



Characterization and development of co-crystallized lemon juice powder for a caipirinha-type beverage

Caracterização e desenvolvimento de suco de limão em pó co-cristalizado para uma bebida tipo caipirinha

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This study aimed to develop and characterize co-crystallized lemon juice powder produced using sucrose syrup and to evaluate its feasibility as an ingredient for preparing a caipirinha-type beverage. Co-crystallization was carried out using lemon juice with different soluble solids concentrations (20 and 40 °Brix) and varying juice addition levels (7 and 10%, w/w). The study showed that the 40° Brix provided the most suitable condition and obtained 70% (w/w) of yield with desirable physicochemical properties (low moisture content and water activity). The co-crystallized powders produced using lemon juice at 40° Brix containing 7 and 10% lemon juice were selected for further characterization and used to prepare a caipirinha-type beverage, which was compared with a commercially available powdered caipirinha mix in a sensory evaluation. SEM, FTIR, and XRD results showed that lemon juice was encapsulated by sugar, maintaining the crystalline structure. DSC analysis showed that the co-crystallization process enhanced the thermal stability of the encapsulated lemon juice. No significant differences in sensory acceptance were observed between the beverages prepared with the co-crystallized powders at a 5 % significance level ($P > 0.05$), but compared to the commercial product, they showed satisfactory scores. The sensory evaluation confirmed that co-crystallized lemon juice is a promising ingredient for natural powdered beverage formulations, aligning with the clean-label trend by avoiding artificial additives.

Keywords: sensory acceptance, brix, encapsulation.

Este trabalho teve como objetivo desenvolver e caracterizar um pó de suco de limão co-cristalizado com xarope de sacarose e avaliar sua viabilidade como ingrediente para a o preparo de uma bebida tipo caipirinha. Os experimentos de co-cristalização foram realizados utilizando diferentes concentrações de sólidos solúveis (20 e 40 °Brix) e variações na adição de suco de limão (7 e 10% p/p). Os resultados deste estudo mostraram que a concentração de 40° Brix proporcionou a condição mais adequada e obteve 70% (p/p) de rendimento com propriedades físico-químicas desejáveis (baixo teor de umidade e atividade da água). Os pós co-cristalizados produzidos com 40° Brix contendo 7 e 10% de suco de limão foram selecionados para caracterização adicional e utilizados no preparo de uma bebida tipo caipirinha, a qual foi comparada com uma mistura comercial em pó para caipirinha em avaliação sensorial. Os resultados de MEV, FTIR e DRX mostraram que o suco de limão foi encapsulado pelo açúcar, mantendo a estrutura cristalina. A análise de DSC mostrou que o processo de co-cristalização aumentou a estabilidade térmica do suco de limão encapsulado. As bebidas co-cristalizadas não apresentaram diferenças significativas de aceitação sensorial a um nível de significância de 5% ($P > 0,05$), mas, em comparação com o produto comercial, apresentaram pontuações satisfatórias. A avaliação sensorial confirmou que o suco de limão co-cristalizado é uma alternativa promissora para formulações de bebidas em pó naturais, alinhando-se com a tendência de rótulos limpos, evitando aditivos artificiais.

Palavras-chave: aceitação sensorial, brix, encapsulação.

1. INTRODUCTION

The alcoholic beverage industry is currently one of the most promising sectors of the food industry, as the global market is projected to grow by approximately 37% by 2028, reaching a value of more than USD 2.1 trillion, compared with 1.53 trillion dollars in 2022 [1]. According to Maharjan (2019) [2], the consumption of spirits and beer increased by 50% in liters per capita. This growth may be attributed to several factors, whether to the expansion in non-traditional markets or driven by consumer preferences, where alcohol consumption represents a common thread in social gatherings and food culture.

Within the alcoholic beverage industry, there has been worldwide growth in innovation in flavored ready-to-drink beverages, typically consist of vodka, gin, tonic water, mixed with spices and artificial aromas, and others such as concentrated fruit preparations to be combined with the consumer's preferred distilled beverage. However, these different products on the market use a lot of artificial aromas and food additives to maintain product quality and achieve the desired sensory profile. According to Fracassetti et al. (2019) [3], innovation in the alcoholic beverage sector remains challenging when the preoccupation is about sustainable approaches and working with low-cost technologies. In addition, the profile of consumers has changed over the years, showing themselves to be more demanding to clean label products, seeking drinks that contain sensory properties that are more pleasant to the palate and with the most natural appearance possible.

The alcoholic beverage industry must assess the technical feasibility of developing increasingly attractive, low-cost products to meet consumer demands for convenience and sensory quality. In this sense, microencapsulation techniques have been widely applied to preserve several functional compounds in foods and beverages. According to Soumya et al. (2021) [4] microencapsulation is a process in which tiny particles or droplets are surrounded by a coating or embedded in a homogeneous matrix giving small capsules with many valuable properties and providing a physical barrier between the core compound and other product components. Spray drying is the most common method used by encapsulation, and this is influenced by wall material to the active ingredient and solution atomized in a drying chamber at elevated air temperatures to form microparticles/capsules [5, 6].

Although spray drying is the most widely used technique in the preserving natural flavors and other bioactive compounds [7, 8], co-crystallization is an emerging encapsulation technology that has attracted several studies due to its excellent results in preserving certain compounds and obtaining value-added products [9]. According to Chezanoglou and Goula (2021) [10], the co-crystallization process includes preparing supersaturated sucrose solution, incorporating the core material, heating the crystallization temperature, and uniform mixing. The products have a porous structure with excellent solubility, hygroscopicity, and flowability properties. Several studies have reported the application of co-crystallization in some juice fruits, soluble fibers, aqueous extracts, and pigments [11-14]. However, few studies have investigated the application of co-crystallized products in beverages, mainly for alcoholic drinks [9, 15].

