distributed in stainless steel trays with 15cm diameter; then the trays were taken to a ultra-freezer (LC 90-40V, Terroni) at -40° C for 24 hours; after this period the pulps were dehydrated in a benchtop lyophilizer (LS 3000, Terroni) for 25 hours (Oliveira *et al*., 2013).

Immediately after lyophilization, the product was taken to a rotatory blade mill (MA 048, Marconi) in order to obtain a homogeneous powder.

Bioactive compounds

The total phenolics were extracted and determined according to the method described by Buci-Kojic *et al*. (2007). The yellow flavonoids were extracted in accordance to Francis (1982), through a solution of ethanol:HCl 1.5 mol L^{-1} (85:15).

The ascorbic acid (vitamin C) was titrated by a solution of 2,6-dichloro-phenol-indophenol as described by Strohecker and Henning (1967).

The extraction of β-carotene was carried out as described by Rodriguez-Amaya (1999) with a little modification. Samples (5g) sample were extracted with cold acetone until the residue becomes colorless; then it was filtered under vacuum using a Büchner funnel. The pigments extracted were then transferred to petroleum ether and washed in distilled water until complete removal of the acetone. The extract concentration was made in a rotatory evaporator at 35º C. The pigments were dissolved in acetone, filtered in a PTFE with 0.22 µm membrane and then analyzed. The β-carotene content was analyzed by HPLC chromatographic conditions developed by Sant'Ana *et al*. (1998) with modifications. The devices utilized are: a liquid chromatograph (model Gilson 321 pump) with automatic injector (model Gilson 234) and handle of 20µL (loop), column ACE 5 C18 (5µm, 150x4.6 mm), visible-UV detector at 450nm (model Gilson 152). The mobile phase was composed of methanol/acetonitrile/ethyl-acetate (80:10:10) at a rate of 1.5 $ml.min^{-1}$ with running time of 60 minutes.

The quantification of β-carotene was attained by external standard curves. The vitamin A activity of β-carotene was obtained according to Bauernfeind (1972) and the conversion factor; the calculation of vitamin A was provided by the National Research Council (NAS, 1980) in which 6 µg of β-carotene correspond to 1 Retinol Equivalent (RE).

Physicochemical determinations

The moisture content was determined gravimetrically at 105° C in an oven until constant weight according to the technique described by Instituto Adolfo Lutz (2005). The water activity (a_w) was determined by a portable measurer (AquaLab®). The pH level was performed by a potentiometer, following the method described by AOAC

(1998), number 31.1.07. The titratable acidity was determined through NaOH (1 mol L^{-1}) until pH 8.1 was reached. The soluble solids were measured by a digital refractometer (Atago, Pocket-pal model) with scale from 0 to 35° Brix (Carvalho *et al*., 1990).

The pulp and powder colors were determined by a colorimeter (Minolta, model CR410, Konica Minolta Sensing, Inc., Japan) in the mode CIE L*a*b* and parameters D65 (Y = 93.9; x = 0.3167; y = 0.3336).

Statistical analysis

All analyses were performed in triplicate (n=3). Data were statistically analysed using the analysis of variance (ANOVA) and the differences between the averages were determined by the Tukey test at 5% probability using the software Statistic, version 7.0 (Statsoft, 2007).

RESULTS AND DISCUSSION

Bioactive compounds of pulp and powder of grugru palm

Table 1 shows the bioactive compounds of IP (integral pulp) and MP (with 8% maltodextrin). The loss of vitamin C in MP was 37.2% and this is linked to the drying additive which could have encapsulated the ascorbic acid molecule, turning it difficult to be extracted. The total phenolics is higher than that found by Kuskoski *et al*. (2005) in cupuassu pulp (20.5 mg GAE 100 g^{-1}) and lower than those found in fruits such as grapes (117 mg GAE 100 g^{-1}) and hog plum (136 mg GAE 100 g^{-1}). The MP flavonoids were 32.1% lower than that of IP, even with no statistical difference at 5% of probability. The pulp flavonoids range similar to those found in kale 266-399 µg g^{-1} (Huber and Rodriguez-Amaya, 2008), and in pulps of orange var. 'Pera' 348 µg g^{-1} and apple var. 'Gala' 277 µg g^{-1} (Arabbi *et al*., 2004). The pulps of grugru palm fruits presented high levels of β-carotene and vitamin A, and these levels were higher than those found by Charoensiri *et al*. (2009) in orange 1.7 µg g^{-1}, watermelon 6.2 µg g^{-1} and papaya 4.7 µg g^{-1}. Ramos *et al*. (2008) reported a content of 49 µg g^{-1} β-carotene in grugru palm pulp. The high content of β-carotene in the pulp of grugru palm is related to the color of fruits and vegetables, which ranges from yellow to red and refers to high levels of carotenoids, according to Uenojo *et al*. (2007).

Table 1. Bioactive compounds of grugru palm pulp, integral and with addition of 8% maltodextrin.

Analyses	IP[a] ±δ	MP[b] ±δ[c]
Total phenolics (mg GAE[d] $100g^{-1}$)	51.3 [a] ±10.7 *	49.8 [a] ±2.03
Yellow flavonoids (µg g^{-1})	372.1 [a] ±76.0	247.8 [a] ±46.3
Vitamin C (mg $100g^{-1}$)	118.2 [a] ±6.01	72.8 [b] ±8.70
β-carotene (µg g^{-1})	35.9 [a] ±1.09	34.7 [a] ±2.03
Vitamin A (RE[e] $100g^{-1}$)	599.7 [a] ±18.1	579.2 [a] ±33.8

[a]IP: integral pulp; [b]MP: pulp with 8% maltodextrin; [c]δ: standard deviation; [d]GAE: gallic acid equivalent; [e]RE: retinol equivalent. * Equal lowercase letters in the same line do not differ statistically by Tukey test at 5% probability.

Table 2 presents the bioactive compounds of grugru palm powder in T1 and T2 (oven drying) and in T3 and T4 (lyophilized). The contents of bioactive compounds (phenolics, vitamin C and vitamin A) in T3 and T4 were the highest, showing lyophilization a better process for retention of these compounds. However, the flavonoids in T1 and T2 were higher than those of T3 and T4; these lower levels in T3 and T4 were probably caused by the freezing period before the lyophilization. Huber and Rodriguez-Amaya (2008) stated that processed products have flavonoids contents substantially lower than those found in fresh fruits.

Table 2. Bioactive compounds of grugru palm pulp obtained by different processes of drying, integral and with 8% maltodextrin.

Analyses	T1[a] ±δ	T2[b] ±δ	T3[c] ±δ	T4[d] ±δ[e]
Total phenolics	82.7 [ab]*	65.1 [b]	89.4 [a]	86.9 [a]
(mg GAE[f] $100g^{-1}$)	±7.98	±6.7	±5.7	±8.5
Yellow flavonoids	135.7 [a]	121.1 [ab]	115.6 [b]	93.6 [c]
(µg g^{-1})	±12.6	±4.47	±3.46	±3.51
Vitamin C	52.1 [b]	51.7 [b]	103.3 [a]	100.7 [a]
(mg $100g^{-1}$)	±5.6	±3.09	±5.6	±5.63
β-carotene	51.5 [bc]	36.1 [c]	80.1 [a]	57.6 [b]
(µg g^{-1})	±0.66	±0.50	±7.3	±3.99
Vitamin A	859.4 [bc]	601.3 [c]	1334.8 [a]	961.4 [b]
(RE[g] $100g^{-1}$)	±10.9	±8.3	±122.0	±66.41

[a]T1: oven drying without maltodextrin; [b]T2: oven drying with 8% maltodextrin; [c]T3: lyophilized without maltodextrin; [d]T4: lyophilized with 8% maltodextrin; [e]±δ: standard deviation; [f]GAE: gallic acid equivalent; [g]RE: retinol equivalent; *Equal lowercase letters in the same line do not differ statistically by Tukey test at 5% probability.

Physicochemical evaluation of pulp and powder of grugru palm

Table 3 presents the physicochemical parameters related to moisture, water activity, acidity, pH, soluble solids and pulp color. In general, the maltodextrin did not change these parameters, except for a_w, and color L*a*. The pulps have showed low moisture and acidity and high soluble solids and a_w, which makes them sweet and susceptible to microbial growth. Different levels of moisture and acidity were found in the literature: Ramos *et al*. (2008) observed a high moisture content (52.99 g 100 g^{-1}) and Hiane *et al*. (2005) reported values of acidity lower than 0.83 g 100 g^{-1}. These variations could have been caused by different factors, e.g., fruit origin, climatic conditions, harvest time, type of pulping, etc. The pulps in this study have showed an overall orange color, but the IP presented a more intense orange color than the MP.

Table 3. Physicochemical features of grugru palm pulp, integral and with 8% maltodextrin.

Analyses		IP[a] ±δ	MP[b] ±δ[c]
Moisture (g $100g^{-1}$)		40.2 [a*] ±0.72	41.3 [a] ±0.31
a_w[d]		0.92 [b] ±0.01	0.94 [a] ±0.002
Titratable acidity (g $100g^{-1}$)		1.43 [a] ±0.07	1.64 [a] ±0.27
pH		5.50 [a] ±0.02	5.68 [a] ±0.14
Soluble solids (°Brix)		29.7 [a] ±0.58	27.6 [a] ±2.81
Color	L*	42.4 [a] ±0.43	41 [b] ±0.69
	a*	5.08 [a] ±0.31	1.46 [b] ±0.17
	b*	17.3 [a] ±1.80	19.4 [a] ±0.36

[a]IP: integral pulp; [b]MP: pulp with 8% maltodextrin; [c]δ: standard deviation; [d]a_w: water activity; L* (lightness-darkness), a* (redness-greenness) and b* (blueness-yellowness).
[*]Equal lowercase letters in the same line do not differ statistically by Tukey test at 5% probability.

Table 4 presents the physicochemical parameters of grugru palm powder of T1 and T2 (oven drying), T3 and T4 (lyophilized). The treatment T3 has showed the lowest moisture and a_w; however, all drying processes have decreased moisture and a_w, which provide the inhibition of microbial growth. According to Ordóñez (2005), foods with a_w below 0.60 are labeled as microbiologically stable without microbial growth, which confirms our results. There was no difference amongst treatments in relation to acidity and pH, which shows that the drying methods did not affect these factors. The T4 has had the highest content of soluble solids followed by the T2, and the explanation for such high levels is the addition of maltodextrin. The grugru palm with the darkest L* parameter was the T2; in relation to the chromaticity a* and b*, the T3 presented the most bright yellow color followed by the T4, thus showing a more attractive color. The other treatments (T1 and T2) presented a paler chromaticity.

Table 4. Characterization of grugru palm powders according to different drying processes and with addition of 8% maltodextrin.

Analyses		T1[a] ±δ	T2[b] ±δ	T3[c] ±δ	T4[d] ±δ[e]
Moisture (g 100g^{-1})		3.28 [ab*]±0.08	3.41 [a] ±0.11	3.12 [b] ±0.09	3.49 [a] ±0.12
a_w [f]		0.21 [a] ±0.01	0.22 [a] ± 0.01	0.12 [b] ±0.01	0.22 [a] ±0.01
Titratable acidity (g 100g^{-1})		2.10 [a] ±0.01	2.47 [a] ±0.55	2.06 [a] ±0.05	2.50 [a] ±0.32
pH		5.57 [a] ±0.02	5.62 [a] ±0.10	5.71 [a] ±0.01	5.60 [a] ±0.02
Soluble solids (°Brix)		40.3 [c] ±0.89	45.7 [ab] ±2.63	45.1 [bc] ±1.28	50.3 [a] ±2.32
Color	L*	49.7 [a] ±0.09	48.8 [b] ±0.23	49.7 [a] ±0.26	49.8 [a] ±0.14
	a*	-1.38 [c] ±0.04	-1.03 [ab] ±0.14	-1.24 [bc] ±0.10	-0.94 [a] ±0.09
	b*	24.6 [c] ±0.16	22.6 [d] ±0.14	27.2 [a] ±0.28	26.3 [b] ±0.35

[a]T1: oven drying without maltodextrin; [b]T2: oven drying with 8% maltodextrin; [c]T3: lyophilized without maltodextrin; [d]T4: lyophilized with 8% maltodextrin; [e]±δ: standard deviation; [f]a_w: water activity; L* (lightness-darkness), a* (redness-greenness) and b* (blueness-yellowness). [*] Equal lowercase letters in the same line do not differ statistically by Tukey test at 5% probability.

