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DANUSA SILVA DA COSTA

REVESTIMENTO COMESTÍVEL INCORPORADO COM TOCOFERÓIS E APLICAÇÃO EM CASTANHA-DO-BRASIL (*Bertholletia excelsa* Bonpl.) PARA MONITORAMENTO DO PERFIL DE OXIDAÇÃO LIPÍDICA

BELÉM – PA, BRASIL

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RESUMO

A castanha-do-brasil é um produto não madeireiro de grande importância para famílias extrativista, possui muitos nutrientes em sua composição e é rica em lipídios. Porém, devido desde a coleta à extração do óleo as castanhas ficarem expostas a diversas condições degradantes como umidade, luz e calor pela exposição ao ambiente, essas condições podem intensificar a oxidação lipídica desse produto. Por isso, desenvolver um revestimento ativo surge como uma alternativa de proteção e preservação da castanha-do-brasil à oxidação lipídica. Objetivou-se desenvolver um revestimento comestível ativo incorporado com tocoferóis e aplicar em castanha-do-brasil para monitorar o perfil de oxidação lipídica das castanhas em diferentes tempos de armazenamento. Foram preparados filmes por casting. Quatro blends, denominadas B, L, LT e LT2 foram preparadas utilizando quantidades fixas de fécula de mandioca + carboximetil celulose + sorbitol e água (3 g + 1,5 g + 0,2 g + 100 g, respetivamente), foi adicionada lecitina de soja na proporção de 20% em relação à mistura de tocoferóis, a formulação L foi preparada com a mesma quantidade de lecitina que a LT2. A quantidade de mistura de tocoferol foi adicionada às misturas nas seguintes proporções: B - 0, L - 0, LT - 0,0005 g e LT2 - 0,010 g. Foram realizados testes de espessura, solubilidade em água, teor de água, gramatura, ângulo de contato, teste de permeabilidade, testes mecânicos, atividade antioxidante, biodegradabilidade, cor, termogravimetria e FTIR. Os filmes foram caracterizados como biodegradáveis, antioxidantes e higroscópicos, sendo indicado para aplicação em forma de revestimento nas castanhas-do-brasil. Foram avaliadas viscosidade cremeação, centrifugação, pH e cor das soluções formadoras dos revestimentos, o que possibilitou caracterizar as blends como fluido não-newtoniano e pseudoplástico, altamente estável. As castanhas-do-brasil foram imersas nas soluções formadoras de revestimento, secas a 45°C por 19h, em seguida foram acondicionadas a 25°C, sendo avaliadas aos 1, 7, 15, 30, 45, 60, 90 e 120 dias de armazenamento. Foram realizados testes de perda de massa, índice de escurecimento, dienos e trienos conjugados, estado oxidativo por métodos oficiais e índice de oxidação acelerado. Todos os revestimentos aplicados promoveram a minimização da oxidação em alguma etapa do armazenamento, porém os filmes adicionados do mix de tocoferóis promoveu a minimização de dienos, trienos, índice de iodo, índice de peróxido, p-anisidina e do índice de oxidação total. O teste oxidação acelerada mostrou que o óleo das castanhas em estudo sofreu algumas alterações no estado oxidativo, embora tenham sido em grande parte, preservadas pelo uso dos revestimentos. Os modelos aplicados aos dados do teste de oxidação acelerada permitiram estimar a meia-vida das castanhas-do-brasil, assim verificou-se que o revestimento LT apresentou valor 129.86h para o tempo de meia-vida no modelo de primeira ordem. A análise de componentes principais (PCA) aplicada aos dados de caracterização físico-química e ao tempo de indução revelou que o óleo das castanhas não revestidas apresentou-se isoladamente sendo extremamente influenciado pelos índices oxidativos avaliados, apresentou uma variabilidade explicada dos dados maior que 75% na maioria dos tempos de armazenamento. A PCA mostrou também que o óleo das castanhas-do-brasil revestidas com LT e LT2 foram influenciados negativamente pela maioria dos índices oxidativos, nesse caso isso é uma vantagem, pois demonstra menores índices de oxidação lipídica dessas amostras. Desenvolver um filme com propriedades antioxidantes e a aplicar como revestimento em castanha-do-brasil possibilitou verificar alguns níveis de proteção das castanhas. Sugere-se a aplicação dos blends desenvolvidos na forma de filme ou revestimento ativo, para outros produtos alimentícios que necessitem de proteção contra luz e oxidação.

Palavras-chave: fruta Amazônica; antioxidante; revestimento ativo; prolongamento da vida útil; oxidação lipídica.

ABSTRACT

Brazil nuts are a non-timber product of great importance to extractivist families. They contain many nutrients and are rich in lipids. However, from collection to oil extraction, Brazil nuts are exposed to various degrading conditions such as humidity, light, and heat due to exposure to the environment, and these conditions can intensify the lipid oxidation of this product. For this reason, developing an active coating is an alternative for protecting and preserving Brazil nuts from lipid oxidation. The aim was to develop an active edible coating incorporated with tocopherols and apply it to Brazil nuts to monitor their lipid oxidation profile at different storage times. Films were prepared by casting. Four blends, called B, L, LT, and LT2, were prepared using fixed amounts of cassava starch + carboxymethyl cellulose + sorbitol and water (3 g + 1.5 g + 0.2 g + 100 g, respectively), soy lecithin was added in a proportion of 20% in relation to the tocopherol mix, formulation L was prepared with the same amount of lecithin as LT2. The amount of tocopherol mix was added to the mixtures in the following proportions: B - 0, L - 0, LT - 0.0005 g, and LT2 - 0.010 g. Tests were carried out on thickness, water solubility, water content, weight, contact angle, permeability, mechanical tests, antioxidant activity, biodegradability, color, thermogravimetry, and FTIR. The films were characterized as biodegradable, antioxidant, and hygroscopic and suitable for application as a coating on Brazil nuts. Viscosity, creaming index, centrifugation, pH, and color of the solutions forming the coatings were evaluated, making it possible to characterize the blends as a non-Newtonian and highly stable pseudoplastic fluid. The Brazil nuts were immersed in the coating-forming solutions, dried at 45°C for 19 hours, stored at 25°C, and evaluated at 1, 7, 15, 30, 45, 60, 90, and 120 days of storage. By official methods, tests were carried out on mass loss, browning index, conjugated dienes and trienes, oxidative status, and accelerated oxidation index. All the coatings applied minimized oxidation at some stage during storage. However, the coatings added with the tocopherol mix minimized dienes, trienes, the iodine index, the peroxide index, p-anisidine, and the total oxidation index. The accelerated oxidation test showed that the nut oil under study underwent some changes in its oxidative state, although the use of the coatings largely preserved them. The models applied to the accelerated oxidation test data made it possible to estimate the half-life of the Brazil nuts, so it was found that the LT coating showed a value of 129.86h for the half-life time in the first-order model. The principal component analysis (PCA) applied to the physicochemical characterization data and the induction time revealed that the oil from nuts uncoated presented itself in isolation as being extremely influenced by the oxidative indices evaluated, presenting an explained variability of the data greater than 75% in most of the storage times. PCA also showed that the oil from Brazil nuts coated with LT and LT2 was negatively influenced by most of the oxidative indices, in which case this is an advantage, as it demonstrates lower rates of lipid oxidation in these samples. Developing a film with antioxidant properties and applying it as a coating on Brazil nuts made it possible to verify some levels of protection for the nuts. We suggest applying the blends developed as a film or active coating to other food products that need protection against light and oxidation.