In response to this growing trend, and considering that consumers increasingly seek convenience, ready-to-drink beverages, and new sensory experiences, powdered formulations inspired by traditional cocktails have emerged on the market. Among these, caipirinha, one of Brazil's most iconic cocktails, traditionally prepared with lemon juice, sugar, ice, and cachaça (or vodka) [16], is also commercially available as a powdered beverage mix for rapid preparation. However, these commercial products generally contain numerous additives, including maltodextrin, flavorings, acidulants, stabilizers, anti-caking agents, antioxidants, and colorings, in addition to sugar and dehydrated lemon solids, to ensure technological stability and sensory quality.

Unlike conventional encapsulation techniques, which require carrier agents not traditionally present in the beverage, co-crystallization uses sucrose as the encapsulating matrix, which is already an essential ingredient in the traditional formulation of caipirinha. This approach may reduce the need for additional carrier agents while preserving the natural characteristics of lemon juice and producing a stable powdered ingredient suitable for beverage preparation. Therefore, this study aimed to develop and characterize co-crystallized lemon juice powder produced using

sucrose syrup and to evaluate its potential application as a natural ingredient in a caipirinha-type beverage. The resulting co-crystallized powder was further applied in the preparation of a caipirinha-type beverage and compared with a commercially available powdered caipirinha mix to evaluate its sensory acceptance.

2. MATERIAL AND METHODS

2.1 Materials

Commercial sucrose, microcrystalline refined sugar, (Alto Alegre, Presidente Prudente, SP, Brazil) was used as an encapsulating matrix. The food-grade potassium citrate was obtained by Adicel Ind. Ltda (Belo Horizonte, MG, Brazil) and fresh lemon Tahiti (*Citrus latifolia*) was acquired in a local supermarket (Maringá, PR, Brazil).

2.2 Preparation of lemon juice

Initially, the lemon were washed and sanitized by immersion in a 200 ppm chlorine solution for 15 min, and then were cut, and a manual juice squeezer extracted the juice. The peels were reserved and dried in oven air circulation at 50 ± 2 °C until constant moisture content. The dried peels were crushed in a knife mill at 35 mesh (ACB Labor, Goiânia, GO, Brazil), stored in glass containers at 25 °C, and reserved to the mixture in the final product co-crystallized in sensory analyses.

The lemon juice was concentrated on a hot plate at 70°C until the desired soluble solids content (°Brix) for the co-crystallization process was reached. The °Brix was measured using a portable refractometer (RZ-117, São Paulo, SP, Brazil) at 20 °C [17]. The pH of the lemon juice was also measured to control the appropriate pH for the co-crystallization [17].

2.3 Co-crystallization process

The co-crystallization process of lemon juice, illustrated in Figure 1, was investigated using two variables: the concentration of soluble solids (20 and 40% w/w) and the amount of juice added (7 and 10% w/w). The pH of the concentrated lemon juice was adjusted to 4.5, using food-grade potassium citrate INS 332ii, based on preliminary tests, which favoured crystal formation and stability [18]. The sugar syrup was prepared at 70 °Brix and preheated in an aluminium pan until supersaturation point (128 ± 2 °C), using a hot plate coupled to a propeller-type mechanical vertical stirrer at 15,000 rpm (Fisatom, 713 model, São Paulo, SP, Brazil). Once the sucrose syrup exhibited slight turbidity at 128 °C, the concentrated juice was rapidly added (based on the weight of the sucrose syrup) under continuous stirring at 3,500 rpm. The syrup containing lemon juice was then cooled in a cold bath to increase sugar supersaturation, which immediately facilitated crystallization and avoided exposure to higher temperatures. The resulting crystals agglomerates were spread on aluminium trays and dried in an oven at 50 °C (according to preliminary tests) until constant weight. After drying, the co-crystallized products were ground to 35 mesh to obtain a uniform particle size. The samples were stored in glass containers for further analysis.

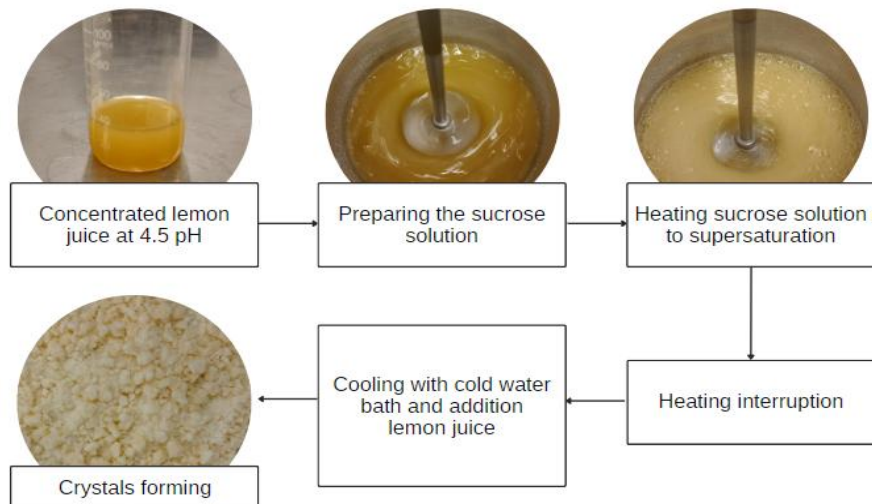


Figure 1: Schematic diagram of the co-crystallization process of lemon juice with sugar.

2.4 Characterization of co-crystallized

2.4.1 Yield of lemon juice crystals and co-crystallization time

The yield of the co-crystallized lemon juice powder was determined based on the initial mass of the syrup and lemon juice before co-crystallization and the final mass of crystals formed. The co-crystallization time was determined according to the methodology proposed by Andrade Pizarro et al. (2017) [11], based on the interval from the beginning of lemon juice addition to the sucrose syrup until the completion of the process, when crystal formation was observed.

2.4.2 Moisture content and water activity

The moisture content (%) was determined gravimetrically at 105 °C [17], and the water activity (aw) was measured using an electronic water activity meter (Aqua-lab, Series 4TE, Meter Group Latam Ltda, USA) at 20 °C.