Effect of drying on bioactive compounds

The technological processing and the types of nutrients in the foods are directly related to losses of bioactive compounds. In this study, there were different levels of loss of bioactive compounds in the two processes of pulp dehydration, i.e., forced air oven drying (T1 and T2) and lyophilization (T3 and T4).

The powders T1 and T2 suffered a reduction in phenolics of 0.32 and 21.4%, respectively. This greater loss of phenolics in T2 is probably associated to the difficulties in extraction and quantification of phenolics due to the addition of the maltodextrin. The powders T3 and T4 did not suffer losses of this constituent.

The levels of flavonoids were the most affected by the treatments, and their losses have ranged from 77.4 to 80.8%. Ewald *et al*. (1999) demonstrated that the great loss of flavonoids in vegetables takes place during peeling, cutting, pre-processing and bleaching, which explains the losses found in this study.

The vitamin C contents have reduced during the drying processes, as expected, because the stability of this vitamin is affected by several factors, such as oxygen, pH, light, enzymes and catalysts (Ordóñez, 2005). The largest losses have occurred in the powders T1 and T2, 72.7 and 72.9%, respectively; the lyophilized products, T3 and T4, have showed the lowest losses: 46.0 and 47.1%, respectively. The lyophilized products have presented the lowest losses because the stability of vitamin C increases along with decreasing temperature (Ordóñez, 2005).

The treatments T1 and T2 have showed the largest losses of β-carotene and vitamin A. The T1 and T2 have lost 11.4 and 37.9% of these constituents, respectively. The levels of β-carotene and vitamin A in the T3 did not change, and in the T4 the loss of these bioactive compounds was 0.67%.

The grugru palm fruits have an interesting nutritive potential. The mesocarp of grugru palm fruit provides significant concentrations of β-carotene, a natural antioxidant and pro-vitamin A; so the pulp can be included in feeding programs of the poor and contribute to the food enrichment as a source of vitamin A. This work contributes to the study of the stability of bioactive compounds and quality parameters of dehydrated grugru palm pulp, aiming to define the best processing and conservation conditions for applying to this raw material as a new ingredient in different formulations of food products.

CONCLUSION

The grugru palm pulp is a product of low acidity, presents an orange color and has excellent levels of bioactive compounds, especially of β-carotene and vitamin A. The addition of 8% maltodextrin did not affect the concentration of vitamin C.

The lyophilized powders T3 and T4 have showed the best results in relation to the physicochemical and bioactive parameters, highlighting the T3 in relation to moisture content, water activity, and levels of β-carotene and vitamin A.

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Hygroscopic Behavior and Degree of Caking of Grugru Palm (Acrocomia aculeata) Powder

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Abstract

This work aims to investigate the hygroscopic behavior of grugru palm powder through adsorption isotherms and its degree of caking. The powders of grugru palm (T1 - without maltodextrin, T2 – with 8 % of maltodextrin) were obtained by oven drying at 65 °C for 25 h. The experimental data was obtained through static gravimetric method at temperatures of 25, 30, 35 and 40 °C with different saturated salt solutions. The models of GAB, BET, Henderson, and Oswin were fitted to experimental data. The values of hygroscopicity were 6.39 and 5.17 % and degrees of caking were 3.11 and 0.03 % for T1 and T2, respectively. The adsorption isotherms from mathematical models can be classified as Type III. The GAB and Oswin models were the best representing the behavior of the powder isotherms, T1 and T2, respectively. The grugru palm powder proved to be non-hygroscopic and nonagglomerating. The T2 with 8%of maltodextrin presented the lowest hygroscopicity.

Keywords: *Acrocomia aculeata*, Sorption isotherms, Food drying, Degree of caking, Isotherms, Mathematical modeling, Powder technology.

Introduction

Brazil has a wide variety of oil plants, natives or introduced; among these species, the grugru palm (Acrocomia aculeata) stands out for being highly productive and native to arid and semiarid regions (Kopper et al. 2009). The pulp is eaten fresh or used in the production of powders that can be used in drinks, candies, ice creams, custards, cakes, jellies and juices (Brasil 2002).

The grugru palm presents a pulp which is consumed fresh or utilized in the production of flour that can be employed in different foods; this fruit is sweet, mucilaginous and can be cooked for soft drinks and ice cream (Brasil 2002).

According to Ramos et al. (2008), the bioavailability of β-carotene, a precursor of vitamin A, in the pulp of grugru palm is greater than the bioavailability of pure β-carotene.

According to Fernandes et al. (2011), it is very important and critical in the economic process to select a suitable drying process for preservation of fruit characteristics. The research on drying of common fruits has been extensively carried out; however, studies on exotic, tropical and native fruits are scarce.

During storage, foods exposed to several relative humidities and temperatures; and tends to reach equilibrium with the environment and accordingly adjust the moisture (Toneli et al. 2008). Mathlouthi and Rogé (2003) stated that to explain the hygroscopic behavior of different food products, mathematical models of adsorption isotherms have been proposed.

The understanding of adsorption isotherms of food moisture is very important in science and technology of food for many purposes, such as design and optimization of industrial processes, e.g., drying, packaging and storing, so it is crucial to model moisture changes taking place during dehydration and in order to predict the stability of shelf life (Jamali et al. 2006).

According to Jaya and Das (2004) the hygroscopicity is the ability of food powder to absorb moisture from high relative humidity environment. In the case of fruit powders, glucose and fructose are responsible for strong interaction with the water molecule due to the polar terminals present in these molecules.

Studies for obtaining grugru palmpowder is very important for food technology, because it is necessary to discover the best techniques to attain the best storage conditions. Hence, the objective is to investigate the hygroscopic behavior, the degree

of caking and to apply mathematical models to predict the adsorption isotherms of grugru palm powders obtained by oven drying with forced air ventilation.

Material and methods

Raw material

The grugru palm fruits were harvested in the AraripePlateau, Cariri region, Ceará State, Brazil, and taken to the Laboratory of Food Quality Control and Drying. The fruits were selected according to the level of maturity and sanity and later were hygienized for the peeled and then the pulp was separated through a stainless steel knife. The pulp was stored in a vertical freezer (Esmaltec, 340) at - 20 °C until the the drying process initiated.

Two grugru palm powders were utilized:

Control treatment (T1) – oven drying of pulp without addition of drying adjunvant.

Treatment 2 (T2) – oven drying of pulp with addition of drying adjuvant (8 % of maltodextrin 'Maltogill 20®', dextrose equivalent - DE 20). An aqueous maltodextrin solution was prepared and homogenized with triturated pulp before drying.

Oven drying

The pulp was unfrozen and crushed, the pulp for T1 was distributed in stainless steel trays with a diameter of 25 cm; the pulp for T2 was prepared with an aqueous maltodextrin solution and was homogenized and distributed into stainless steel trays with a diameter of 25 cm; and the trays were placed in an oven (Tecnal, model TE-394/l) with forced air ventilation at 65 °C for a period 25 h.

Right after drying, the product was taken to a rotatory blade mill (MA 048, Marconi) and then sieved (mesh of 1,200 μm) to obtain a homogeneous powder.

Modeling of adsorption isotherms

In determining the moisture adsorption isotherms, a static gravimetric method described by Wolf et al. (1985) was applied using saturated solutions of salts as described by Greenspan (1977). The salt solutions were prepared and put in tempered glass containers sealed with silicone and stored at room temperature (21±2 °C).

Measurements of adsorption isotherms were performed in triplicate. For each cell about 0.20 g of each treatment were weighed in pre-librated aluminum crucibles.

Afterward, the crucibles were taken to cells, which contained the saturated solutions of salts.

The process was monitored through sample weighing on analytical balance every 24 h during 10 days until equilibrium moisture was reached. After balanced moisture, the water activity of the samples was determined at temperatures of 25, 30, 35 and 40 °C using a_w meter (AQUALab 4TEV). Then were determinate of moisture content in an oven (Tecnal, model TE-394/l) with forced air ventilation at 75 °C to constant weight.

The equilibrium moisture (Xeq, Equation 1) was calculated by the difference between the mass of balanced sample and the dried mass:

$$X_{eq} = \frac{m_{eq} - m_s}{m_s} \quad (1)$$

Where: Xeq = equilibrium moisture (d.b.); m_{eq} = mass of balanced sample (g); m_s = mass of dried sample (g).

For adjusting of the experimental data from adsorption isotherms of grugru palm powder, mathematical models of GAB, BET, Henderson and Oswin were used, represented respectively by the equations in Table 1. The parameter calculations for each model were performed by the software Statistic, version 7.0 (Statsoft 2007).

The quality of adjusting different models was evaluated through the best values of the determination coefficient (R2) and the relative mean deviation (E%, Equation 6), defined by Iglesias and Chirife (1976):

$$E\% = \frac{100}{n}\sum_{i-1}^{n}\frac{|Xeq_e - Xeq_p|}{Xeq_e} \quad (6)$$

Where: E%= relative mean error; X_{eqe}= experimental values; X_{eqp}= values predicted by the model; n = number of experimental data.

Table 1. Mathematical models used to adjust the experimental data of adsorption isotherms.

Models	Equations [a]	
GAB (Guggenheim–Andersen–de Boer) (Van Den Berg and Bruin, 1981)	$X_{eq} = \frac{X_m . C . K . a_w}{(1-)K . a_w . (1 - K . a_w + C . K . a_w)}$	(2)
BET (Brunauer el al, 1938)	$X_{eq} = \frac{X_m . C . a_w}{1 - a_w}\left[\frac{1 - (n+1).(a_w)^2 + n.(a_w)^{n+1}}{1 - (1-C).a_w - C.(a_w)^{n+1}}\right]$	(3)
Henderson (Henderson, 1952)	$X_{eq} = \left[\frac{-\ln(1 - a_w)}{b}\right]^{\frac{1}{a}}$	(4)
Oswin (Oswin, 1946)	$X_{eq} = a.\left[\frac{a_w}{1 - a_w}\right]^b$	(5)

[a] a_w = water activity; X_m = moisture content in the molecular monolayer (g H_2O g^{-1} dry basis); X_{eq} = equilibrium moisture content (g H_2O g^{-1} dry basis); C = constant related to heat of sorption of the molecular layer; a, b, K = adjusting parameters.

Hygroscopicity

The analysis was performed using the methodology A14a, described by GEA Niro Research Laboratory (2003), consisting of powder exposure to air relative humidity (RH) of 79.5 % and checking of weight increase every 10 min until the maximum weight is reached. Approximately 0.5 g of the sample was weighed and evenly spread on a plate, then the plate was placed in the apparatus and the analysis was started. The calculation of hygroscopicity is given by Equation 7. The parameters used to characterize the hygroscopicity of grugru palm powder were obtained as described by GEA Niro Research Laboratory (2003).

$$\%\ \text{Hygroscopicity} = \frac{(\%WI + \%FW)\times 100}{100 + \%WI} \quad (7)$$

Where: $\%FW$ = % free water;

$\%WI = ((c-b)/(b-a)) \times 100$; a = weight of plate (g); b = weight of plate + powder (g); c = weight of plate + powder in equilibrium (g).