Keywords: Amazon fruit; antioxidant; active coating; shelf life extension; lipid oxidation.

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1 INTRODUÇÃO GERAL

O bioma Amazônico detém biodiversidade gigantesca, possui inúmeros vegetais com potencialidades de aplicação em alimentos, produtos farmacêuticos, usos terapêuticos e químicos (PEREIRA et al., 2017; RUIZ-GARCÍA et al., 2017). A castanha-do-brasil (*Bertholletia excelsa* Bonpl.) é um dos três dos produtos alimentares amazônicos mais reconhecidos. A sua cadeia de valor global vale quase 450 milhões de dólares por ano. No Brasil, 60.000 famílias extrativistas, fazem do país o maior produtor do mundo, com 33.000 toneladas em 2020 (TRIDGE, 2020). De acordo com o Instituto Brasileiro de Geografia e Estatística - IBGE, a produção de castanha-do-brasil em 2021/2022 foi de 38.160 toneladas, com predominância na região Norte, cuja produção foi de 35.964 toneladas, e o estado do Pará foi responsável pela produção de 8.807 toneladas nesse período (IBGE, 2024).

A castanha-do-brasil é um dos produtos florestais não madeireiros mais importantes, e a subsistência de milhares de famílias tradicionais depende de sua comercialização (RIBEIRO et al., 2014; SILVA et al., 2016; CHIRIBOGA-ARROYO et al., 2020; RÊGO et al., 2021). O que é de grande importância na atualidade, pois manter-se a árvore de pé, coletar um fruto sem precisar desmatar faz parte do sistema de bioeconomia, ou seja, a castanha-do-brasil faz parte de um sistema que precisa preservar a flora para que continue se perpetuando. As castanhas-do-brasil são largamente consumidas, por serem uma boa fonte nutricional (YANG, 2009), e possuir em sua composição, proteínas (16,03 g 100 g^{-1}), lipídios (58,52 g 100 g^{-1}), carboidrato (19,61 g 100 g^{-1}), cinzas (3,35 g 100 g^{-1}) (VASQUEZ-ROJAS et al., 2021), além de 5,24 µg g^{-1} de Selênio (BOTELHO et al., 2019).

Desde a produção, processamento, distribuição até armazenamento, os alimentos sofrem deterioração de processos químicos e microbiológicos. Uma das soluções é usar embalagens comestíveis preservando a qualidade, segurança e transferência de informações para os consumidores (GOMEZ-ESTACA et al., 2014). Os filmes e revestimentos são adotados para minimizar a deterioração dos gêneros alimentícios, reduzir o risco de contaminação e manter o produto seguro para ser comercializado. Esta prática reduz os danos sensoriais, proporcionando um bloqueio semipermeável em torno do produto (NAIR et al., 2020).

Embalagem ativa de alimentos é uma das novas tecnologias inovadoras no campo da embalagem inteligente que combina os alimentos, o ambiente de embalagem e as suas interações com polímeros naturais ativos para assegurar a preservação da qualidade e aumentar a vida útil dos materiais biológicos alimentares, protegendo o consumidor e o ambiente através da preservação dos alimentos de microrganismos patogênicos (YILDIRIM et al., 2017). Embalagens com propriedades antioxidantes têm recebido atenção especial, alternativamente às embalagens tradicionais, pois os alimentos revestidos ou embalados podem obter redução da oxidação lipídica, uma das principais causas de deterioração de produtos alimentícios (LÓPEZ-DE-DICASTILLO et al., 2012).

De maneira geral para proteger da oxidação lipídica, os filmes e revestimentos comestíveis além de apresentar capacidade antioxidante, devem ter propriedades eficazes de barreira ao oxigênio (ATARES; CHIRALT, 2016). Foram relatados diversos estudos sobre a incorporação de antioxidantes a filmes e revestimentos como α-tocoferol (ZAMBRANO-ZARAGOZA et al., 2014; SCARFATO et al., 2017).

Porém, devido à elevada concentração de ácidos graxos insaturados da castanha-do-brasil ela se torna bastante perecível, especialmente quando exposta a comercialização por longo período de tempo, pois é submetida a condições de temperatura e umidade relativa elevada, ficando exposta a processos oxidativos que podem contribuir para a perda de nutrientes e ocorrência de odor e sabor rançoso nesse alimento (RIBEIRO et al., 1993a; SILVA; ASCHERI; SOUZA, 2010).

Alguns estudos avaliaram o efeito das condições de armazenamento nas alterações oxidativas da castanha-do-brasil, como por exemplo, os estudos de Ribeiro, Reginato-D'Arce, et al. (1993), que embalaram castanhas-do-brasil em sacos de papel kraft e armazenaram em temperaturas de -15, 2 e ambiente - variando de 18 a 25°C, e Casagrande et al. (2019) armazenaram castanhas-do-brasil em recipientes de vidro e plástico transparentes e opacos e armazenaram em temperaturas de 11 e 24°C.

No estudo de Leme et al. (2023) desenvolveram embalagens termoplásticas de amido/poli(adipato de butileno-co-tereftalato) (TPS/PBAT) contendo curcumina e pinhão solúvel em água, as castanhas-do-brasil foram embaladas e acondicionadas em potes herméticos armazenados a 10, 25 e 50ºC, a oxidação lipídica foi determinada aos 0, 5, 10, 15 e 30 dias de armazenamento. No entanto, não foram encontrados estudos sobre a monitorização do perfil de oxidação lipídica de castanhas-do-brasil embaladas com um revestimento comestível à base de amido de mandioca/carboximetil celulose incorporado com tocoferóis e armazenadas por diferentes períodos.

O estilo de vida contemporâneo demanda que os alimentos tenham sua vida útil prolongada, mantendo a qualidade e promovendo a segurança do produto (BIJI et al., 2015). Por isso, é de grande interesse o desenvolvimento de um revestimento comestível que contenha antioxidantes, como estratégia de proteção para castanhas-do-brasil que minimize os níveis de oxidação lipídica. O objetivo deste estudo será desenvolver um revestimento comestível incorporado com tocoferóis e aplicar em castanha-do-brasil para monitorar o perfil de oxidação lipídica das castanhas em diferentes tempos de armazenamento.

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2 OBJETIVOS

2.1 Objetivo geral

Desenvolver revestimento comestível ativo incorporado com tocoferóis e aplicar em castanha-do-brasil para monitorar o perfil de oxidação lipídica das castanhas em diferentes tempos de armazenamento.