2.4.3 Solubility and Hygroscopicity

The solubility of the co-crystallized products was evaluated according to the methodology described by Beristain et al. (1996) [19], which corresponds to the time required for the complete dissolution of 10 g of co-crystallized product in 100 mL of distilled water at 25°C, under constant stirring at 50 rpm, until no solid particles were observed in the liquid phase.

The hygroscopicity of the samples was determined according to the methodology described by Cai and Corke (2000) [20]. Briefly, 5 g of the co-crystallized powder were placed in a Petri dish and stored in a desiccator containing a saturated NaCl solution (75% relative humidity) for 7 days at 25°C. Hygroscopicity was expressed as grams of moisture adsorbed per 100 g of dry powder.

2.4.4 Color parameters

The samples were spread in a thin layer in Petri dishes (5 cm in diameter). The color characteristics of the co-crystallized products were determined using the CIELAB color parameters L* (lightness), a* (redness/greenness), and b* (yellowness/blueness), measured with a Minolta colorimeter (CR-400, Konica-Minolta Sensing, Japan) under illuminate D65 and a 10° standard observer. The hue angle (H°) and chroma (C*) were calculated from the a* and b* values according to Equations (1) and (2), respectively.

$$H^{\circ} = \tan^{-1} \left(\frac{b^{*}}{a^{*}} \right) \quad (1)$$

$$C^{*} = \sqrt{a^{*2} + b^{*2}} \quad (2)$$

2.4.5 Bulk density and repose angle

Bulk density was determined as the ratio of the mass of the co-crystallized powder to the volume it occupied in a graduated cylinder, according to the method described by Goula and Adamopoulos (2005) [21]. The angle of repose was measured following the methodology described by Bhandari et al. (1998) [22]. Briefly, the powder was introduced into a metal funnel with a diameter of 10 cm, mounted on a metal support, and allowed to flow freely onto a Petri dish. A sufficient amount of powder was used to cover the base plate and form a conical pile. The angle of repose was calculated from the radius of the Petri dish and the height of the cone formed by the powder.

2.4.6 Morphology

The surface morphology of the encapsulated and sugar samples was evaluated by scanning electron microscopy (SEM) (Quanta 50 model, Fisher Scientific, FEI, Czech Republic). The samples were previously prepared from carbon tape fixation on a steel surface, then metalized with a thin gold layer (≈ 50 nm) in the metallizer (SCD 50, Bal-Tec, Switzerland) and subsequently evaluated under high vacuum condition at 2000x magnification with an accelerating voltage of 20 kV.

2.4.7 Fourier-Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) (Nicolet iN10 model, Thermo Fisher Scientific, Brazil) defined the functional present in the co-crystallized products and sucrose samples. The samples evaluated were prepared by mixing with potassium bromide (KBr) at 1:100 (w/w). The FTIR spectra were recorded at a resolution of 4 cm^{-1} over the wavenumber range of $4000\text{-}500 \text{ cm}^{-1}$.

2.4.8 X-ray diffraction (XRD)

The crystalline structure of the co-crystallized products and sucrose was characterized using an XRD diffractometer (XRD-600, Japan). XRD measurements were collected with Cu radiation ($\lambda = 1.5406 \text{ \AA}$) at parameters: diffraction angles 2θ ($5\text{-}60^{\circ}$), filter wave at 40 kV, current 30 mA, the field of 0.01° at a scan rate of 2°min^{-1} for a total time of 218.77 s.

2.4.9 Differential Scanning Calorimetry (DSC)

The thermal properties of the co-crystallized products and sucrose were determined from DSC analysis. Differential scanning calorimetry (DSC) was performed using a differential scanning calorimeter (DSC-60 Shimadzu, Japan), using a hermetically sealed aluminium pan with about 3 mg of sample. The analysis was performed, at a heating rate of $10^{\circ}\text{C}/\text{min}$, from 20 to 320°C .

2.5 Application in beverage and sensory analysis

The co-crystallized lemon juice powder produced under the selected processing conditions was evaluated as an ingredient in the preparation of a caipirinha-type beverage. A commercially available powdered caipirinha mix (Cia Müller, Brazil) was used as the control sample. According to the ingredient list provided on the product label, this product contained sugar, dehydrated lemon pulp (1%), maltodextrin, citric acid (INS 330), natural lemon flavoring, tricalcium phosphate (INS 341iii), trisodium citrate (INS 331iii), ascorbic acid (INS 300), and natural colorants (INS 141ii and INS 100).

The research was reviewed and approved by the Human Research Ethics Committee of the State University of Maringá (Paraná, Brazil), under the number 65421722.3.0000.0104, and informed consent was obtained from each subject prior to their participation in the study. The sensory analysis panel consisted of 100 untrained evaluators, aged 18 to 25 years, who were usual consumers of caipirinha, and received the samples in monadic form, coded with three digits, and randomized to avoid bias. A randomized complete block design was adopted, that is, all evaluators analyzed the samples using the scales described below. The evaluators were asked to drink a glass of room-temperature water to rinse their mouths before each evaluation.

Each participant evaluated 10 mL of each of the three beverages: a commercial caipirinha lemon powder mix (Cia Müller, Brazil), a co-crystallized powder at 40 °Brix with 7 % juice, and a co-crystallized powder at 40 °Brix with 10 % juice. The beverages were prepared using the following method: a blend of vodka and water (50 % v/v) served as the base mixture, with the commercial powdered caipirinha mix was added to the base mixture for the sample control. The remaining samples included 12.5 % co-crystallized (40 °Brix 7 % juice and 40 °Brix 10 % juice, respectively) and 1.5 g of dehydrated lemon peel. The sensory acceptance of the samples was evaluated using a structured hedonic scale of nine points (1: dislike extremely; 2: dislike very much; 3: dislike moderately; 4: dislike slightly; 5: neither like nor dislike; 6: like slightly; 7: like moderately; 8: like very much; 9: like extremely).