Degree of caking

The analysis was performed through the methodology A15a, described by GEA Niro Research Laboratory (2003), which consists of exposing the powder to absorb air moisture (79.5% relative humidity) until reaching of equilibrium as described in hygroscopicity. Afterward, the powder was oven dried at 105 °C and then sieved under standard conditions (sieve mesh modified to 1,200 μm). What was left in the sieve was expressed as degree of caking. The calculation of the degree of caking is given by Equation 8. The parameters to characterize the degree of caking of grugru palm powder were obtained as described by GEA Niro Research Laboratory (2003).

$$\%\, Degree\ of\ Caking = {}^{b\ \times 100}/_{a} \quad (8)$$

Where: a= grams of powder used; b = grams of powder retained in the sieve.

Statistical analysis

All analysis was determined in triplicate. Data were analyzed statistically through analysis of variance (ANOVA), and differences between the averages for hygroscopicity and degree of caking were determined by Tukey test at 5 % probability using the software Statistic, version 7.0 (Statsoft 2007).

Results and discussion

The adsorption isotherms of grugru palm powders showed a behavior characteristic to Type III, according to Brunauer classification (Rizvi 1995), as can be seen in Figs. 1a and b. Note that the values of the experimental data of equilibrium moisture (X_{eq}) increased due to the increased water activity under condition of constant temperature, as e.g. at 25 °C.

From the analysis of Figs. 1a and b, the grugru palm powder tend to gain moisture under environment with a_w above 0.1, indicating increased X_{eq} independently of the conditions of relative humidity. Noting at these figures, one can say that for T1 (Fig. 1a) GAB, Oswin and BET models adjusted best in the range of a_w from 0.1 to 0.6, but all models had excellent fits from 0 6 to 1.0. In Fig. 1b, all models adjusted to the

experimental data of a_w from 0.1 to 0.3 and from 0.8 to 1.0, and only GAB, Henderson and Oswin models presented the best adjustments for a_w from 0.3 to 0.7.

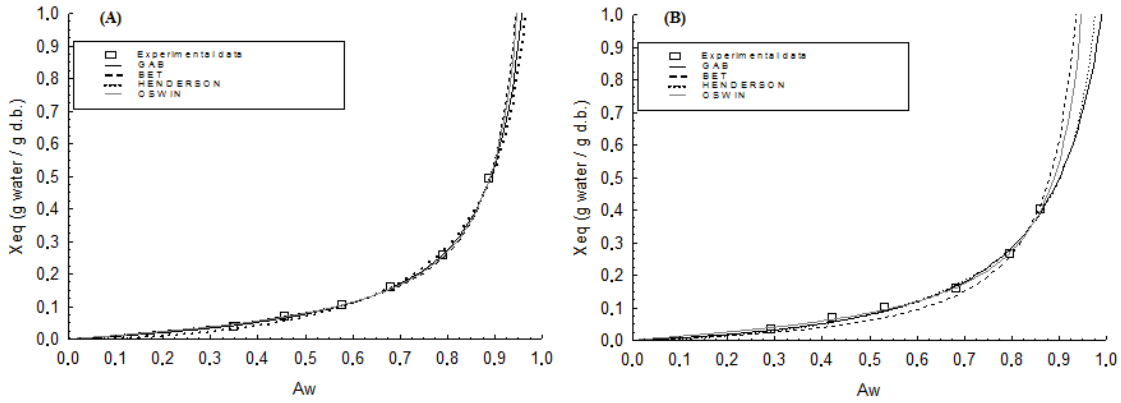

Fig. 1 Adsorption isotherms of grugru palm powder at 25 °C without maltodextrin - T1 (a) and with maltodextrin 8 % - T2 (b). (Abbreviations: a_w = water activity; X_{eq}= equilibrium moisture content g H_2O g^{-1} dry basis).

Table 2 contains the parameters obtained by GAB, BET, Henderson and Oswin models for grugru palm powders (T1 and T2) measured at temperatures of 25, 30, 35 and 40 °C.

The parameters of the models applied to the experimental data of adsorption isotherms of grugru palm powders (determination coefficients - R2 and the relative average deviations - *E%*) were utilized as evaluation criteria for the representation of isotherms are shown in Table 2.

According to the results shown in Table 2, all models studied (GAB, BET, Henderson and Oswin) can represent the behavior of grugru palm powders, as they exhibited excellent values of R^2 and *E%*, respectively from 0.97411 to 0.99961 and from 0.26 % to 1.82 %; these values are below the criteria recommended by Aguerre et al. (1989), where *E%* less than 10 % indicates a reasonable representation of the models, and by Labuza et al. (1985), in which the representation of isotherms is considered extremely good for *E%* less than 5 %. Thus, the GAB and Oswin models were the best representing the experimental data of T1 and T2, respectively.

The GAB model is reported by several researchers in forecasting adsorption isotherms of various dehydrated foods, particularly Ferreira and Pena (2003) in obtaining adjustment of this model with R^2 of 0.9996 and 0.9991 for peach-palm

powder, Silva et al. (2005) which found R^2 values higher than 0.99 for yellow mombin powder, and Alam and Singh (2011) who obtained the maximum R^2 for aonla flakes.

The moisture values in the monolayer (Xm) of GAB model for T1, between temperatures 25 and 40 °C, showed random fluctuations (Table 2) ranging from 0.079002 g H_2O g^{-1} to 0.090334 g H_2O g^{-1} on dry basis, with the highest value in the temperature of 35 °C. The same was observed for constants *C* and *K*. The values of *C* and *K* correspond to classification Type III, according to Brunauer $0<K<1$ and $0\leq C\leq 2$ (Blahovec, 2004).

Gabas et al. (2007) reported that strong adsorbate-adsorbent interactions are favored by lower temperatures, causing an increase in *C*, which explain the temperature variation and may be the result of a mathematical compensation between *C* and *K*.

The values of X_m are in accordance to the values reported for fruits comprising 4–15 % on dry basis (Moreira et al. 2008); however, Koua et al. (2012) obtained values for cassava ranging from 6.16g H_2O.100 g^{-1} to 3.66 g H_2O.100 g^{-1} on dry basis. The (GAB) X_m parameter is important because it can be related to the beginning of a series of chemical reactions of food decaying (Ferreira and Pena 2003).

The X_m parameter also characterizes the moisture content under which food is stable, because at lower values of X_m biochemical changes can be observed - lipid oxidation (Ordóñez 2005). Thus, the values of X_m (Table 2) for grugru palm show low moisture content in the monolayer, hence lipid oxidation can occur during storage in the temperatures here studied. So to prevent and minimize such oxidative processes, packaging should be impervious to air and light.

The Oswin model also showed the best fitting for dehydrated garlic with R^2 ranging from 0.9148 to 0.9488 (Arora et al. 2011); Basu et al. (2011) studying pectin found values of R^2 between 0.82 and 0.97, while *E%* was less than 0.012. Lomauro et al. (1985), quoted by Al-Muhtaseb et al. (2002), reported the Oswin model just fitted 57 % of the isotherms described for foods. For grugru palm powder in this study, the Oswin model presented R^2 value of 0.99818 and *E%* of 0.52.

Jain et al. (2010) stated that the Oswin model can be used to plan and evaluate drying, storage conditions and moisture amount to be removed during dehydration. Sopade et al. (1996) reported that this model can also be used to predict the extension of hydration or dehydration required.

Table 2. Adjustment results of experimental data of adsorption isotherms at 25, 30, 35 and 40°C for powders of grugru palm.

Models	Parameters[c]	T1[a]				Models	Parameters[c]	T2[b]			
		25°C	30°C	35°C	40°C			25°C	30°C	35°C	40°C
GAB	X_m	0.0790	0.0749	0.0903	0.0887	GAB	X_m	0.1351	0.9838	0.1101	0.1129
	C	1.0632	1.1767	0.8827	0.9021		*C*	0.5786	0.0912	0.7175	0.7018
	K	0.9674	0.9901	0.9807	0.9995		*K*	0.8969	0.7516	0.9453	0.9451
	R^2	0.9996	0.999	0.9991	0.9983		R^2	0.9928	0.9851	0.9825	0.9956
	E%	0.26	0.43	0.40	0.47		*E%*	0.98	1.34	1.32	0.79
BET	X_m	0.0772	0.1079	0.1390	0.1619	BET	X_m	0.2083	0.2341	0.111	0.1129
	C	1.4445	0.7924	0.6164	0.4364		*C*	0.3615	0.2541	0.8590	0.8719
	n	1.3535	1.2986	1.2656	1.1246		*n*	-0.7521	-0.5822	1.3158	1.2681
	R^2	0.9979	0.999	0.9989	0.9983		R^2	0.9741	0.9708	0.9882	0.9987
	E%	0.62	0.41	0.41	0.46		*E%*	1.82	2.31	1.00	0.41
Henderson	a	0.5935	0.5691	0.5655	0.5473	Henderson	a	0.661	0.6737	0.6324	0.6248
	b	3.3831	3.1328	3.0034	2.8181		b	3.6838	3.6527	3.3485	3.1871
	R^2	0.9969	0.9946	0.9964	0.9951		R^2	0.9905	0.9863	0.9758	0.9897
	E%	0.70	0.88	0.77	0.89		*E%*	1.18	1.31	1.56	1.23
Oswin	a	0.0794	0.0799	0.0831	0.0843	Oswin	a	0.0842	0.0874	0.0849	0.0902
	b	0.8758	0.9382	0.9626	1.0187		b	0.8525	0.8571	0.9311	0.9398
	R^2	0.999	0.999	0.9989	0.9983		R^2	0.9981	0.996	0.9875	0.9984
	E%	0.38	0.41	0.38	0.47		*E%*	0.52	0.68	1.05	0.45

[a] T1: powder without addition of maltodextrin; [b]T2: powder with addition of 8% maltodextrin. [c]Abbreviations: R^2 = determination coefficient. *E%* = relative average error. X_m = moisture content in the monolayer (g H_2O g^{-1} dry basis). C = constant related to the heat of sorption in the molecular layer. K = GAB constant related to multi-layers. n = BET constant related to multi-layers. a & b = adjusting parameters of the Henderson and Oswin models.

Figures 2a and b show the graphical presentations of adsorption isotherms of moisture for grugru palm powder, in all temperatures, adjusted by GAB and Oswin models for T1 and T2, respectively, which represented the best adjustment.

Figures 2a and b show that with increasing temperature there is an increase in equilibrium moisture values of a_w higher than 0.6 for T1 and T2. Also there is little influence of temperature on X_{eq} of grugru palm powder with a_w below 0.6. Studies carried out with surinam cherry powder indicated influence of temperature on equilibrium moisture from a_w above 0.3 (Vieira et al. 2007); this shows the grugru palm powder has a hygroscopicity lower than that of surinam cherry powder.

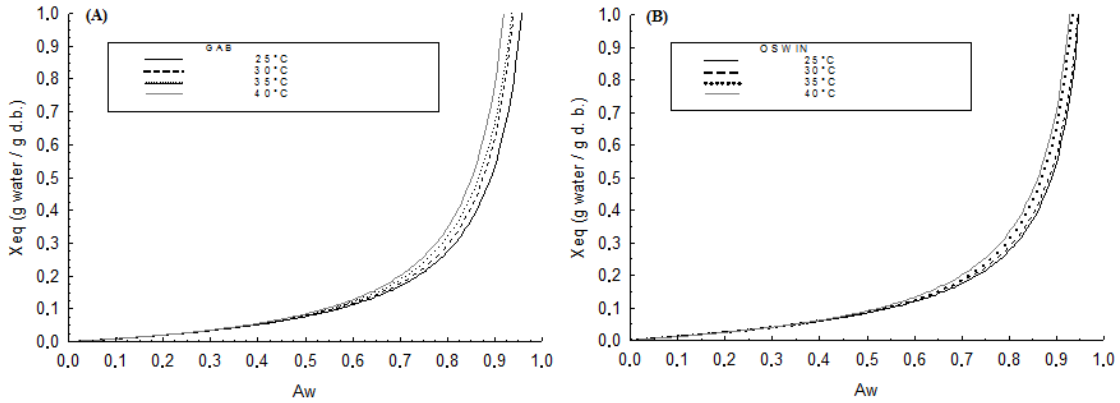

Fig. 2 Adsorption isotherms at different temperatures of the best model (GAB) for grugru palm powder without maltodextrin - T1 (a), and best model (Oswin) of for grugru palm powder with maltodextrin 8 % - T2 (b). (Abbreviations: a_w = water activity; X_{eq} = equilibrium moisture content g H_2O g^{-1} dry basis)

Table 3 shows the values of hygroscopicity and degree of caking for T1 and T2. Treatment T1 rendered higher hygroscopicity and caking degree in relation to T2. This can be related to the addition of drying adjuvant (maltodextrin), which results in decreased powder hygroscopicity, and this fact was also found in the adsorption isotherms of this work.