2.2 Objetivos específicos

- Desenvolver formulações de soluções filmogênicas emulsionadas para serem aplicadas como revestimento comestível ativo à base de fécula de mandioca e carboximetil celulose incorporado de tocoferóis;
- Avaliar os parâmetros reológicos e de estabilidade física da solução filmogênica emulsionada, que será aplicada como revestimento comestível ativo, por meio da viscosidade, índice de cremeação e centrifugação;
- Determinar as propriedades físicas, de barreiras e mecânicas do filme ativo, para se buscar características próximas as que seriam as características de um revestimento, pois um revestimento depois de aderido a superfície de um alimento, dificilmente se desprenderá no formato de um corpo de prova ideal para os testes;
- Aplicar as soluções filmogênicas como revestimento comestível ativo em castanhas-do-brasil, para armazenar e acompanhar o nível oxidativo das castanhas;
- Avaliar a vida útil das castanhas-do-brasil revestidas e não revestidas em diferentes tempos de armazenamento, para verificar o possível prolongamento da vida útil;
- Monitorar o perfil de oxidação lipídica das castanhas-do-brasil revestidas e não revestidas em diferentes tempos de armazenamento, para verificar se os revestimentos aplicados promovem proteção quanto aos danos oxidativos.

3 ESTRUTURA DA TESE (CAPÍTULOS)

A tese foi dividida em três capítulos, que serão citados resumidamente a seguir:

- No Capítulo 1 da tese foi realizada uma revisão utilizando-se ferramentas da declaração de itens de relatório preferidos para revisões sistemáticas e meta-análises, mas sem focar em uma análise randomizada. Em 24 de janeiro de 2022 foram pesquisados artigos de pesquisa do ano de 2017 a 2022; Nenhuma restrição de idioma foi aplicada; Termos descritores e operadores booleanos usados para pesquisar nas bases de dados PubMed, Science direct, Scopus e Web of Science: ALL FIELDS = "tocopherol coatings" AND "tocopherol films" e ALL FIELDS = "tocopherol coatings" OR "tocopherol films". Além disso, foi realizada uma análise bibliométrica, na qual ao realizar a busca nas bases de dados, com descritores e operadores booleanos, eles foram inseridos no software VOSviewer versão 1.6.17, no campo de busca "Tópico", que busca palavras em títulos, resumos, palavras-chave de autor e palavras-chave WoS – também chamadas de Keywords Plus, essas estratégias foram necessárias para a seleção do conjunto de dados final e análise de co-ocorrência de palavras-chave.
- No Capítulo 2 da tese foram desenvolvidos filmes por casting, a base de um blend de fécula de mandioca e carboximetil celulose adicionado de um mix de tocoferóis. Os filmes foram identificados como controle B (sem lecitina de soja e sem o mix de tocoferol), L (com lecitina de soja e sem mix de tocoferol) e LT e LT2 (com lecitina de soja e mix de tocoferol). Foram realizados testes de espessura, solubilidade em água, teor de água, gramatura, ângulo de contato, permeabilidade, testes mecânicos, atividade antioxidante, biodegradabilidade, cor, termogravimetria e FTIR.
- No Capítulo 3 da tese foram preparadas as mesmas formulações desenvolvidas no capítulo 2 da tese, porém foram aplicadas na forma de revestimento em castanhas-do-brasil para acompanhamento do grau de oxidação lipídica durante o tempo de armazenamento. Foram realizadas avaliações de estabilidade, viscosidade, pH e cor das soluções formadoras dos revestimentos. E as castanhas foram imersas nas soluções por 30s, seguida de secagem a 45°C por 19h e colocadas em incubadora a 25ºC, sendo avaliadas aos 1, 7, 15, 30, 45, 60, 90 e 120 dias de armazenamento. Foram realizados testes de perda de massa, índice de escurecimento, dienos e trienos conjugados, estado oxidativo por métodos oficiais e índice de oxidação acelerado.

CAPÍTULO 1

TIPO DE CAPÍTULO: REVISÃO SISTEMÁTICA E ANÁLISE BIBLIOMÉTRICA

PROPERTIES OF FILMS AND COATINGS ADDED OF TOCOPHEROL FOR FOOD PACKAGING: Tool-based review for systematic reviews and bibliometric analysis

Manuscrito publicado na *British Food Journal*

Ainda em fase de confecção do pdf de publicação (Carta de aceite **- Anexo A**)

PROPERTIES OF FILMS AND COATINGS ADDED OF TOCOPHEROL FOR FOOD PACKAGING: Tool-based review for systematic reviews and bibliometric analysis

Purpose
The aim was not to perform a systematic review but firstly to search in PubMed, Science Direct, Scopus, and Web of Science databases on the articles published in the last five years using tools for reviewing the statement of preferred information item for systematic reviews without focusing on a randomized analysis and secondly to perform a bibliometric analysis on the properties of films and coatings added of tocopherol for food packaging.
Approach
On January 24, 2022, information was sought on the properties of films and coatings added of tocopherol for use as food packaging published in PubMed, Science Direct, Scopus, and Web of Science databases. Further analysis was performed using bibliometric indicators with the VOS viewer.
Findings
The searches returned 33 studies concerning the properties of films and coatings added of tocopherol for food packaging, which were analyzed together for a better understanding of the results. Data analysis using the VOSviewer tool allowed a better visualization and exploration of these words and the development of maps that showed the main links between the publications.
Originality
In the area of food science and technology, the development of polymers capable of promoting the extension of the shelf life of food products is sought, so the knowledge of the properties is vital for this research area since combining a biodegradable polymeric material with a natural antioxidant active is of great interest for modern society, since they associate environmental preservation with food preservation.
Keywords: Antioxidant. Functionality. Review. Packaging. VOSviewer. Bibliometric research.

1 INTRODUCTION

Coatings and packaging are essentially adopted to minimize the deterioration of foodstuffs, reduce the risk of contamination, and keep the product safe to be marketed. This practice reduces sensory damage by providing semi-permeable blockage around the product (NAIR et al., 2020). Edible coatings extend the shelf life of foods because they inhibit oxidation and protect against pathogenic microorganisms and moisture (AL-TAYYAR et al., 2020; IQBAL et al., 2021; TAHIR et al., 2019a). Allied with these characteristics, the application of edible coating films can also promote food preservation through the incorporation of antimicrobial, antioxidant, and antifungal agents into the polymer matrix (EL-SAYED et al., 2020; TAHIR et al., 2019b).

Knowing the properties of films and coatings for food is essential from the point of view of developing a package that will have direct contact with the food product, as it may or may not produce desirable effects on the food. Therefore, several works seek not only to develop polymers but also to evaluate the peculiar properties of films or coatings (COSTA et al., 2022; EMRAGI; KALITA; JAYANTY, 2022; NURHAYATI et al., 2022; RADI; AHMADI; AMIRI, 2022). It also happens in films and coatings in which tocopherol is added to generate antioxidant properties. There is a wide range of studies on tocopherol-added polymers, such as the works of Agudelo-Cuartas et al. (2020) who developed whey protein-based films; Hamid et al. (2018) who developed carrageenan semi-refined films (SRC) from Eucheuma cottonii and Tongdeesoontorn et al. (2021) who developed cassava starch/gelatin films.