2.6 Data Analysis

Statistical analysis of the co-crystallization process was performed with a one-way analysis of variance (ANOVA) and the Tukey test at a 5 % significance level ($P < 0.05$), using the Statistica version 12.0 (StatSoft Inc., Tulsa, Oklahoma, USA). The FTIR, XRD, and DSC analysis were run in OriginPro Software.

Consumer preferences were further compared using an internal preference map, generated by cluster analysis followed by multidimensional scaling using the Statistica program version 12.0 (StatSoft Inc., Tulsa, Oklahoma, USA). Beverage samples were arranged in columns (variables), and the individual consumer scores were placed in the rows (cases). First, a cluster analysis using the Euclidean distance as the distance measure and single linkage as the rule of amalgamation was applied to the individual data for each consumer, and the midpoint of the largest increment was used to separate the groups. The matrix obtained from the cluster analysis was then subsequently subjected to multidimensional scaling.

3. RESULTS AND DISCUSSION

3.1 Co-crystallization process

Table 1 presents the co-crystallization yield and physicochemical properties of the products obtained under different °Brix levels and juice concentrations.

The results demonstrated that both juice concentration and proportion significantly influenced the co-crystallization process, affecting crystal yield, process time, and product stability, except for hygroscopicity, which showed no significant differences at the 5 % significance level ($P > 0.05$).

Table 1: Characteristics of °Brix and juice concentration on co-crystallization yield and physicochemical properties.

Treatments	Responses					
	Yield (% w/w)	CT (s)	MC (% w/w)	aw	ST (s)	HG (%w/w)
20°Brix/7%	61.87 ± 0.75 ^a	116.7 ± 1.5 ^a	5.17 ± 0.22 ^a	0.513 ± 0.01 ^a	24.2 ± 0.8 ^a	7.48 ± 1.3 ^a
20°Brix/10%	66.79 ± 1.29 ^b	129.3 ± 3.4 ^b	5.02 ± 0.83 ^a	0.662 ± 0.02 ^b	29.1 ± 1.6 ^b	8.57 ± 0.28 ^a
40°Brix/7%	66.63 ± 0.75 ^b	87.7 ± 1.5 ^c	2.15 ± 0.24 ^b	0.449 ± 0.01 ^c	21.3 ± 1.5 ^c	8.52 ± 0.99 ^a
40°Brix/10%	72.8 ± 1.46 ^c	127.0 ± 3.0 ^b	3.61 ± 0.56 ^c	0.433 ± 0.01 ^c	20.0 ± 1.1 ^c	8.11 ± 0.31 ^a

Results are expressed as mean ± standard deviation of triplicates. Means in the same column followed by different letters in superscript differ significantly at $P \leq 0.05$. Where: CT (co-crystallization time); MC (moisture content); ST (solubilization time); HG (hygroscopicity).

The crystal yield relative to the initial mass was highest for the treatment with 40° Brix and 10% juice, exceeding the mean yield of the other treatments by approximately 10%. This result can be attributed to the higher soluble solids content of the syrup combined with a lower amount of water introduced by the lemon juice, which reduced dilution of the heated solution and ultimately resulted in a greater mass of co-crystallized material. Similar findings have been reported by other authors for orange juice co-crystallization [23].

Regarding processing time, the longest times were observed for the 10% juice treatments, with no significant difference between them at the 5% significance level ($P > 0.05$). The shortest co-crystallization time was obtained for the treatment with 40 °Brix and 7% juice. During the addition of concentrated lemon juice, the temperature of the sucrose solution decreased, promoting the transition from the metastable zone to the labile zone. However, higher juice concentrations caused greater dilution of the mixture, decreasing the degree of supersaturation and consequently increasing the time required for crystal formation [17]. Despite this effect, the co-crystallization times observed in this study were shorter than those reported by Torres-Gallo et al. (2021) [23] and Tzatsi and Goula (2021) [5] for fruit juices, which ranged from 13.8 to 46 min and approximately 3.5 min, respectively. These differences may be attributed to the lower stirring applied in those and to higher soluble solids content of the orange and passion fruit juices used.

It can also be observed that increasing the juice addition from 7% to 10%, that is, increasing its mass fraction in the supersaturated syrup, increased the co-crystallization time, while also improving crystal yield. In general, higher degrees of supersaturation promote faster crystal formation, whereas lower degrees of supersaturation slow the crystallization process. Therefore, as more lemon juice is added to the sucrose syrup, the degree of supersaturation decreases due to dilution, leading to longer co-crystallization times. This relationship between juice addition and co-crystallization time indicates a potential strategy for controlling and optimizing the process.

The moisture content (MC) of the co-crystallized products obtained at 40° Brix was approximately 40% lower than that of the co-crystals produced at 20° Brix. Similarly, the water activity (aw) decreased from 0.662 in the 20° Brix and 10% treatment to values between 0.433 and 0.449 in the 40° Brix treatments, representing a substantial reduction. This finding is particularly relevant because water activity values below approximately 0.6 are generally considered to inhibit the growth of most bacteria, thereby improving the microbiological stability and extending the shelf life of dehydrated products. Therefore, beyond the statistical differences, the lower aw values further support the selection of the 40° Brix treatments as the most suitable processing conditions. The moisture content observed in this study was higher than values reported in the literature for co-crystallized extracts [24], although comparable to those reported for co-crystallized fruit juices [23].

The solubilization time (ST) of the co-crystallized products showed an inverse relationship with soluble solids content. This behavior is attributed to the higher sucrose content of the formulations with greater soluble solids, as sucrose was used as the encapsulating matrix. The solubilization times were similar to those reported for guava juice co-crystallization (11.7 and 22 s) and within the range reported for passion fruit juice co-crystallization (25 to 49 s) [23]. The porous structure of the co-crystallized matrix enhances water penetration, resulting in rapid dissolution and stability [25]. This characteristic is particularly important, as the product is intended for use in the preparation of alcoholic beverages, where rapid solubilization and good reconstitution properties are essential.