The powder caking is an undesirable reaction, consisting initially in the powder transformation into an agglomerated and sticky material and resulting in decreased functionality, smoothness and quality loss; the main cause of agglomeration is the presence of plasticizing water onto the surface of particles (Aguilera et al. 1995).

According to GEA Niro Research Laboratory (2003), which characterizes the hygroscopic behavior and the degree of caking, the grugru palm powder is classified as a non- hygroscopic and non-caking product.

Studies about sucrose by Mathlouthi and Rogé (2003) reported that caking is linked to solid bridges created during the agglomeration process, which turned sucrose more hygroscopic. The same can be seen in T1 in which the powder had the highest caking and thereafter making grugru palm powder with higher hygroscopicity, because this is a sweet product with about 17.5 % of sucrose.

Costa et al. (2003) reported that the variations in hygroscopic behavior of food powders are also assigned to the powder granulation, as products of finer particles have a greater surface of contact and therefore a greater number of active sites. This report can explain the non-hygroscopicity of grugru palm powder when compared to other high hygroscopic fruits described in the literature, as the particle size of powder of grugru palm is 1,200 μm and most of other fruit powders present a particle size ranging from 500 μm to 600 μm.

Conclusion

The adsorption isotherms of grugru palm powders adjusted by the mathematical models can be classified as Type III.

All models adjusted to the powder of grugru palm. However, it is recommend the GAB model in describing the equilibrium of adsorption isotherms for T1 and Oswin for T2.

The grugru palm powder showed a relative increase in moisture in the monolayer (X_m) along with increasing temperature. The values of *C* and *K* are in conformity with the classification Type III, and according to Brunauer $0<K<1$ and $0\leq C\leq 2$.

The grugru palm powder is characterized as non-hygroscopic and non-caking. The T2 treatment, with maltodextrin, showed lesser hygroscopicity.

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Hygroscopic Behavior of Lyophilized Powder of Grugru Palm (*Acrocomia aculeata*)

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ABSTRACT

The objective of this study was to determine adsorption isotherms and hygroscopic behavior of lyophilized powder from grugru palm. The powders of grugru palm were obtained by lyophilization process without maltodextrin (T1) and with 8% matodextrin (T2). The experimental data were obtained through the static gravimetric method at temperatures (25°C, 30°C, 35°C and 40°C), with different saturated solutions of salts. The models of GAB, BET, Henderson and Os-win were fitted to experimental data. The values obtained for hygroscopicity were 7.68% and 6.86% and the degree of caking was 0.33% and 0.09% for T1 and T2, respectively. Mathematical models of adsorption isotherms for grugru palm powders can be classified as Type III. The GAB and Oswin models represented better the behavior of isotherms for T1 and T2. Grugru palm powder showed an increase in the humidity of the monolayer X_m along with increasing temperature. The grugru palm powder demonstrated to be a non-hygroscopic product, non-caking features.

Keywords: *Acrocomia aculeate*; Adsorption Isotherms; Hygroscopicity; Degree of Caking

1. Introduction

Grugru palm (*Acrocomia aculeata* (Jacq.) Lodd. ex-Mart.) is native to the grasslands, savannas and open forests of Tropical America and occurs in dense populations in some localities [1].

It presents a fresh pulp, which is consumed fresh or used in the production of flour that can be utilized in different foods; it has sweet taste and is mucilaginous. This fruit can be eaten fresh or cooked, and used in soft drinks and ice creams [2].

Ramos *et al.* [3] demonstrated the potential of grugru palm pulp as a nourishing food, able to contribute to the enrichment of the regional diet in supplemental feeding programs, and a natural source of *β*-carotene, vitamin A and minerals as copper, potassium and zinc.

Dehydration of fruits and food are used as a method of preservation and storage, providing a new product on the market, which has motivated the investments in agricultural production and processing [4,5].

According to Toneli *et al.* [6], foods in storage period are exposed to various relative humidities and to different conditions of temperature; so it is necessary to adjust the moisture in order to reach equilibrium with the environment. These authors observed physical changes (caking) in inulin powder when there is humidity variation.
Mathlouthi and Rogé [7] stated that to explain the behavior of different insoluble food products several mathematical models were proposed for sorption isotherms. Studies by Kaymak-Ertekin and Gedik [8] for sorption isotherms in apples and potatoes showed an increase in temperature, resulting in decrease of equilibrium for moisture content.

The understanding of moisture sorption isotherms in food has great importance in food science and technology for many uses, such as design and optimization of industrial processes, e.g., drying, assessing the problems of packaging, modeling of moisture changes that occur during drying, forecasting the stability of shelf life and predicting mixtures of ingredients [9].

Obtaining grugru palm powder is still handmade, and studies are necessary to show the best techniques for obtaining and storing this product. Given the above, the objective of this work was to evaluate the hygroscopic behavior and the application of mathematical models in predicting the adsorption isotherms of lyophilized powders from grugru palm.

2. Material and Methods

2.1. Raw Material

Grugru palm fruits were harvested in Araripe Plateau in the Cariri Region, Ceará State, Brazil, and taken to the Laboratory of Food Quality Control and Drying (UFCE), which were selected and sanitized and subsequently peeled for pulp separation. The pulp was stored at -20°C until the lyophilization process.

Two lyophilized powders of grugru palm were used: one treatment without addition of drying adjuvant and other with maltodextrin of dextrose equivalent (DE) 20.

Control treatment (T1) - powder without maltodextrin Treatment with maltodextrin (T2) - powder with addition of maltodextrin 8%.

2.2. Lyophilization

The pulp was unfrozen and triturated; after this process the pulp of T1 and T2 were spread on stainless steel trays with diameter of 15 cm. The trays were placed in ultra-freezer (CL 90 - 40 V, Terroni) at -40°C for 24 hours; after this period the pulp was taken to a bench lyophilizer (LS 3000, Terroni) for 25 hours. Right after the lyophilization, the dried product was taken to a rotating knives mill (MA 048, Marconi) in order to get a powder with homogeneous granularity.

2.3. Modeling of Adsorption Isotherms

In determining the moisture adsorption isotherms, a static gravimetric method described by Wolf *et al.* [10] was applied using saturated solutions of salts according to Greenspan [11], at room temperature of 25°C ± 2°C. The solutions of salts were prepared and put in containers of tempered glass closed with silicone.

Measurements of adsorption isotherms were performed in triplicate. For each cell about 0.20 g of each treatment were weighed in pre-librated aluminum crucibles. Afterward, the crucibles were taken to cells, which contained the saturated solutions of salts.

The process was monitored by weighing of samples on analytical balance every 24 hours to obtain equilibrium. After balance the water activity of the samples was determined at temperatures of 25°C, 30°C, 35°C and 40°C, using a_w meter, model AQUALab 4TEV. Then they were taken to a lab chamber with air circulation at 105°C for determination of moisture content.

The equilibrium moisture (X_{eq}, Equation (1)) was calculated as the difference between the mass of balanced sample and the dried mass:

$$X_{eq} = \frac{m_{eq} - m_s}{m_s} \quad (1)$$

Where: X_{eq} = equilibrium moisture (d.b.); m_{eq} = mass of balanced sample (g); m_s = mass of dried sample (g).

For adjusting of the experimental data from adsorption isotherms of grugru palm powder, mathematical models of GAB (Equation (2)), BET (Equation (3)), Henderson (Equation (4)) and Oswin (Equation (5)) were used, represented respectively by the equations in **Table 1**. The calculations of parameters of each model were performed using the software Statistic version 7.0 [12].

Table 1. Mathematical models used to adjust the experimental data of adsorption isotherms.

Models	Equations [a]	
GAB (Guggenheim–Andersen–de Boer) (Van Den Berg and Bruin, 1981)	$X_{eq} = \frac{X_m . C . K . a_w}{(1 -)K . a_w . (1 - K . a_w + C . K . a_w)}$	(2)
BET (Brunauer el al, 1938)	$X_{eq} = \frac{X_m . C . a_w}{1 - a_w}\left[\frac{1 - (n+1).(a_w)^2 + n.(a_w)^{n+1}}{1 - (1 - C).a_w - C.(a_w)^{n+1}}\right]$	(3)
Henderson (Henderson, 1952)	$X_{eq} = \left[\frac{-\ln(1 - a_w)}{b}\right]^{\frac{1}{a}}$	(4)
Oswin (Oswin, 1946)	$X_{eq} = a.\left[\frac{a_w}{1 - a_w}\right]^b$	(5)

[a] a_w = water activity; X_m = moisture content in the molecular monolayer (g H_2O g^{-1} dry basis); X_{eq} = equilibrium moisture content (g H_2O g^{-1} dry basis); C = constant related to heat of sorption of the molecular layer; a, b, K = adjusting parameters.

The quality of adjusting different models was evaluated through the best values of the determination coefficient (R^2) and the relative mean deviation (E%, Equation (6)), defined by Iglesias and Chirife [17]:

$$E\% = \frac{100}{n}\sum_{i-1}^{n}\frac{|Xeq_e - Xeq_p|}{Xeq_e} \quad (6)$$

Where: E%= relative mean error; X_{eqe}= experimental values; X_{eqp}= values predicted by the model; n = number of experimental data.

2.4. Hygroscopicity

The analysis was perform described by GEA Niro Research Laboratory [18], consisting in powder exposure to air relative humidity (RH) of 79.5%, which was adsorbed through the powder sample until a constant weight is reached. The calculation of hygroscopicity is given by Equation (7). The parameters used to characterize the hygroscopicity of grugru palm powder were obtained according GEA Niro Research Laboratory [18].

$$\%\ \text{Hygroscopicity} = \frac{(\%WI + \%FW)\times 100}{100 + \%WI} \quad (7)$$

Where: $\%FW$ = % free water;
$\%WI = ((c-b)/(b-a)) \times 100$; a = weight of plate (g); b = weight of plate + powder (g); c = weight of plate + powder in equilibrium (g).

2.5. Degree of Caking

The analysis was perform A15a, described by GEA Niro Research Laboratory [18], which consists of exposing the powder to absorb mois- ture from the air (79.5% relative humidity) until reaching equilibrium. Afterward, the powder was dried and sieved under standard conditions (sieve mesh modified to 1200 μm). What was left in the sieve was expressed as degree of caking. The calculation of the degree of caking is given by Equation (8). The parameters used to characterize the degree of caking of grugru palm powder were obtained according to GEA Niro Research Laboratory [18].

$$\%\ Degree\ of\ Caking = {}^{b}\times 100/_{a} \quad (8)$$

Where: a= grams of powder used; b = grams of powder retained in the sieve.

3. Results and Discussion

3.1. Adsorption Isotherms

The adsorption isotherms of lyophilized powders from grugru palm fruits showed a behavior characteristic to Type III, according to Brunauer classification [19]. It is can be observed in **Figures 1** and **2** that the values of the experimental data for equilibrium moisture (X_{eq}) of lyophilized grugru palm powders raised in accordance to increasing water activity, under conditions of constant temperature, e.g. at 25°C shown in Figures cited.