Bibliometric analysis of journals can be used by editorial boards to make decisions about developing future publications (MOKHTARI et al., 2020). In addition, it can contribute new ideas to researchers who study the development and use of food packaging (ÖĞRETMENOĞLU et al., 2021). In the works of Azevedo et al. (2022), Fasogbon & Adebo (2022), Rigueto et al. (2023), Vila-Lopez and Küster-Boluda (2021), and Wang et al. (2021) one observes the application of the bibliometric analysis is observed in the search for information on active flexible food films; a global and African view of 3D food printing; gelatin-based polymeric films for food packaging applications; research on sustainable food packaging; sustainable Chinese packagings, respectively.

However, a search and a bibliometric analysis that addressed information on assembly polymers/tocopherol properties for use in the food sector were not found. Thus, the aim of this research was not to perform a systematic review but firstly to search in PubMed, Science Direct, Scopus, and Web of Science databases on the articles published in the last five years using tools for reviewing the statement of preferred information item for systematic reviews without focusing on a randomized analysis and secondly to perform a bibliometric analysis on the properties of tocopherol-added films and coatings for food packaging.

2 FILMS AND COATINGS

Films and edible coatings are thin membranes applied to the surface of the food product to preserve shelf-life and quality (DÍAZ-MONTES; CASTRO-MUÑOZ, 2021). The difference between films and coatings is in the forming ingredient and the manner of application because edible coatings are usually applied directly on the product by dipping or spraying followed by a drying process, while films can be made by first spreading the polymer solution on support (casting), followed by drying and then applying it to the food (GALUS; KADZIŃSKA, 2015; MOHAMED; EL-SAKHAWY; EL-SAKHAWY, 2020).

The employment of biodegradable polymers as edible coatings is getting significant attention due to the environmental problems associated with non-degradable plastic materials (ABDEL AZIZ; SALAMA, 2021; SALAMA et al., 2021). Taking the advantages of biocompatibility, biodegradability, and edibility, biopolymers are considered ideal candidates for the production of edible coatings (ABDEL AZIZ; NAGUIB; SAAD, 2015; SALAMA; ABDEL AZIZ, 2020). The most commonly natural biopolymers used are starch, cellulose, chitosan, alginate, whey protein, and collagen (ROSSETO et al., 2020).

Polysaccharide-based are the most studied biopolymers, among them starch, cellulose, chitosan, and agar. The technological innovation in biopolymer technology has conduced to the development synthetic biopolymers like polylactic acid, polycaprolactone, polyglycolic acid, polyglycolic acid, polybutylene succinate, and polyvinyl alcohol. These synthetic biopolymers have several advantages over natural biopolymers, including better mechanical and other barrier properties (SHANKAR; RHIM, 2018).

The main advantages associated with biopolymers are their environment-friendly nature, renewability, biocompatibility, non-toxic, low cost, availability, and biodegradability (WANKHADE, 2020). The high-quality, eco-friendly, biodegradable, and natural base materials have gained demand in packaging applications, along with active ingredients that can extend the shelf life of food materials (MAHMUD; ISLAM; TAHERGORABI, 2021; VARGHESE; SIENGCHIN; PARAMESWARANPILLAI, 2020).

Each food has different packaging requirements and several quality factors, such as color, oxidation, microbiology, structure, flavor, enzymatic degradation, photooxidation, and chemical changes such as hydrolysis, protein denaturation, and cross-linking. That is, films and coatings for food use are highly complex to develop, as a strategy must be drawn up to develop the ideal packaging, respecting the peculiar aspects of the food (LINDSTRÖM; ÖSTERBERG, 2020).

The design for the development of eco-friendly package materials has received significant attention (DEHGHANI; HOSSEINI; REGENSTEIN, 2018) like materials based on protein or polysaccharide biopolymers used as an alternative to synthetic petroleum derivatives (DAMMAK et al., 2017). According to European Bioplastics (2022), the global capacity of bioplastics was 1,792 million tons in 2021, 2,217 million tons in 2022, and is forecast to reach 6,291 million tons in 2027, of which 58.50 %, 51.51 %, and 56.23 % respectively are biodegradable.

Complementarily, additives added to biopolymers can improve optical properties, mechanical strength, barrier properties, and other functionalities. Such active agents can, in many cases, be diffused for a long time into the food and extend the applied effect. However, because some assets are volatile, insoluble in water, and chemically unstable, the incorporation directly into the polymer matrix of these assets is a challenge, as it can negatively impact the properties of the film (RANJBARYAN; POURFATHI; ALMASI, 2019). Figure 1 shows advances in films and coatings - active packaging.

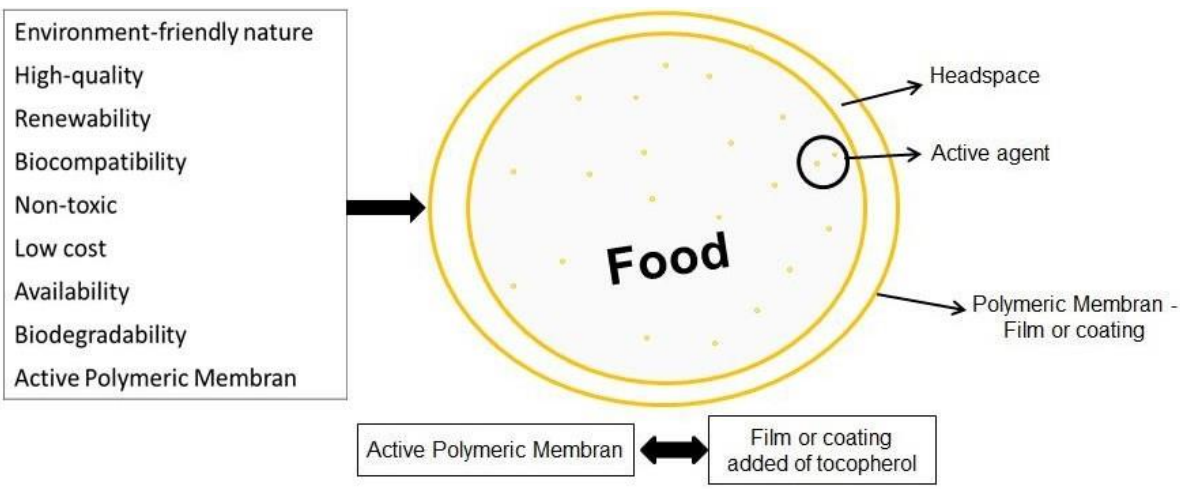

Figure 1. Advances in films and coatings - active packaging.

3 ACTIVE FILMS AND COATINGS

The first citation referring to the terms active packaging and intelligent packaging was made in Regulation 2004/1935 / EC of the European Parliament and the Council, which states that: "all substances incorporated into foodstuffs coming from packaging must meet the criteria set out in Directive 89 / 107 / EC on food additives" (EUROPEAN PARLIAMENT, 2004).