Regarding hygroscopicity (HG), no significant differences were observed among treatments at the 5% significance level ($P > 0.05$). Although the material contains highly hygroscopic components such as fructose, glucose, and sucrose, the co-crystallized lemon juice exhibited the characteristics of a low-hygroscopicity products (below 10%) [26]. Compared with other studies, the HG values obtained in this work were lower than Poniol extract co-crystallized (11%) [27] and higher than chokeberries (0.09%) [5] and *Basella rubra extract* (0.16-1.07%) [28] in the same relative humidity. These differences probably could be associated with final moisture content of the samples, which may have contributed to increased water adsorption during storage.

Overall, the results indicate that concentration of 40° Brix provided the most favorable processing conditions and physicochemical properties. Therefore, samples containing 7 and 10% lemon juice at this concentration were selected for further characterization to evaluate the feasibility of producing a co-crystallized powdered caipirinha-type beverage.

3.2 Physical, morphology, structural, and thermal characterization of the co-crystallized

The most suitable co-crystallization condition identified were physically characterized, as presented in Table 2.

Table 2: Characterization physicochemical of co-crystallized powders.

Characteristics	40 °Brix 7 % juice	40 °Brix 10 % juice
L*	96.31±0.17 ^a	96.32±0.25 ^a
a*	-0.83±0.02 ^a	-0.60±0.03 ^b
b*	7.04±0.24 ^a	7.13±0.10 ^a
H (°)	96.71±0.40 ^a	96.71±0.40 ^a
C	7.09±0.24 ^a	7.15±0.10 ^a
Repose angle (°)	40.08±0.02 ^a	34.46±0.02 ^b
Bulk density (g.cm⁻³)	0.664±0.06 ^a	0.766±0.03 ^b

Results are expressed as mean ± standard deviation of triplicates. Means in the same line followed by letters in superscript don't have significance difference at $P \leq 0.05$.

The results showed that color parameters did not differ significantly at the 5% significance level ($P > 0.05$). Lightness values indicated that the samples were very light (L^* value close to 100), with a greenish-yellow coloration.

The angle of repose is related to the characterization of powder flow behavior and is associated, with the ability of solid particles to form a stable pile due to internal cohesive forces [29]. Several factors influence the angle of repose, particularly moisture content and particle size are the most relevant, as they directly affect powder flowability [30].

Powders with angles of repose greater than 50° exhibit higher cohesiveness, poor flowability, and greater adherence, whereas angles lower than 45° indicate free-flowing behavior and improved powder fluidity [22]. As shown in Table 2, the co-crystallized powders containing 7% and 10% lemon juice were classified as free flowing in agreement with results reported in the literature for co-crystallized peppermint extract [24].

Bulk density is defined as the mass per unit volume of a material in its bulk or loose state, including void spaces between particles. This property is essential in several industrial applications, as it influences material handling, processing, packaging, and distribution [31]. In the present study, bulk density values were within the range reported by Peleg and Pollak (1982) [32] for food powders (0.30 to 0.80 g.cm⁻³). In addition, powders containing 10 % (w/w) lemon juice showed higher bulk density, likely due to the encapsulation of a greater number of soluble solids within the crystalline matrix.

Further characterization of the co-crystallized products was carried out by comparison with commercial sucrose to evaluate the effectiveness of lemon juice encapsulation. In this context, scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and differential scanning calorimetry (DSC) are commonly used techniques to evaluate the morphology, structure, composition, and physical properties of co-crystallized products.

Scanning electron micrographs (SEM) of sugar and co-crystallized products are represented in Figure 2.

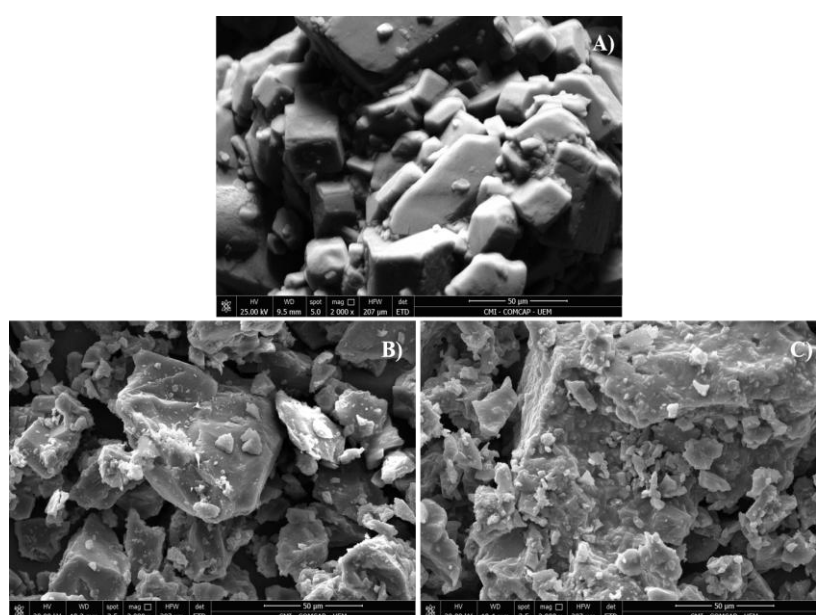


Figure 2: Micrographs of the surface morphology of the sugar and co-crystallized particles evaluated at 2000x magnification. A) Sugar; B) 40° Brix 7 % juice; C) 40° Brix 10 % juice.

As shown in Figure 2A, commercial sucrose shows a well-defined crystalline structure with regular surface edges, justified by regular arrangements of specific atoms or molecules, according to Sardar and Singhal (2013) [33]. In contrast, the micrograph shown in Figures 2B and 2C, common characteristics of co-crystallized products were identified, given the appearance of agglomerates with a rough and disorderly structure. These results agreed with those reported by Tzatsi and Goula (2021) [5] and Karangutkar and Ananthanaravan (2020) [28], that co-crystallized products may have an amorphous appearance due to the disorder generated during the process.