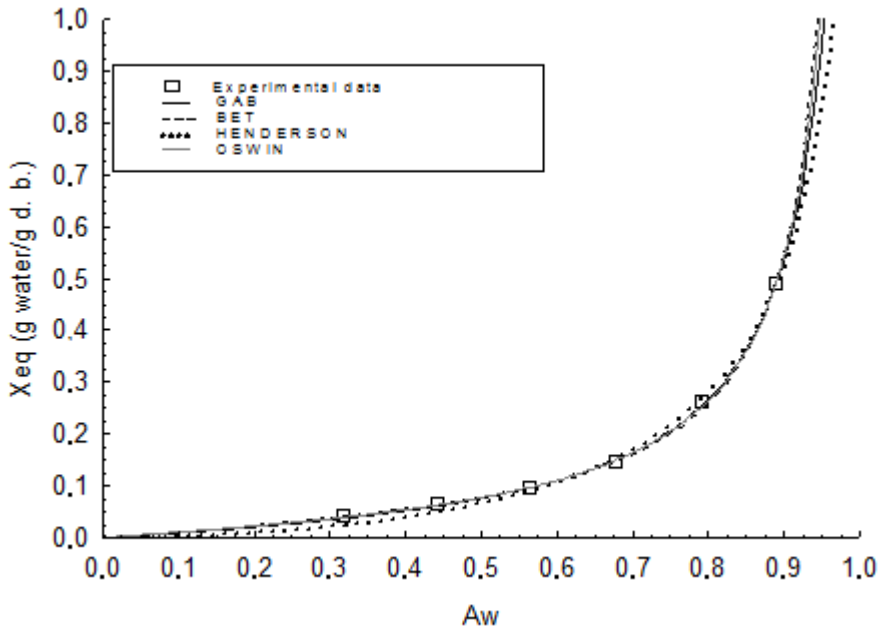

Figure 1. Adosrption isotherms at 25°C of lyophilized powder of grugru palm without addition of maltodxtrin (T1).

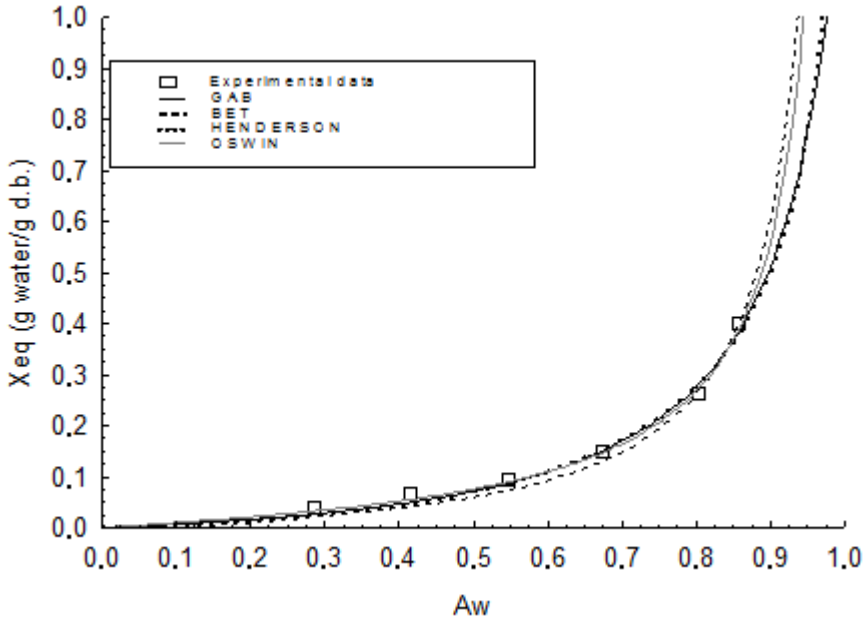

Figure 2. Adsorption isotherms at 25°C of lyophilized powder of grugru palm with addition of 8% maltodextrin (T2).

The parameters of the models applied to experimental data of adsorption isotherms of lyophilized powders of grugru palm, the determination coefficients (R^2) and the relative average deviations (*E*%) used as evaluation criteria for the representation of isotherms are shown in **Table 2**.

According to the results presented in **Table 2**, one notes that among the models studied (GAB, BET, Henderson and Oswin) all can represent the behavior of grugru palm powders because they showed representative R^2 e *E*%, respectively, from 0.9843% to 0.9996% and 0.23% to 1.57%, values below the criteria recommended by Aguerre *et al.* [20], where *E*% less than 10% indicates reasonable representative models, and by Labuza *et al.* [21], in which the representation of isotherms is considered extremely good for *E*% less than 5%. However, the GAB model (tri-parametric) represents more accurately the T1 treatment and the Oswin model (bi-parametric) the T2. Oliveira *et al.* [22] also obtained better adjustments of GAB model and Oswin model for lyophilized sapodilla.

The GAB model is reported by several researchers in forecasting adsorption isotherms for various dehydrated foods, as Ferreira and Pena [23] in obtaining adjustment of the model for peach palm powder; Silva *et al.* [24], for yellow mombin

powder; Kaymak-Ertekin and Gedik [8] for dehydrated grapes and potatoes; and Goula *et al.* [25], who obtained the best prediction for tomato powder.

The Oswin model did not fit to mathematic modeling in predicting the behavior of foods studied by Goula *et al.*[25] and Al-Muataseb *et al.* [26]. This report does not fit to that observed in T2 in this study, which showed R^2 = 0.9961 to 0.9979 and *E*% = 0.63 to 0.79. Kaymak-Ertekin and Gedik [8] also observed that the Oswin model presented data well correlated for apples and potatoes. Lomouro *et al.* (1985), quoted by Al-Muataseb *et al.* [27], reported that the Oswin model accounted for only 57% of the isotherms described for foods.

The values of moisture in the monolayer (X_m) in the GAB model for T1, between the temperatures of 25 and 40°C, has raised with increasing temperature (**Table 2**). Similar behavior was observed in the determination of adsorption isotherms of flour of peach palm (*Bactris gasipaes*) at 15°C [23] for surinam cherry powder at temperatures of 20° C and 40°C [28].

The values of water content in the monolayer with increasing temperature of T1 varied from 0.07 to 0.10 g water g^{-1} dry basis, and in T2 it was 0.07 to 0.08 g water g^{-1} dry basis; values of T2 has the same trend observed in the range presented by Rahman (1995), quoted by Cova *et al*. [29], which is from 0.05 to 0.08 g water g^{-1} dry basis for water content in the monolayer of starch rich products. The addition of maltodextrin in T2 may be the explanation for this treatment because it stands in the profile of starch rich products. However, Talla *et al.* [30] found values for banana, mango, pineapple between 0.080 and 0.185 g water g^{-1} dry basis, values higher than previously reported.

The values of *K* in this study were inferior to 1.0 at all temperatures, presenting little variation between 0.97 and 0.99. Vieira *et al.* [28], quoting Fernandez (1995), reported that values of *K* lower than 1.0 show a characteristic indicating that the isotherm for food products tends to an asymptote when activity equals to 1.0.

One can observe that increasing temperature from 25°C to 40°C leads a reduction in values of the constant *C*; a compatible behavior was observed by Gabas *et al.* [31] for pineapple powder with addition of maltodextrin, which demonstrates temperature is dependent to the constant *C*. These same authors reported that strong adsorbate-adsorbent interactions are favored by lower temperatures, reflecting on *C* increase and showing that this variation along with temperature may be the result of a mathematic compensation between *C* and *K*.

Table 2. Adjustment results of experimental data of adsorption isotherms at 25, 30, 35 and 40°C for lyophilized powders of grugru palm.

Models	Parameters[c]	T1[a] 25°C	30°C	35°C	40°C	Models	Parameters[c]	T2[b] 25°C	30°C	35°C	40°C
GAB	X_m	0.0705	0.0709	0.0825	0.1027	GAB	X_m	0.1122	0.1166	0.1156	0.0981
	C	1.2663	1.2072	0.9623	0.6949		*C*	0.5982	0.5626	0.5596	0.7402
	K	0.9791	0.9925	0.9886	0.9796		*K*	0.9278	0.9369	0.9459	0.9822
	R^2	0.9996	0.9993	0.9992	0.9996		R^2	0.9897	0.9900	0.9904	0.9965
	E%	0.23	0.31	0.40	0.29		*E%*	1.13	1.24	1.16	0.69
BET	X_m	0.0755	0.2301	0.0690	0.1579	BET	X_m	0.2241	0.1259	0.1387	0.1524
	C	1.4186	0.2770	1.3286	0.4451		*C*	0.3314	0.6404	0.5478	0.4968
	n	1.3371	-0.6423	1.0522	1.1523		*n*	-0.7423	1.3501	1.3105	1.2144
	R^2	0.9988	0.9916	0.9991	0.9994		R^2	0.9859	0.9961	0.9954	0.9976
	E%	0.39	1.39	0.40	0.34		*E%*	1.39	0.76	0.79	0.58
Henderson	a	0.5836	0.5591	0.5533	0.5450	Henderson	a	0.6155	0.6047	0.5942	0.5873
	b	3.3685	3.1554	3.0070	2.8519		b	3.5360	3.3701	3.2653	3.0546
	R^2	0.9951	0.9940	0.9950	0.9966		R^2	0.9843	0.9833	0.9828	0.9883
	E%	1.01	1.08	0.93	0.78		*E%*	1.45	1.62	1.57	1.29
Oswin	a	0.0773	0.0766	0.0801	0.0827	Oswin	a	0.0756	0.0775	0.0780	0.0837
	b	0.8859	0.9421	0.9687	1.0099		b	0.9120	0.9417	0.9647	0.9962
	R^2	0.9995	0.9993	0.9991	0.9993		R^2	0.9961	0.9958	0.9954	0.9979
	E%	0.26	0.32	0.40	0.31		*E%*	0.63	0.79	0.79	0.54

[a] T1: lyophilized powder without addition of maltodextrin; [b]T2: lyophilized powder with addition of 8% maltodextrin. [c]Abbreviations: R^2 = determination coefficient. *E%* = relative average error. X_m = moisture content in the monolayer (g H_2O g^{-1} dry basis). C = constant related to the heat of sorption in the molecular layer. K = GAB constant related to multi-layers. n = BET constant related to multi-layers. a e b = adjusting parameters of the Henderson and Oswin models.

In **Figures 3** and **4**, there is a graphical representation of the adsorption isotherms of moisture of grugru palm powder, for all temperatures studied, adjusted by GAB and Oswin model for T1 and T2, respectively.

There is little influence of temperature on equilibrium moisture content of grugru palm powder for water activity below 0.55 in T1, being observed from this point that for the same a_w there is an increase in the equilibrium moisture content along with rising temperatures (**Figure 3**). In T2, this effect was observed from water activity above 0.50 (**Figure 4**). In studies carried out with Surinam cherry powder, it was observed an influence of temperature on the equilibrium moisture content from a_w above 0.30 [28]; this shows that grugru palm powder presents a hygroscopicity below that observed in Surinam cherry powder.

As may be observed, the curves of **Figures 3** and **4** do not intersect themselves with the increasing temperature, a fact also reported by Kaymak-Ertekin and Gedik [8] for apples with high content of pectin and sugar, and for potatoes with high content of starch. This similar behavior of grugru palm powder to data of the previously cited authors is due to its sweet fruit with approximately 35% carbohydrates [32], being 13.3% glucose [33].