The primary mechanism consists of immobilizing the active compound in the polymer matrix by covalence to act immediately when the food is in contact with the film. In a second mechanism, the active compound is incorporated into the matrix in the dry state so that when the film is placed in contact with moist food, the compound is released, acting directly on the food (CHEN et al., 1996; BUONOCORE et al., 2005).

Active packaging is a promising future in the packaging market, with the ability to slowly release functional additives on the surface of food, with an antioxidant and antimicrobial role, extending the shelf life of products, storage of oils, the use of biodegradable packaging in the treatment of food spoilage is also of great importance (ESPITIA et al., 2014). Consequently, the effectiveness of edible films or coatings depends on three criteria: (i) the selected materials for their preparation, (ii) the technical and operational parameters of their application on the food product, and (iii) the specific requirements of the food product (BIZYMIS; TZIA, 2021).

Active packaging acts as a barrier to external detrimental factors and has an active role in food preservation, maintaining or prolonging its shelf-life. There is a diversity of active packaging systems that are comprised of additives that release properties, absorption, removal, and control of microbial and quality. Besides this, several studies are interested in the properties of essential oils and their actives, such as antioxidants, polyphenols, and tocopherols (ATARÉS; CHIRALT, 2016; ALFONZO et al., 2017; RIBEIRO-SANTOS et al., 2017; KUMAR et al., 2020).

Active food packaging is one of the new innovative packaging technologies that combine the food and packaging environment and their interactions to ensure the preservation of quality and increase the shelf life of food biological materials in natural polymers to protect the consumer and the environment by preserving food (YILDIRIM et al., 2017).

3.1 ANTIOXIDANT ACTIVITY IN THE POLYMERIC FILMS OR COATINGS AND ACTIVE FILMS AND COATINGS WITH TOCOPHEROL

When a polymeric matrix is developed by adding antioxidant compounds, some advantages are observed, such as protection against free radicals and minimization of oxidation. Together with other benefits to food systems, they could present anti-inflammatory and antimicrobial action (BRITO et al., 2021). Films and coating containing active antioxidant agents prolong the food shelf life, and these agents are incorporated into films and coating (KUMAR et al., 2021; TANWAR et al., 2021).

Antioxidants are stable molecules, and they can donate electrons to unstable molecules. These antioxidants react with unstable molecules known as free radicals and reactive oxygen species (ROS) and terminate the chain reaction that can spoil the food products (LOBO et al., 2010). They inhibit or delay food oxidation by limiting the initiation or propagation of oxidative chain reactions (SINGH et al., 2022).

Antioxidants have been incorporated as active ingredients into plastic films for polymer stabilization, protection from oxidative degradation, and prevention of discoloration, rancidity, and food degradation (DUTTA; SIT, 2023). Among the most common is tocopherol (AVRAMESCU et al., 2020). Natural agents such as polyphenols, tocopherols, plant extracts, and essential oils are becoming increasingly popular in the application of active packaging materials (IVERSEN et al., 2022).

Tocopherols are widely known for preventing lipid oxidation in food products. In addition, tocopherols come in four different forms (β, α, γ, and δ) (MOURE et al., 2001; BAROUH et al., 2022). The addition of tocopherol in films and coatings has been studied in several studies over time due to its antioxidant action, such as in the works of Zhu et al. (2012), Dias et al. (2018), Ferreira et al. (2021), and Keshari et al. (2022) which developed low-density polyethylene (LDPE)/polypropylene (PP) blend films; studied chitosan films on salmon fillet; applied coatings of thermoplastic starch and chitosan with α-tocopherol/bentonite in special green coffee beans; and applied sodium alginate coating on minimally processed carrots, respectively.

4 METHODOLOGY

This review was performed by stating preferred reporting items for systematic reviews and meta-analyses (PRISMA) (MOHER et al., 2009) but without focusing on a randomized analysis. On January 24, 2022, information on the properties of tocopherol-added films and coatings for use as food packaging published in PubMed, Science Direct, Scopus, and Web of Science databases was sought. Research articles from the year 2017 to 2022 were searched. No language restrictions were applied. The descriptor terms “tocopherol coatings” and “tocopherol films" were applied in ALL FIELDS, and the Boolean operators “AND” and “OR” were used to search the PubMed, Science Direct, Scopus and Web of Science databases.

When performing a search in the databases, with descriptors and Boolean operators listed, they were inserted in the "Topic" search field, which searches for words in titles, abstracts, author keywords, and WoS keywords – also called Keywords Plus. These strategies were necessary for the selection of the final dataset. The records were exported, according to the necessary formatting of the document about each database, to be then analyzed through an analysis of co-occurrences of keywords, which allows the visualization of spatial proximity and shows the relationships between the data and the information found (INOMATA et al., 2015).

Keyword co-occurrence analysis was performed using VOSviewer version 1.6.17, a software tool to create maps based on network data and to visualize and explore these maps (VAN ECK; WALTMAN, 2021). The graphics were constructed with at least 5 occurrences of the keywords among the works published in the "Web of Science" databases; "Science Direct," "PubMed" and at least 20 occurrences for those from "Scopus," as this database presented a more significant number of files to be analyzed when compared to the others, so to improve the visualization of information in the graphs it was essential to increase the minimum number of keywords. Two evaluators analyzed the dataset obtained; a third evaluator analyzed the material in case of doubt. Duplicate articles were excluded, and after reading the title and abstract, articles that did not meet the inclusion criteria were excluded.

Figure 2 shows the evaluation flowchart of the articles resulting from the bibliographic survey.

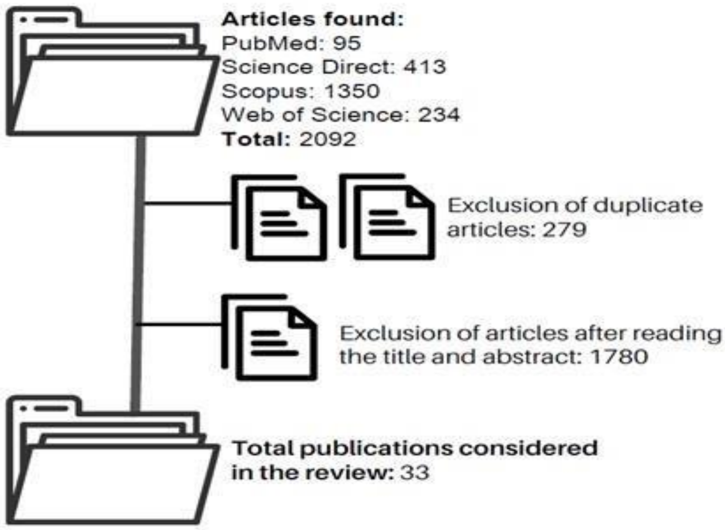

Figure 2. Evaluation flowchart of articles resulting from the search in PubMed, Science direct, Scopus, and Web of Science databases.