It is also observed in Figure 2B that the co-crystallized 40 °Brix 7 % juice has lower amounts of agglomerated crystals than the co-crystallized 40 °Brix 10 % juice (Figure 2C), which makes it possible to assert that the increased juice concentration proposes a higher proportion in sucrose encapsulation. Therefore, both results suggest that the products formed in the present study have a porous structure as a place for trapping organic compounds [34].

Figure 3 shows that infrared spectroscopy (FTIR) was performed to assess chemical structure and identify absorbance bands of the functional groups in the samples.

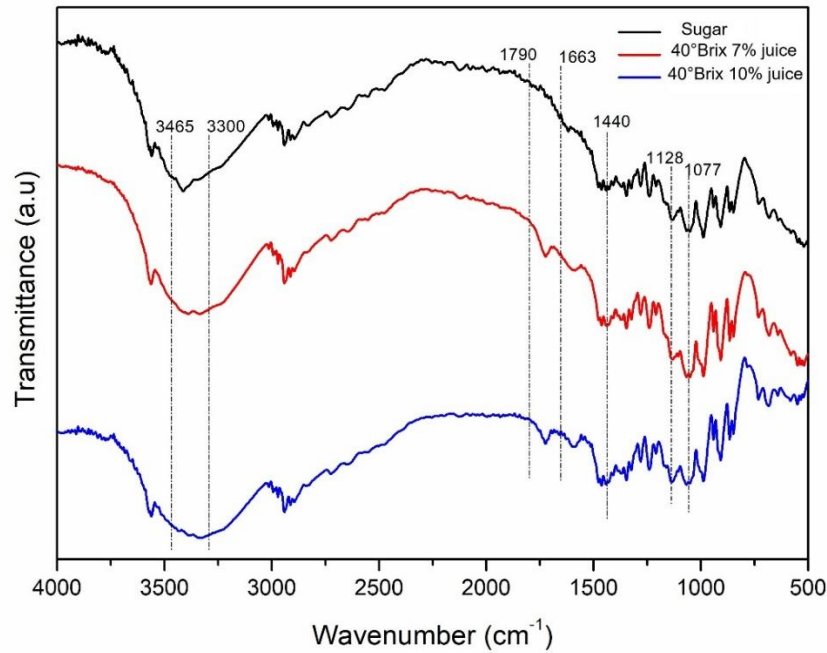


Figure 3: FTIR spectrum of sugar and co-crystallized samples.

The FTIR spectrum of commercial sucrose exhibited the characteristic groups of this type of simple carbohydrates, such as sucrose. The main functional groups identified were the stretching of hydroxyl groups (O-H) present in the glycosidic ring between 3300-3465 cm^{-1} , the stretching of carbonic bonds (C-C), and the bending (C-H), both around 1200 cm^{-1} . After the aggregation of juice in sugar, a new band was identified between 1790-1663 cm^{-1} , noted for the stretching vibrations of the carbonyl group (-C=O). Results of interaction between the encapsulated components are also observed by forming of a broad band near 1077 cm^{-1} , corresponding to a structural modification involving saccharide bonds given by C-O stretching vibrations caused by the incorporation of the fructose molecules [35]. In the spectra of the co-crystallized, another broadband formation was observed near 1440 cm^{-1} , has been associated with the presence of citric acid from lemon [36], justified by the addition of lemon juice concentrates.

XRD diffractograms relate the crystallinity of the sucrose present in the sugar to the materials produced by co-crystallization with lemon juice (Figure 4). Other studies also demonstrate the maintenance of the crystalline structure of sucrose after its encapsulation [11, 14, 28].

When comparing the X-ray diffraction profiles of the sugar with the co-crystallized products containing 7% and 10% lemon juice, as illustrated in Fig. 4, similarities were observed in their main peaks, located near $2\theta = 7^\circ, 11^\circ, 13^\circ, 18^\circ, 19^\circ, 22^\circ, 25^\circ, 31^\circ$ in all samples. Notably, no additional diffraction peaks were observed in the co-crystallized products, suggesting that the incorporation of lemon juice did not alter the crystalline structure of the sucrose matrix, consistent with the findings of Ali et al. (2023) [27]. However, a reduction in the intensity of several diffraction peaks was observed after co-crystallization, which may be attributed to the formation of smaller crystalline domains and a partial reduction in crystallinity resulting from structural modifications of the crystalline matrix [14].

Therefore, the co-crystallization process did not alter the overall crystal structure of sucrose, although it reduced its crystallinity [26]. The characteristic diffraction pattern of sucrose has several sharp and higher-intensity diffraction peaks, suggesting high crystallinity, as indicated by Queiroz et al. (2022) [14]. Materials are classified as highly crystalline when crystallinity exceeds 50%, moderately crystalline between 20–50%, and low crystallinity below 20% [26].

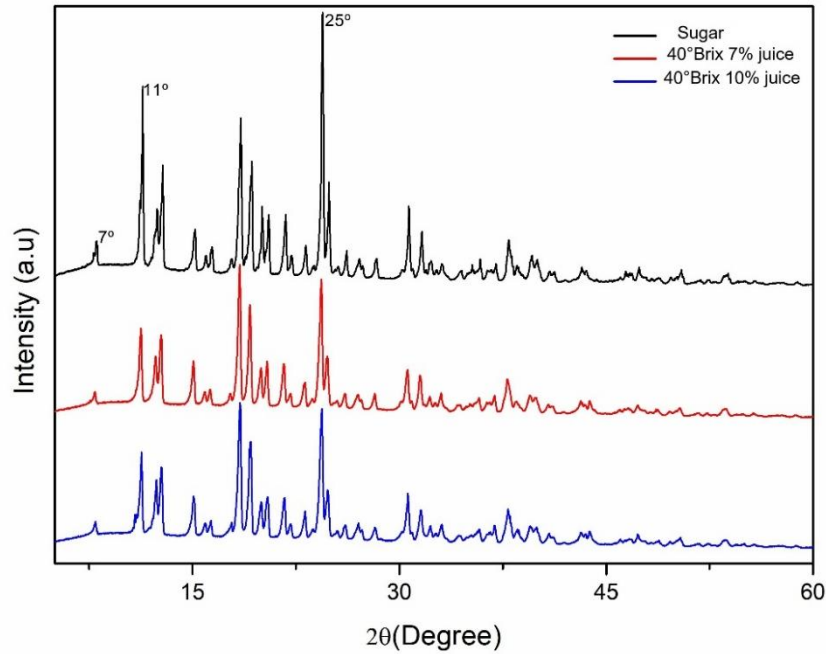


Figure 4: Crystallinity profiles of sugar and co-crystallized samples by diffractograms XRD.