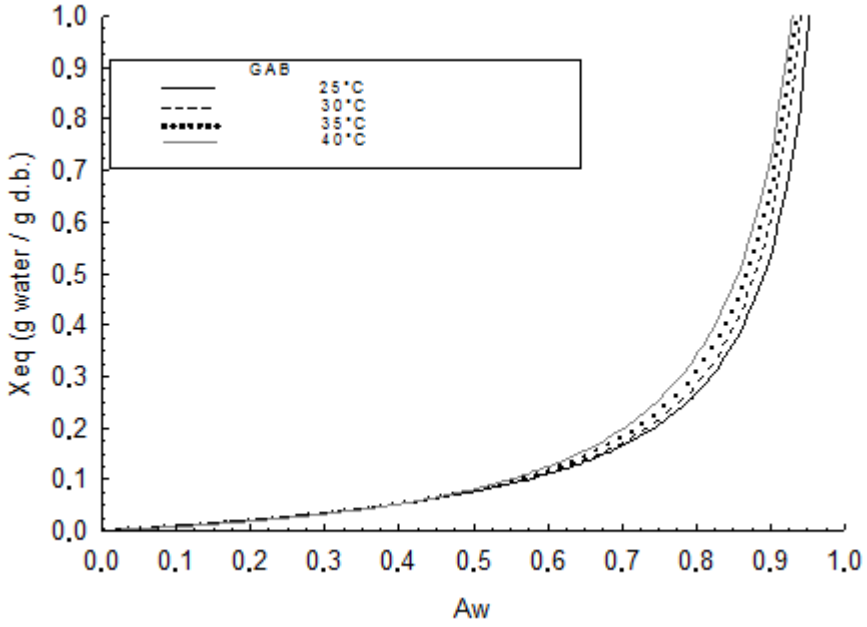

Figure 3. Best model (GAB) for adsorption isotherms at different temperatures of lyophilized grugru palm powder without maltodextrin (T1).

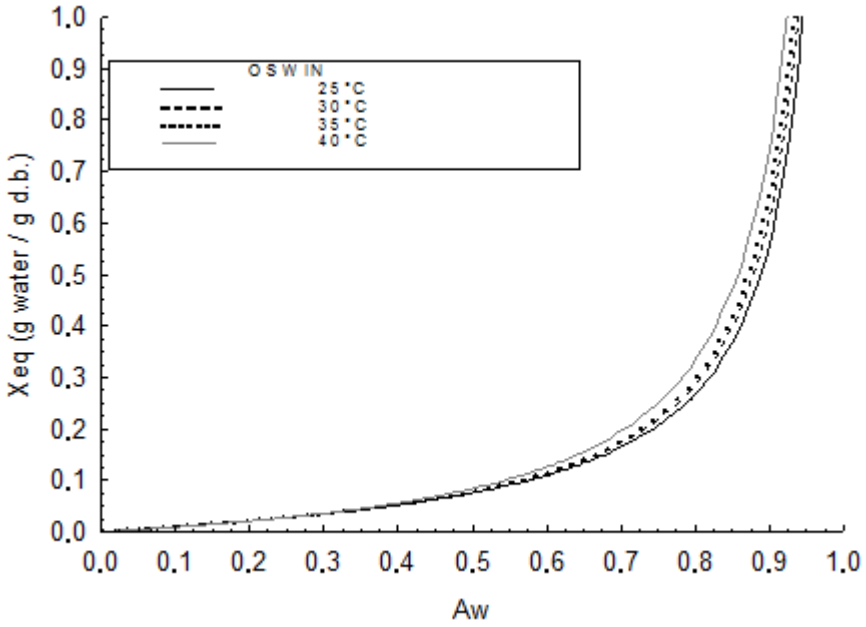

Figure 4. Best model (Oswin) for adsorption isotherm at different temperatures of lyophilized grugru palm powder with 8% maltodextrin (T2).

3.2. Hygroscopicity and Degree of Caking

Table 3 shows the values of hygroscopicity and degree of caking for T1 and T2. Treatment T1 presented greater caking in relation to T2. This fact may be related to the addition of drying adjuvant, maltodextrin, in which its use entails a reduction in the powder hygroscopicity; this fact is also evidenced in adsorption isotherms of this work.

Table 3. Hygroscopicity and degree of caking of lyophilized powders from grugru palm.

Treatment[a]	Hygroscopicity (%)	Degree of caking (%)
T1	7.68 a*	0.33 a
T2	6.86 a	0.09 b

[a] T1 = lyophilized powder without maltodextrin; T2 = lyophilized powder with 8% maltodextrin. * Equal lower-case letters in the same column do not differ by Tukey test at 5% probability.

Powder caking is an undesirable phenomenon, which initially consists of transformation of powder into solid and sticky material, resulting in decreased

functionality and fluidity, so quality loss, and the main cause of agglomeration is the presence of water induced by plasticization of the surface of particles [34].

In accordance with GEA Niro Research Laboratory [18], in which is characterize the hygroscopic behavior and degree of caking, respectively, the grugru palm powder is classified as a non-hygroscopicity and non-caking product, Oliveira *et al.* [35] showed similar response about this classification (hygroscopicity 5.17% and 6.39%; degree of caking 0.03% and 3.11%) for powder grugru palm obtained by oven dryer.

In studies carried out with sucrose, Mathlouth and Rogé [7] reported the formation of agglomerates is related to the solid bridges created during the process, which turns out sucrose more hygroscopic. The same can be observed in T1, where the powder formed the greatest caking and consequently making the grugru palm powder more hygroscopic.

Costa *et al.* [36] stated that different variations in the hygroscopic behavior of food powders are also attributed to their size, as finer particles have higher contact surface and therefore higher number of active sites. This report may explain the non-hygroscopicity behavior of grugru palm powder when compared to that of fruits found in the literature, which showed high hygroscopicity, as the particle size of grugru palm powder is 1200 µm and the particle size of most fruit powders is in the range from 500 to 600 µm.

4. Conclusions

Mathematical models of adsorption isotherms for grugru palm powders can be classified as Type III, according to Brunauer classification.

All models studied adjust themselves to lyophilized powder of grugru palm, and GAB and Oswin models represent best the behavior of the adsorption isotherms for T1 and T2, respectively.

Grugru palm powder showed an increase in the humidity of the monolayer X_m along with increasing temperature. The values of *C* were reduced along with increasing temperature. The constant *K* suffered small variations in function to temperature.

Lyophilized powders of grugru palm were characterized as non-hygroscopic and non-caking product.

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Stability of bioactive compounds and quality parameters of grugru palm powder (*Acrocomia aculeata*) in different drying conditions

Stability of bioactive compounds of grugru palm powder

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Abstract

The objective was to study the stability of physical, physicochemical and bioactive components of grugru palm powder. The fruits were selected, sanitized and pulped; subsequently, the pulp was crushed and submitted to four treatments: oven drying (T1: without maltodextrin; T2: with 8% of maltodextrin) and freeze-drying (T3: without maltodextrin; T4: with 8% of maltodextrin). The powders of each treatment were packed in: P: plastic; L: laminate; LV: laminate and vacuum packed. Physical, physicochemical and bioactive compounds analyses were made every 30 days during 120 days of storage. An increase in moisture content and water activity was observed, and a fluctuation occurred in color saturation and in values of phenolic and flavonoids. The content of β-carotene decreased during the storage, and their values were influenced by the dehydration method and by the type of package. Thus, the optimal condition for preserving the grugru palm powder is freeze-drying T3 in packaging LV.

Keywords: *Acrocomia aculeata*, dehydration, preservation, packaging, β-carotene.

Practical application

Acrocomia aculeata is a palm tree highly productive and with great economic value. All plant parts are utilized and its fruits have an interesting nutritive potential. The mesocarp of grugru palm fruit provides significant concentrations of β-carotene, a natural antioxidant and pro-vitamin A; so the pulp can be included in feeding programs of the poor and contribute to the food enrichment as a source of vitamin A. This work contributes to the study of the stability of bioactive compounds and quality parameters of dehydrated grugru palm pulp, aiming to define the best processing and conservation conditions for applying to this raw material as a new ingredient in different formulations of food products.

1. Introduction

The palm species is very important to the tropical region due to the wide variety of products that can be obtained from (Lorenzi, 2006). Many palm trees native to the Amazon region and other tropical regions in Latin America have been subject to research and development (Clement et al., 2005). Brazil's arid and semiarid regions have a wide variety of oil plants, native or exotic, among which grugru palm (*Acrocomia acuelata*) is highlighted because it is highly productive (Rezende, 2009) and potentially nutritive (Ramos et al., 2008; Oliveira et al., 2013a).

Grugru palm, according to Oliveira et al. (2013), presents a high respiratory rate, making it a highly perishable fruit. Thus, processing and storage techniques have to be applied to obtain a longer useful life. The choice of the technique should be careful in order to keep the nutritional characteristics of the fresh product into the processed products. One of the processes most commonly used in food preservation is drying (Park et al., 2002); in this context it is very important to know the nutritional properties of the raw material (Park et al., 2006).

Several studies are carried out with grugru palm; however, most of these studies are focused on the production of biofuel or edible oil, especially the high content of oleic acid from the mesocarp. This is also used for industrial purposes as for the manufacture of soap (Ciconini et al., 2013; Silva and Andrade, 2013); this way many studies are aimed at the characterization of the oil and its extraction method. The related literature reports the use of dehydrated pulp in the preparation of food products (Brasil, 2002): the powder is utilized for preparation of various foods; however, no study on the

techniques of drying and quality analysis of this powder was found. Thus, it is necessary to know the behavior of nutrients of grugru palm pulp through the processes of drying and storage period in order to use this raw material in the preparation of food products.

This paper presents a study of the stability of the physical, physicochemical and bioactive components of grugru palm powder obtained by two methods of drying, packaged in different containers and stored at room temperature (± 25° C) for 120 days.

2. Material and Methods

2.1 Raw Material

The grugru palm fruits were harvested in the Araripe Plateau, Cariri region, Ceara State, Brazil, and taken to the Laboratory of Food Quality Control and Drying.

The fruits were selected and sanitized; subsequently, they were pulped. The pulp was stored in a freezer at -20° C until the drying process.

The pulps were unfrozen, crushed and separated into two pieces for drying (Figure 1).

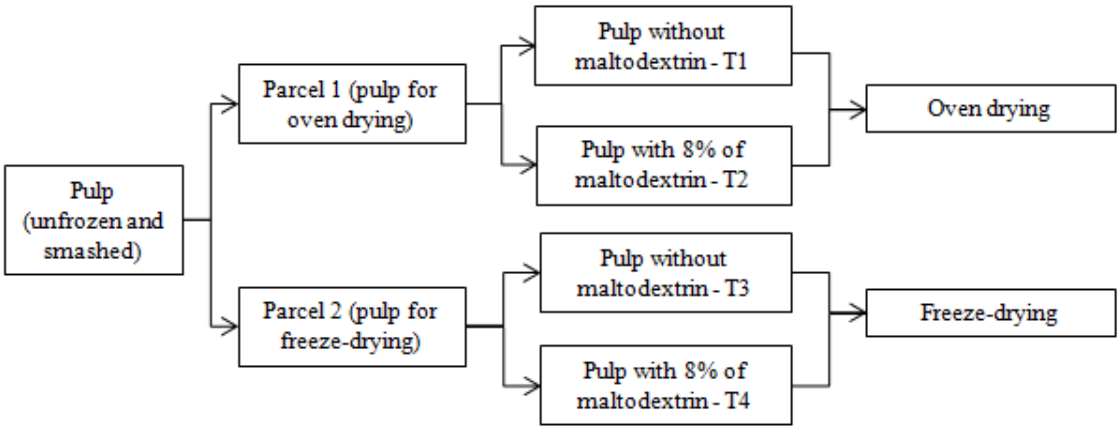

Figure 1. Flowchart identifying the treatments and the drying methods.

2.2. Application of maltodextrin

An aqueous solution of maltodextrin was prepared as follows: for each 100g of pulp, 8.0 g of maltodextrin, dextrose equivalent (DE = 20), were dissolved in 10 ml of drinking water. Afterward, this solution was added in the pulps of T2 and T4 and homogenized.

2.3. Oven drying

The previously prepared pulps (T1 and T2) are separately distributed on stainless steel trays with 25 cm diameter and taken to an oven with air circulation at 65° C for 25 hours (Oliveira et al. 2012). After drying, the product was taken to a rotary slicer (MA 048, Marconi) to obtain a homogeneous particle size product which was passed through a sieve with a mesh of 0.12 cm.