Figure 3 shows the step-by-step of the entire bibliographic search in the databases until the bibliometric analysis to obtain the graphs in the Vosviewer software.

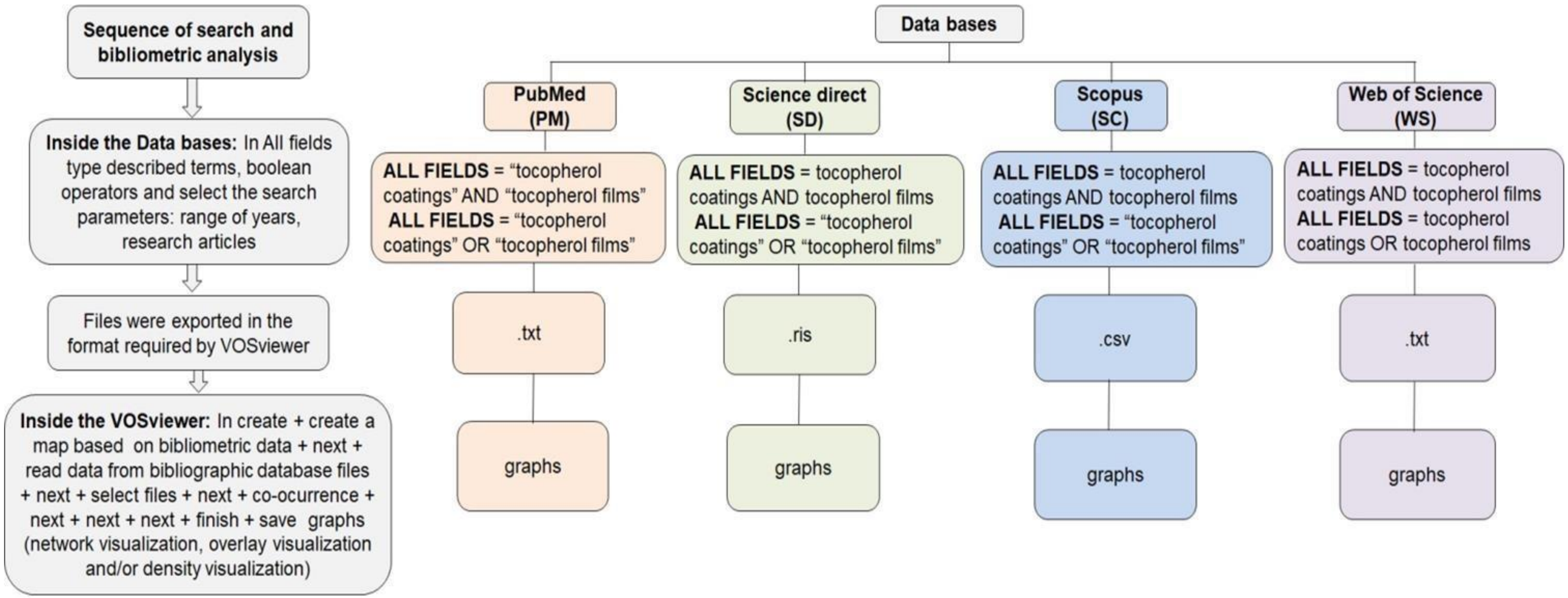

Figure 3. Step-by-step of the entire bibliographic search in the databases until the bibliometric analysis to obtain the graphs in the Vosviewer software.

5 RESULTS AND DISCUSSION

5.1 FILMS AND COATINGS ADDED OF TOCOPHEROL FOR FOOD PACKING

The survey returned 33 studies referring to the properties of films and coatings added with tocopherol for food packaging (Table 1).

Table 1. Systematization of the 33 works referring to the properties of films and coatings added with tocopherol for food packaging resulting from searches in PubMed, Science Direct, Scopus, and Web of Science databases.

N°	ARTICLE TITLE	USE	PURPOSE OF THE ARTICLE	COMPOSITION MATERIAL	PROPERTIES EVALUATED IN FILMS/COATINGS OR FOOD PRODUCTS	PRINCIPAL RESULTS OF THE ARTICLE	REF.
01	Development of chitosan based extended-release antioxidant films by control of fabrication variables	Films	Evaluate the influence of the concentration of tween 80, as emulsifying agent, the stirring speed of homogenization and the presence of ethanol, as the solvent of α -tocopherol, on the physicochemical properties of the α -tocopherol incorporated chitosan film and the release of α-tocopherol into ethanol 95 %, as the fatty food simulant	- Chitosan - Glycerol - Tween 80 - α-tocopherol	- Thickness - Water content - Opacity - Solubility in water - Tensile strength, Elongation at break - WVP - Release of α-tocoferol - FTIR - DSC	Incorporation of the α-tocopherol, changes the textural and optical properties of chitosan films. Preparing conditions including concentration of emulsifier and speed of homogenization as well as incorporation of ethanol as a co-surfactant could affect the release rate of antioxidant. The higher concentration of emulsifier and higher speed of homogenization reduced the release rate of antioxidant. The addition of ethanol strongly decreased the rate of tocopherol release in the early stages of measurement. Promoting a slower, proper start and proper later releases. Therefore, increasing the stirring speed of homogenization and ethanol addition produced an adequate release of α-tocopherol from chitosan-based films, promoting adequate long-term conditions to minimize lipid oxidation of foods	Darbasi et al. (2017)

02	Edible carboxymethyl cellulose films containing natural antioxidant and surfactants: α-tocopherol stability, in vitro release and film properties	Edible film	Develop edible films containing Carboxymethylcellulose (CMC), α-tocopherol (α-TC) as an antioxidant and surfactants for food applications	- CMC - Tween 80 - Lecithin - α-tocopherol	- Thickness - SEM - WVP - Tensile strength, Elongation at break, modulus of elasticity - In vitro release and quantification - DPPH and ABTS	Tocopherol incorporated into CMC films showed satisfactory stability over 8 weeks. It was possible to control the tocopherol release profile from the CMC matrix by altering the ratio of lipophilic/hydrophilic surfactant used to stabilize the tocopherol droplets in the polymer. The addition of lecithin to the CMC films helped to maintain the stability of tocopherol after its release due to chemical interactions, which contributed to the higher antioxidant activity	Martelli et al. (2017)
03	Physical and antioxidant properties of films based on gelatin, gelatin-chitosan or gelatin-sodium caseinate blends loaded with nanoemulsified active compounds	Films	Develop and characterize active gelatin-based films, gelatin-chitosan or gelatin-sodium caseinate mixtures, apply active compounds (α-tocopherol, garlic essential oil and cinnamaldehyde) nanoemulsified in water	- Gelatin - Chitosan -Sodium caseinate - Glycerol - α -tocopherol	- Thickness - Water content - Solubility in water and swelling - WVP - FTIR - SEM - Contact angle - ABTS	All films added with tocopherol nanocapsules showed antioxidant activity, but the film with the highest activity was the films obtained from the Gelatin-Sodium Caseinate mixture. The nanoencapsulated active compounds were well distributed throughout the biopolymer matrix. Films developed based on gelatin and gelatin-chitosan or gelatin-sodium caseinate blends loaded with NACs showed adequate physical properties and strong antioxidant activity	Pérez-Córdoba, Sobral (2017)
04	Efficacy of whey protein coating incorporated with lactoperoxidase and α-tocopherol in shelf life extension of Pike-Perch fillets during refrigeration	Coati ng	Design an active coating package based on whey protein incorporated with Lactoperoxidase system (LPOS) and α-tocopherol for the control of two main factors involved in the deterioration of food quality in to prolong the shelf life of pike perch fillets (Sander lucioperca, Linnaeus 1758) stored under refrigeration (4°C)	- Whey protein - Ethanol - LPOS - D-α -tocopherol	In fillet of the fish: - Psychrotrophic bacteria determination - pH - Thiobarbituric Acid - Total volatile basic nitrogen - Sensory evaluation	The results indicated that whey protein coating incorporated with LPOS and α-tocopherol could maintain the microbial, chemical, and sensory qualities of Pike-Perch fillets during 16 days of refrigeration storage (4ºC). Although interaction between LPOS and α-tocopherol in some cases led to the mutual antagonistic effect in both cases, the obtained results indicated that the combination of LPOS and α-tocopherol can donate antibacterial and antioxidant properties to WPS coating	Shokri, Ehsani (2017)