Thus, a particularly relevant aspect was the decrease in intensity in the region of $2\theta = 25^\circ$, which showed an intensity of 90% for sugar, reducing to 60% and 40% in the co-crystallized products (7% and 10% juice), respectively. Furthermore, the intensity in the region of $2\theta = 11^\circ$, which was expressed as 24% in sugar, was reduced to 50% of this value after the process. A similar trend was observed at $2\theta = 13^\circ$, where the intensity was reduced by 40–50% after the co-crystallization. In the region of $2\theta = 8^\circ$, there was a reduction in intensity compared to that of sugar (16%) for the co-crystallized materials 10% (7% juice) and 13% (10% juice). This suggests that the co-crystallization process reduced the degree of crystallinity of sucrose, which may be attributed to the amorphous nature of the incorporated extract [26].

Reductions in crystallinity in the region of $2\theta = 7^\circ$, 10° , and 25° were also observed by Ma et al. (2021) [37] during fructose-based catalytic production, corresponding to structural modifications of sucrose in the co-crystallized products. Similarly, Yao et al. (2020) [38] associated the structural modifications designated by the diffraction patterns of fructose, which can be observed through a structural rearrangement facilitated by hydrogen bonds related to D-fructose when observed at $2\theta = 7$ and 13° . In this way, the crystalline structure of sucrose was maintained after co-crystallization with lemon juice.

Differential explorations follow the effects of heat associated with phase transitions and other thermal events as a function of the temperature that undergoes an increase at a constant rate. The thermal properties are directly related to the stability of the co-crystallized, thus determining the decomposition temperature and the enthalpy variation involved in the phenomenon.

Figure 5 shows the thermographs of the sugar and co-crystallized samples, all of which exhibited two endothermic transition peaks. Furthermore, it can also verify that all the samples indicate first-order transitions, which corresponding to the crystallization and melting processes.

The sugar thermograph shows a main endothermic peak around 180°C related to sucrose melting point with 121.72 J.kg^{-1} enthalpy. Authors reported endothermic peaks at 190°C [26, 39, 40]. On the other hand, co-crystallized samples showed endothermic peaks of the sucrose melting to the left around 150°C . This behavior is also related by López-Córdoba et al. (2014) [41] in yerba mate extract and, Bajaj and Singhal (2021) [42] in vitamin B12 extract co-crystallization. A probable formation of irregular crystals at the end of the process, which increased the amorphous form of the sucrose, and other compounds present in the lemon juice could explain this peak dislocation, reducing the amount of energy necessary to melt crystalline sucrose present in the samples co-crystallized, since the enthalpies for 7% was 91.34 J.kg^{-1} and for 10% was 80.96 J.kg^{-1} [11].

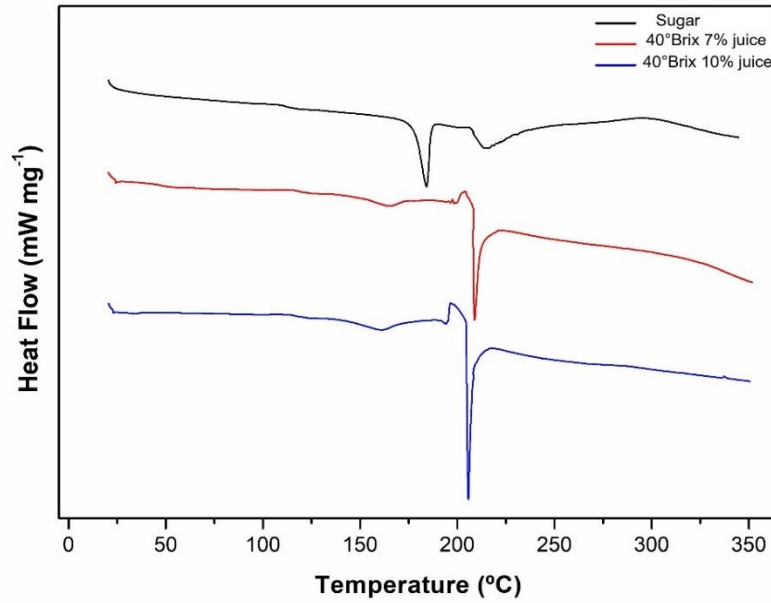


Figure 5: DSC curves of sugar and co-crystallized samples.

A second endothermic thermal event was observed at around 220°C and an enthalpy of 112.86 J.kg⁻¹ which has been associated with the thermal degradation of sucrose according to Sarabandi et al. (2019) [34]. The addition of 7 % and 10% lemon juice led to an increase in melting temperatures to 212 and 221°C, respectively, as well as enthalpy values of 240.52 and 259.41 J.kg⁻¹ showing that due to a higher concentration of organic compounds, a more significant amount of thermal absorption would be necessary for the degradation of these compounds. Authors also reported endothermic peaks in the 221-222°C [27] and 216-235°C [43]. Compared with sugar sample, it can be inferred that the co-crystallization process increases the thermal stability of bioactive compounds in the juice and provides encapsulated protection, mainly at 10% with lemon juice. Studies using sucrose as an encapsulation matrix obtained results like those described in this work [40, 41]. Therefore, it is stated that the co-crystallization process provides encapsulated protection and assists in their thermal stability.