2.4. Lyophilization

The earlier prepared pulps (T3 and T4) were separately distributed on stainless steel trays with 25 cm diameter and taken to an ultra-freezer (LC 90-40V, Terroni) at -40° C for 24 hours; thereafter, they were dried in a bench-top lyophilizer (LS 3000, Terroni) for 25 hours (Oliveira et al. 2013b). After drying, the product was taken to a rotary slicer (MA 048, Marconi) to obtain a homogeneous particle size powder, which was passed through a sieve with mesh of 0.12 cm.

2.5. Storage

After obtaining the powder, each treatment was packed in the following packages: plastic - P, laminate – L, and laminate with vacuum packaging - LV.

Three samples of each treatment containing 100g of powder and stored at room temperature (± 25 º C) were reserved and analyzed at the time zero and at every 30 days for a period of 120 days.

Container specifications:

Plastic: PET + POLI/PET + PE transparent film 80g m^{-2}: weight 100g m^{-2}. Dimension: 195(height) x 100(length) x 60(width).

Laminate: aluminum/PET 17g m^{-2} + ADES 2g + aluminum 21,6 + ADES 2g + PE film 80g m^{-2}: weight 122g m^{-2}. Dimension: 180(height) x 80(length) x 50(width).

2.6. Physical and physicochemical analyses

The moisture content was determined gravimetrically at 105° C in an oven until constant weight according to the technique described by Instituto Adolfo Lutz (2005). The water activity (a_w) was determined by a portable measurer (AquaLab®).

The samples colors were determined by a colorimeter (Minolta, model CR410, Konica Minolta Sensing, Inc., Japan) in the mode CIE L*a*b* and parameters D65 (Y = 93.9; x = 0.3167; y = 0.3336).

2.7. Bioactive compounds

The total phenolics were extracted and determined according to the method described by Bucic-Kojic et al. (2007). The yellow flavonoids were extracted in accordance to Francis (1982), through a solution of ethanol:HCl 1.5 mol L^{-1} (85:15).

Para a determinação da Vitamina A foi realizado a extração do β-caroteno e a atividade vitamínica A do β-caroteno foi obtida segundo Bauernfeind (1972), no qual foi utilizado o cálculo do valor de vitamina A fornecido pela National Council Reserch (1980) em que 6µg de β-caroteno correspondem a 1 Retinol Equivalente (RE).

The extraction of β-carotene was carried out as described by Rodriguez-Amaya (1999) with a little modification. Samples (5g) sample were extracted with cold acetone until the residue becomes colorless; then it was filtered under vacuum using a Büchner funnel. The pigments extracted were then transferred to petroleum ether and washed in distilled water until complete removal of the acetone. The extract concentration was made in a rotatory evaporator at 35º C. The pigments were dissolved in acetone, filtered in a PTFE with 0.22 µm membrane and then analyzed. The β-carotene content was analyzed by HPLC chromatographic conditions developed by Sant'Ana et al. (1998) with modifications. The devices utilized are: a liquid chromatograph (model Gilson 321 pump) with automatic injector (model Gilson 234) and handle of 20µL (loop), column ACE 5 C18 (5µm, 150x4.6 mm), visible-UV detector at 450nm (model Gilson 152). The mobile phase was composed of methanol/acetonitrile/ethyl-acetate (80:10:10) at a rate of 1.5 $ml.min^{-1}$ with running time of 60 minutes. The quantification of β-carotene was attained by external standard curves.

2.8. Data Analysis

For the stability analysis, three samples of each packaging type and treatment were separated. All analyses were performed in triplicate (n = 3). Data were analyzed statistically using the analysis of variance (ANOVA), and the differences between the averages were determined by the Tukey test at 5% probability using the software Statistic, version 7.0 (Statsoft, 2007).

3. Results and Discussion

The determination of moisture content and water activity in food is important because these are factors that support microbial growth, and according to Ribeiro and Seravalli (2007), low values of these factors will provide the conditions for food stability and preservation. The values for moisture content (g $100g^{-1}$) and water activity are shown in Table 1. All powder moisture values at the zero time storage are in accordance to the Brazilian Legislation (Brasil, 1978), which allows a maximum of 5g $100g^{-1}$. According to FAO (1995), different moisture contents are reported for several dehydrated foods.

Table 1. Stability of moisture content and water activity in grugru palm powder stored in different containers at room temperature (25 º C) for 120 days.

Treatment*	Packaged**	Umidade (g $100g^{-1}$)			Atividade de água - a_w		
		0 day	30 days	120 days	0 day	30 days	120 days
	P	$3.28^{ab\#}$	4.96^{b}	7.27^{a}	0.20^{a}	0.29^{b}	0.41^{b}
T1	L	3.28^{ab}	3.77^{def}	5.96^{c}	0.20^{a}	0.18^{de}	0.22^{de}
	LV	3.28^{ab}	3.68^{ef}	3.85^{f}	0.20^{a}	0.17^{e}	0.18^{gh}
	P	3.40^{ab}	5.22^{ab}	7.19^{a}	0.22^{a}	0.29^{b}	0.39^{b}
T2	L	3.40^{ab}	4.03^{cde}	4.63^{d}	0.22^{a}	0.20^{d}	0.23^{de}
	LV	3.40^{ab}	3.93^{de}	4.11^{ef}	0.22^{a}	0.19^{de}	0.20^{fg}
	P	3.12^{b}	5.39^{a}	7.42^{a}	0.12^{b}	0.33^{a}	0.44^{a}
T3	L	3.12^{b}	3.78^{def}	4.25^{e}	0.12^{b}	0.18^{de}	0.20^{fg}
	LV	3.12^{b}	3.55^{f}	4.03^{ef}	0.12^{b}	0.17^{e}	0.17^{h}
	P	3.48^{a}	5.23^{ab}	6.56^{b}	0.22^{a}	0.34^{a}	0.41^{b}
T4	L	3.48^{a}	4.34^{c}	4.78^{b}	0.22^{a}	0.25^{c}	0.28^{c}
	LV	3.48^{a}	4.11^{cd}	4.01^{ef}	0.22^{a}	0.24^{c}	0.23^{d}

* T1: oven drying without maltodextrin; T2: oven drying with 8% of maltodextrin; T3: freeze-drying without maltodextrin; T4: freeze-drying with 8% of maltodextrin.
** P: plastic package; L: laminate package, LV: laminate with vacuum packaging.
*** Same lowercase letters in the column and same uppercase letters in the line do not differ by the Tukey test ($p>0.05$) in the analyses.

The values of moisture and water activity during the storage time for all treatments and packages were gradually increasing, except for water activity in T1 and T2 packed in LV, which showed decreasing values. This may have been influenced by the vacuum closed laminate package. Costa et al. (2013) studied the passion fruit powder and also observed an increase in moisture content and water activity, corroborating the findings of this study. At the end of the 120 days of storage, all treatments stored in plastic - P and T1 with laminate package extrapolated the maximum moisture allowed by the legislation; so the package LV was the only one effective against to increasing moisture content and water activity of the powders in this study. The low water activity levels support the powder security, since reactions (chemical and enzymatic) is reduced or halted under the conditions given in this study. Furthermore, according to FAO (1995), water activity values below 0.4 prevents caking when the products are stored at room temperature.

The color of food products is an important evaluation parameter; it directly represents the quantity of certain bioactive compounds (e.g. carotenoids) and it has an attractive power to the consumer. The color parameters (L*, a* and b*) are presented in Figure 2.

The parameter L* (0: black and 100: white) determines the brightness of the product. The T2 powder at zero time was the darkest one in relation to the other treatments and during the storage period minor changes occurred. The T1 showed the most visible changes of brightness in the samples at the end of the experiment: the plastic package (P) did not change, while L package showed trends for whitening and LV for darkening.

The parameters a* and b* are the chromatic coordinates: -a* represents the direction to green and +b* the direction to yellow. One can observe that the values for -a* were low, demonstrating a grayish tonality; this coordinate showed a non significant decrease during the storage time. The chromaticity +b* in all treatments showed a yellow tone, with T3 and T4 presenting a more saturated color in relation to T1 and T2. T1 suffered the greatest loss of yellow saturation (discoloration) during the storage time. An effectiveness of packing on the conservation of powder color was observed: the package LV kept the yellow color saturation. Qian et al. (2012) stated that appropriate storage conditions for products containing β-carotene prevent color fading and loss of bioactive potential.

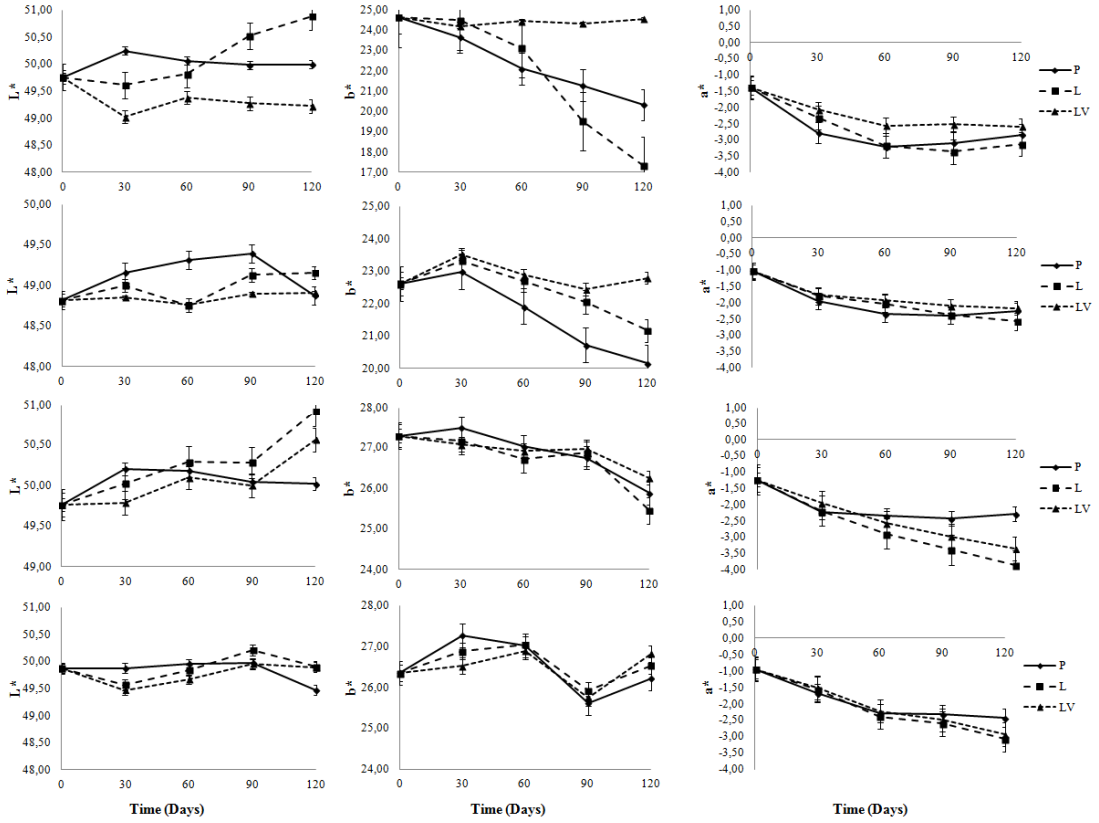

Figure 2. Stability of color parameters (CIE L*a*b*) under different treatments and drying methods with three types of packages at room temperature for 120 days of storage.

(T1: oven drying without maltodextrin; T2: oven drying with 8% of maltodextrin; T3: freeze-drying without maltodextrin; T4: freeze-drying with 8% of maltodextrin. P: plastic package; L: laminate package, LV: laminate with vacuum packaging. Color parameters: brightness - *L; chromaticity - *a and *b).