05	Influence of α-tocopherol/MCM-41 assembly on physical and antioxidant release properties of low-density polyethylene antioxidant active films	Films	Investigate the influence of addition amount of α-tocopherol/ mesoporous silica (Mobil Composition of Matter No. 41-MCM-41) assembly on the properties of low-density polyethylene (LDPE) films including physical properties and release profile of the antioxidant in the films	- LDPE - MCM-41 - α-tocopherol	- SEM - FTIR - XRD - DSC - WVP - Transmittance - Tensile strength, Elongation at break - Migration Test	The addition of the α-tocopherol/MCM-41 set has little effect on the melting temperature of the LDPE films, however the films showed a decrease in crystallinity with the increase in the amount of the set, the same trend occurred in the evaluation of tensile strength and film stretching. The antioxidant release rate can be affected by the addition of the set, it was found that large addition of the set can contribute to the slow release of the antioxidant in the polymer	Sun et al. (2017a)
06	Development of low-density polyethylene antioxidant active films containing α-tocopherol loaded with MCM-41 (Mobil Composition of Matter No. 41) mesoporous silica	Films	To develop a new type antioxidant active packaging of low density polyethylene (LDPE) containing α-tocopherol adsorbed on MCM-41 mesoporous silica	- LDPE - MCM-41 - α-tocopherol	- Thickness - XRD - FTIR - TGA - WVP - GP - Tensile strength, elongation at break - Quantification of α-tocopherol in fatty food simulant - Migration Test - DPPH	In the migration tests, the developed films showed that the adsorption of α-tocopherol in MCM-41 has a significant influence on the antioxidant release profile, the diffusivity for adsorbed a-tocopherol decreased approximately 53% compared to that of free a-tocopherol and water vapor permeability increased. α-Tocopherol maintained its antioxidant activity in the newly developed film	Sun et al. (2017b)
07	Effect of active films incorporated with montmorillonite clay and α-tocopherol: Potential of nanoparticle migration and reduction of lipid oxidation in salmon	Films	To evaluate the montmorillonite (MMT15A) and α-tocopherol migration potential and antioxidant effect of chitosan/MMT15A/α-tocopherol active films on reduction of lipid oxidation in fresh salmon	- Chitosan - MMT15A - α- tocopherol	- Thickness - Energy-dispersive X-ray spectroscopy - WVP In salmon fish: - Thiobarbituric Acid - Water content - Ether extract - Ashes - Quantification of tocopherol - Color analysis - Minerais (Mg e Si)	The use of chitosan films with 15% α-tocopherol + 1% MMT15A is recommended in order to obtain high barrier of vapor permeability and a controlled release of α-tocopherol at storage time, and it can be used as a packaging antioxidant and enrich nutritionally the food	Dias et al. (2018)

08	Physico-chemical, antimicrobial and antioxidant properties of gelatin-chitosan based films loaded with nanoemulsions encapsulating active compounds	Films	To develop and characterize gelatin-chitosan based films that incorporate nanoemulsions loaded with a range of active compounds (α-tocopherol, cinnamaldehyde, garlic oil)	- Gelatin - Chitosan - canola oil - Cinnamaldehyde - Garlic oil - Glycerol - Tween 20 - α-tocopherol	- Thickness - Water content - Solubility in water and swelling - Tensile strength, elongation at break and modulus of elasticity - Light transmission and transparency - XRD - DSC - Atomic force microscopy analyses - SEM - Antimicrobial activity - DPPH, ABTS, FRAP	The films demonstrated a homogeneous structure with good distribution of nanoencapsulated active compounds (NAC) throughout the biopolymer matrix and without unfavorable effects on the original film thickness, water content, glass transition and melting temperature. The nanoemulsion filler increased the film's resistance to water, reducing its solubility and increasing the film's elongation at break and light barrier properties, in addition to directly affecting its transparency, reducing its tensile strength and stiffness and increasing its stiffness. surface. Nanoemulsions encapsulating active compounds are suitable for producing G-Ch-based films	Pérez-Córdoba et al. (2018)
09	PVA antioxidant nanocomposite films functionalized with α-tocopherol loaded solid lipid nanoparticles	Films	To develop active packaging films of PVA incorporated with different amounts of α-tocopherol-loaded SLN and to evaluate the influence of these nanoparticles on by fluorescence analysis, antioxidant activity, α-tocopherol release in fat food simulant, as well as morphology, X-ray, thermal properties and contact angle	- Polyvinyl alcohol - Soy lecithin - α-tocopherol	- SEM - Wettability and surface free energy - FTIR - XRD - TGA and DSC - Fluorescence analysis - DPPH and ABTS - Release of α-TC from PVA films incorporated with α-TC-NLS	Poly (vinyl alcohol) (PV) films embedded with solid lipid nanoparticles (NL) containing α-tocopherol (TC) at different concentrations showed good stability for 12 weeks. The PV/TC-NL films showed a rapid initial release followed by an equilibrium state between the α-TC transferred through the film to the simulator and the natural migration of α-TC from the simulator to the film. The rate of α-TC release increased with increasing percentage of α-TC-NL added to PV/TC-NL films, explaining the higher antioxidant activity with increasing addition of α-TC-NL to PV/TC-NL films. Morphologically showed that the incorporation of α-TCNL was homogeneous and resulted in a matrix with a rough surface and less cohesive cross-section with greater volume than pure PVA. The films added with NL tocopherol showed greater thermal stability and lower degree of crystallinity than the pure PVA films	De Carvalho et al. (2019)