3.3 Sensory evaluation of alcoholic beverages from lemon juice co-crystallized

Therefore, the co-crystallization process did not alter the overall crystal structure of sucrose, although it reduced its crystallinity [26]. The characteristic diffraction pattern of sucrose has several sharp and higher-intensity diffraction peaks, suggesting high crystallinity, as indicated by Queiroz et al. (2022) [13]. Materials are classified as highly crystalline when crystallinity exceeds 50%, moderately crystalline between 20–50%, and low crystallinity below 20% [26].

Table 3: Consumer acceptance scores for the sensory attributes of the caipirinha formulations.

Attributes	Commercial	40° Brix 7% juice	40° Brix 10% juice
Aroma	8.0 ± 1.2 ^a	6.3 ± 1.6 ^b	6.4 ± 1.9 ^b
Freshness	7.8 ± 1.2 ^a	6.5 ± 1.7 ^b	6.7 ± 1.6 ^b
Bitterness	7.0 ± 1.7 ^a	6.2 ± 1.9 ^b	6.2 ± 1.9 ^b
Flavor	7.8 ± 1.2 ^a	6.3 ± 1.9 ^b	6.6 ± 1.9 ^b
Overall Acceptance	7.8 ± 1.1 ^a	6.4 ± 1.7 ^b	6.7 ± 2.0 ^b

Results are expressed as mean ± standard deviation of the consumers. Means in the same line followed by different letters in superscript differ significantly at $P \leq 0.05$.

No significant differences in sensory acceptance were observed between the beverages prepared from the co-crystallized powders ($P > 0.05$). In addition, the commercial sample exhibited higher sensory mean scores than the co-crystallized samples across all assessed parameters. This outcome can be attributed to the commercial formulation's utilization of artificial flavoring, which imparts a slightly more pronounced lemon flavor than the other samples.

Considering that the primary objective of this study is to develop a more "natural" fruit preparation devoid of synthetic additives, aligning with the current clean-label product trend [44], the positive acceptance scores received from consumers represent a promising outcome despite their minor mean score relative to those of commercial preparation.

The cluster analysis of overall acceptance for the beverages yielded two distinct groups: the commercial drink and the co-crystallized samples (Figure 6 A). The results indicate that the co-crystallized drinks exhibited similar overall acceptance, distinguishing them from the acceptance pattern of the commercial beverage.

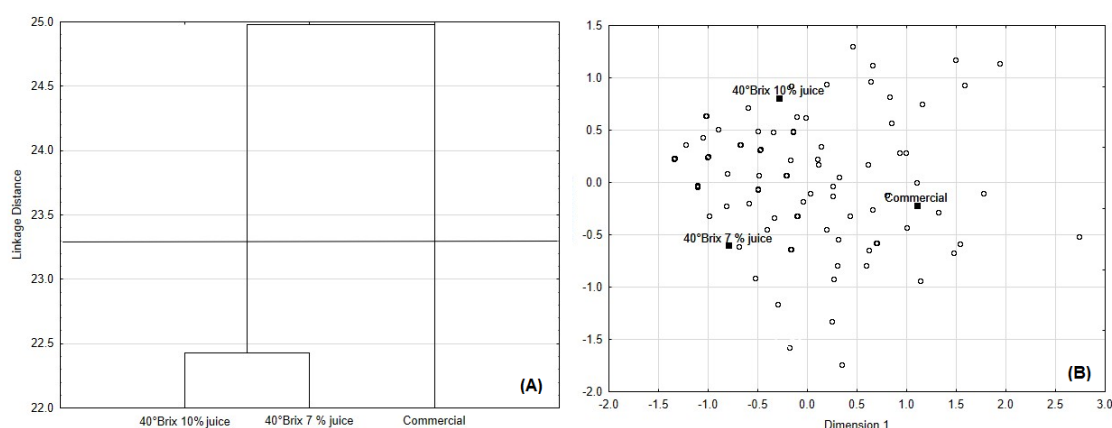


Figure 6: Dendrogram (A) and internal preference map (B) for overall acceptance of the caipirinhas-flavored beverage.

The internal preference map (Figure 6 B) provided a clearer visualization of the potential grouping of the evaluated beverages, revealing a proximal relationship among the co-crystallized samples. Each point on the internal preference map represents an individual consumer, and a higher concentration of consumers.

The preference map shows that although the co-crystallized samples had lower overall acceptance (Tukey's test) than the commercial sample, a large specific group of consumers may be interested in them. This result supports the earlier discussion of the samples' sensory profiles, indicating the presence of two distinct consumer preference profiles: one prefers the commercial sample flavor, and the other shows a greater affinity for the more natural flavors of the samples produced in this experiment. Moreover, all the beverages tested were located close to each other, showing good overall acceptance by consumers.

Previous studies by Muzi Marpaung et al. (2020) [9] and BehnamNik and Vazifedoost (2020) [1] also showed in their research that co-crystallization was a viable process for obtaining potential functional beverages with butterfly pea flower and with *Securigera securidaca* seed extract, respectively. The application of co-crystallized lemon juice in a caipirinha-type product corroborates the literature, highlighting that co-crystallization using sugar with other compounds is an innovative possibility [45]. It improves the properties of the encapsulated material, thus enhancing the product line in the industrial sector. Therefore, these findings highlight co-crystallization as a viable method for producing natural, stable, and consumer-accepted powdered beverages.

4. CONCLUSION

This study demonstrated that lemon juice concentration and °Brix level significantly influenced the co-crystallization process, affecting crystal yield, processing time, moisture content, water activity and dissolution time, except to hygroscopicity. Among the evaluated conditions, 40° Brix provided the most favorable properties, resulting in a product with improved dispersibility and microbiological stability.

Structural, chemical, and thermal analyses demonstrated that co-crystallization preserved the crystalline structure of the sucrose matrix while reducing crystallinity due to the formation of amorphous regions associated with encapsulation. These results confirm the technical feasibility and stability of the co-crystallization process.

Sensory evaluation demonstrated positive consumer acceptance of beverages prepared with co-crystallized lemon juice. Although acceptance was slightly lower than that of commercially available formulation, the results highlight the potential of co-crystallization as a natural alternative for the development of powdered beverage products.

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