Figure 3 shows the stability of β-carotene content in the grugru palm powder. Grugru palm β-carotene is highly bioavailable when compared to pure β-carotene (Ramos et al., 2007). The β-carotene in the plant kingdom is present in vegetables with dark-green leaves and in yellow to red fruits (Uenojo et al., 2007; Tang et al., 2009). The pulp dehydration process influences the amount of this carotenoid: treatments T3 and T4, which were lyophilized, presented higher levels of β-carotene in relation to the oven dried pulps (T1 and T2). Another factor influencing this carotenoid content was the addition of 8% of maltodextrin. The addition of this drying adjuvant may have hindered the extraction of β-carotene. Costa et al. (2013) observed a decrease in the

content of β-carotene in passion fruit powder. The β-carotene is susceptible to the action of light, heat and oxygen, and can undergo auto-oxidation (Lin & Chen, 2005).

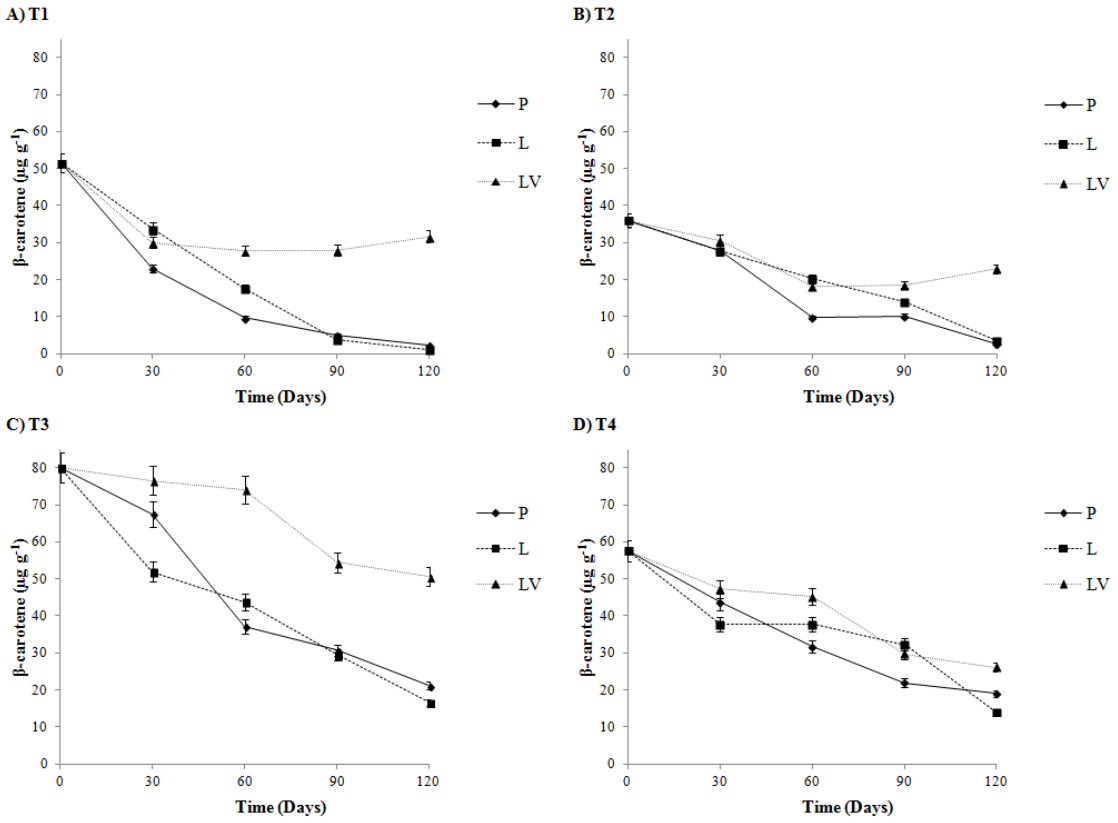

Figure 3. Stability of β-carotene (µg.g^{-1}) under different treatments and drying methods packaged in three types of packaging at room temperature during 120 days of storage.

(A-T1: oven drying without maltodextrin; B-T2: oven drying with 8% of maltodextrin; C-T3: freeze-drying without maltodextrin; D-T4: freeze-drying with 8% of maltodextrin. P: plastic package; L: laminate package, LV: laminate with vacuum packaging. Color parameters: brightness - *L; chromaticity - *a and *b).

During the storage period, the β-carotene has degraded regardless of treatment and packaging. P and L packages were not effective as to losses, and the LV package was the best in the conservation of grugru palm powder. According to FAO (1995), the vacuum packaging prevents the action of atmospheric oxygen, especially in products containing β-carotene.

The phenolic compounds present biochemical properties that are important to human health (Meot-Duros & Magne, 2009); this bioactive component is subject to

some losses after food processing. According to Patras et al. (2011), during the fruit processing, the cells are ruptured and the pulp undergo non-enzymatic oxidation, resulting in loss of phenolic compounds. However, as shown in Figure 4, the phenolic components were not influenced by the different kinds of drying and the addition of 8% of maltodextrin did not affect the results. The storage time for all treatments caused a reduction in phenolic levels at the end of 120 days, and the analysis every 30 days of storage confirmed that only the 90-day period showed a significant difference ($p>0.5$) between LV and P packaging in the treatment T2, being LV the highest value; in the other storage periods, there was no difference between packages for each treatment, and the same behavior was observed by Bakowska-Barczak and Kolodziejczyk (2011) in the study on strawberry powder encapsulated with maltodextrin with different DE (11, 18, 21).

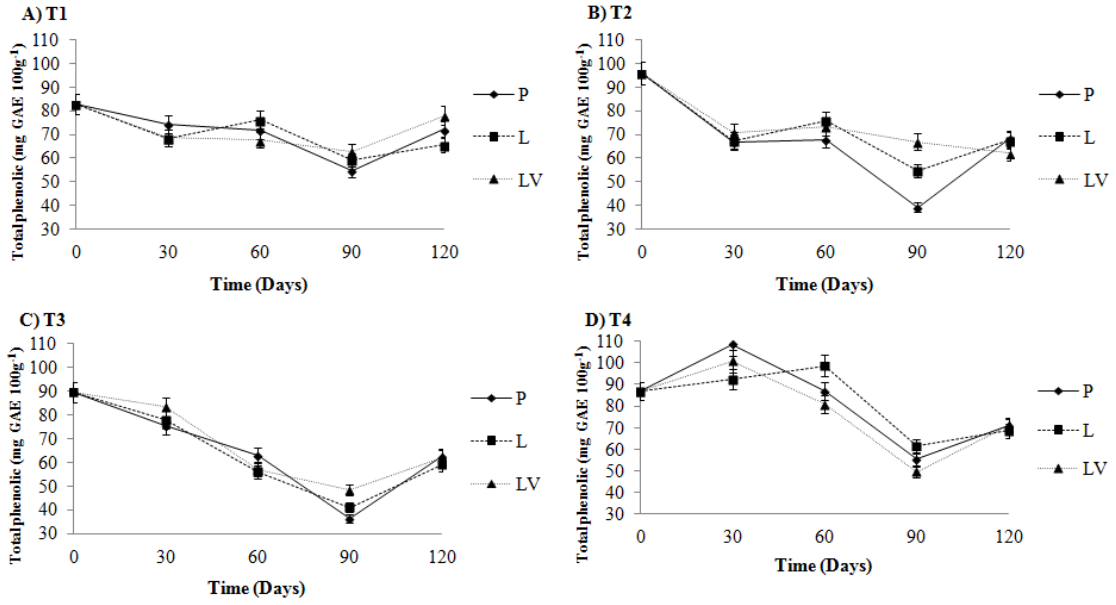

Figure 4. Stability of total phenolic content (mg.100g^{-1}) under different treatments and drying methods packaged in three types of packaging at room temperature for 120 days of storage.

(A-T1: oven drying without maltodextrin; B-T2: oven drying with 8% of maltodextrin; C-T3: freeze-drying without maltodextrin; D-T4: freeze-drying with 8% of maltodextrin. P: plastic package; L: laminate package, LV: laminate with vacuum packaging. Color parameters: brightness - *L; chromaticity - *a and *b).

Figure 5 shows the levels of yellow flavonoids in different grugru palm powders. A slight value fluctuation was observed in all treatments with an increase at 120 days of storage. Costa et al. (2013) found the same behavior in passion fruit powders during the storage time. The T1 and T2 showed the highest values in comparison to the lyophilized powders; this fact may have been due to the freezing step before grugru palm pulp was submitted to freeze-drying. The content of flavonoids in processed products degrades, making it smaller than in fresh fruits (Huber & Rodriguez-Amaya, 2008). Each processing stage (peeling, pulping, pre-processing and bleaching) of vegetables causes a loss of flavonoids (Ewald et al., 1999).

The addition of 8% of maltodextrin in T2 and T4 showed no positive effects for the stability of bioactive compounds and for the quality parameters in relation to treatments without maltodextrin (T1 and T3); thus, further studies should be conducted to justify its use in the process of drying grugru palm pulp.

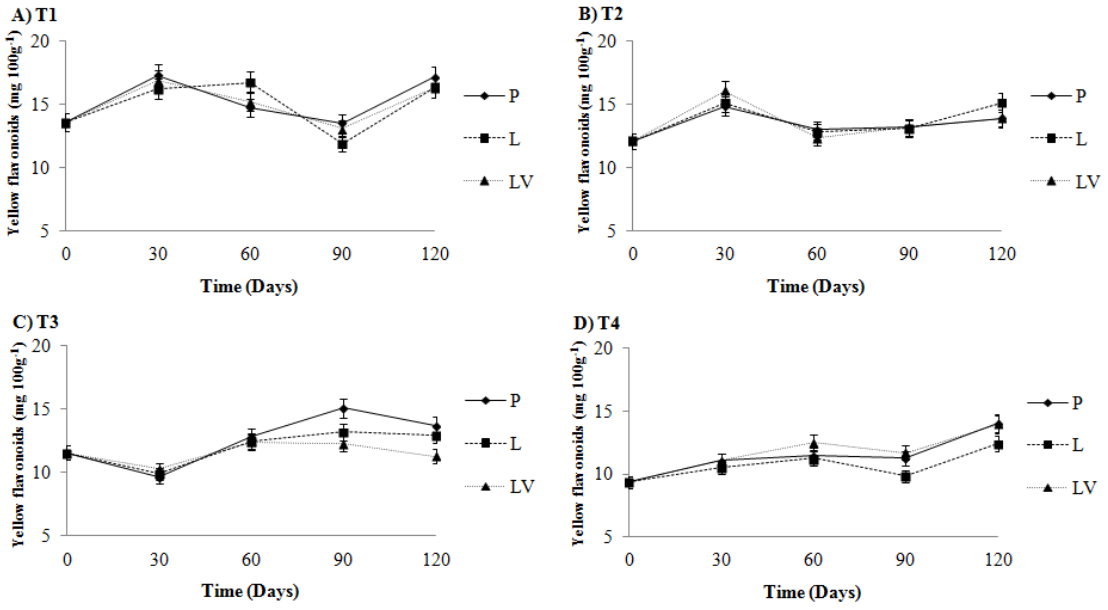

Figure 5. Stability of yellow flavonoids (mg.100g^{-1}) under different treatments and drying methods packaged in three types of packaging at room temperature for 120 days of storage.

(A-T1: oven drying without maltodextrin; B-T2: oven drying with 8% of maltodextrin; C-T3: freeze-drying without maltodextrin; D-T4: freeze-drying with 8% of maltodextrin. P: plastic package; L: laminate package, LV: laminate with vacuum packaging. Color parameters: brightness - *L; chromaticity - *a and *b).

Conclusion

The stability of bioactive compounds and the quality parameters of grugru palm powder changed during the storage time: moisture, water activity, color and content of β-carotene were affected by the treatments applied and by the packages used in the storing period.

The ideal conditions for the preservation of the grugru palm powder that ensures greater stability of bioactive compounds and quality attributes are freeze-drying without addition of maltodextrin (T3) and vacuum packaged (LV).

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