10	Supercritical CO_2 impregnation of α-tocopherol into PET/PP films for active packaging applications	Films	Obtain an active packaging, using SC-CO_2 that incorporates TOC into multilayer PET/PP films. To optimize the asset packaging, a comparison between a film in which TOC is impregnated on the surface of untreated PET (ut-PET) and a film in which TOC is adsorbed on the surface of PET subjected to corona discharge treatment (ct-PET).	- Polyethylene terephthalate - Polypropylene - α-tocopherol	- Field emission scanning electron microscopy (FESEM) - FTIR - DSC - Migration Test - DPPH	Obtaining loaded tocopherol in PP films was the best option to produce a controlled release package with high TOC loading values. The results obtained for monolayers, to create multilayer active films, impregnation of TOC with SC-CO_2 were studied considering the PP surface of the PET/PP film. Migration tests demonstrated that impregnation of TOC in polymeric films using $SCCO_2$ induced a prolonged release of the vitamin, confirming that the controlled release in the package production process was effective	Franco, Incarnato, De Marco (2019)
11	Semi-refined carrageenan (SRC) film incorporated with α-tocopherol: Application in food model	Films	To develop and characterize active packaging film from SRC plasticized with glycerol (G) and incorporated with different concentrations of α-tocopherol (0.1%, 0.2%, 0.3%, and 0.4% [v/v]).	- Semi-refined carrageenan - Glycerol - α-tocopherol	- Thickness - FTIR - TGA - SEM In meat patties: - Thiobarbituric Acid - Metmyoglobin assay - pH	Themally of the SRC-based film improved when α-tocopherol and G were incorporated into the film matrix. The antioxidant effect of α-tocopherol in the SRC-based films was tested using beef burgers and the greatest antioxidant effect was demonstrated by incorporating the highest concentration of α-tocopherol into the SRC-based film. The antioxidant film delayed the development of lipid oxidation and the formation of brown coloration in the hamburgers during storage	Hamid et al. (2019a)
12	Semirefined Carrageenan (SRC) Film Incorporated with α-Tocopherol and Persicaria minor for Meat Patties Application	Films	To analyse the antioxidant effect of new semi-refined carrageenan (SRC) active packaging films that incorporated α-tocopherol (0.4% [v/v]) and Persicaria minor (PM) (0.4% [v/v]) in beef burgers, in addition, changes in pH and brown color development in ground beef burgers stored for 14 days at 4 °C were evaluated	- Semi-refined carrageenan - Persicaria minor - Glycerol - α -tocopherol	- FTIR - Total phenolic content (TPC) - DPPH, ABTS and FRAP In meat patties - Thiobarbituric Acid - Metmyoglobin assay - pH	α-Tocopherol and Persicaria minor (PM) extract exhibited different levels of phenolic content and antioxidant activity. The addition of α-tocopherol and PM extract into the SRC-based films delayed the lipid oxidation and metmyoglobin formation in the meat patties throughout the 14-day refrigerated storage	Hamid et al. (2019b)

13	Preparation of low-density polyethylene film with quercetin and α-tocopherol loaded with mesoporous silica for synergetic-release antioxidant active packaging	Films	To develop an antioxidant active LDPE film containing α-tocopherol controlled by Mesoporous Molecular Sieves MCM-41 and Quercetin	- Low-density polyethylene - MCM-41 - Quercetin - α-tocopherol	- Tensile strength, elongation at break -WVP - GP - Migration Test - DPPH	The adsorption capacity of α-tocopherol is approximately 40% by weight. The migration test proves that being loaded with MCM-41 decreases α-tocopherol diffusivity by approximately 48.2%, while increasing quercetin diffusivity by approximately 39.5% (MCM-41+α-tocopherol+quercetin) and 50.5% (α-tocopherol+quercetin) after the introduction of α-tocopherol than Q. The DPPH radical scavenging increased after the addition of α-tocopherol	Li et al. (2019)
14	Optimisation, antioxidant attributes, stability and release behaviour of carboxymethyl cellulose films incorporated with nanoencapsulated vitamin E	Films	To optimize vitamin E (α-tocopherol) loaded polycaprolactone (PCL) nanocapsules into the carboxymethyl cellulose (CMC) film	- CMC - PLC - Glycerol - α-tocopherol	- DPPH - Quantification of initial α-tocopherol in film samples - Release test of α-tocopherol nanocapsules from film samples - Release kinetics of encapsulated ingredient	The preparation of α-tocopherol nanocapsules using polycaprolactone by the nanoprecipitation method was successfully performed, considering the high encapsulation efficiency and favorable suspension stability obtained in this research, which means that the encapsulation of this ingredient in industrial films used in packaging and factories of food is also applicable. The antioxidant properties of the core material and the controlled release of α-tocopherol (from these films) in fatty foods are among the most important effects of these nanoparticles observed in this research	Mirzaei-Mohkam et al. (2019)

15	Poly (-Dodecyl-Glutamate) (PAAG-12) and Polylactic Acid Films Charged with α-Tocopherol and Their Antioxidant Capacity in Food Models	Films	PAAG-12 films were prepared and enriched with 5% α-tocopherol, with the aim of using them as novel antioxidant active packaging for food applications.	- PAAG-12 - Polylactic Acid (PLA) - α-tocopherol	- SEM - TGA and DSC - Food simulation	The increase in the initial temperature of the PAAG-12 film by the addition of the natural antioxidant α-tocopherol validates the improvement in the thermal stability of the branched polymer, which implies better processability for industrial applications in food packaging. When the concentration of ethanol was higher in the simulators, the migration of α-tocopherol from the films was higher. PLA allowed greater migration of antioxidants to the food simulation medium than PAAG-12 in short contact times, which demonstrates that this new polymer is a promising matrix for applications in active packaging. The peroxide test of the oil/water emulsions showed high levels of protection of the active films, capable of increasing the shelf life of up to 29 days	Villasante et al. (2019)
16	Preparation of α-tocopherol-chitosan nanoparticles/chitosan/ montmorillonite film and the antioxidant efficiency on sliced dry-cured ham	Films	To prepare a novel antioxidant film by incorporating α-tocopherol-chitosan nanoparticles (TOC-CSNPs) with chitosan/montmorillonite film (namely, TOC-CSNPs/CS/MMT), and investigate the antioxidant activity of TOC-CSNPs/CS/MMT film on sliced dry-cured ham in a period of 120 days at 4 °C	- Chitosan (CS) - Montmorillonite (MMT) - Acetic acid - α-tocopherol (TOC)	- Solubility in water - Swelling ratio - WVP - FTIR - SEM - Cumulative release of TOC - DPPH In Sliced dry-cured ham: - Peroxide value - Thiobarbituric Acid	TOC-CSNPs/CS/MMT film with added TOC-CSNPs demonstrated long-term, stable and enhanced antioxidant activity during 120 days of storage of sliced cured ham. The film can be applied as edible packaging wrap for food products, maintaining quality and prolonging shelf life without chemical preservatives	Yan et al. (2019)

17	Characterization and release kinetic of crosslinked chitosan film incorporated with α-tocopherol	Films	To produce the in-situ crosslinking emulsification chitosan film by the casting solution method and to study the effect of sodium tripolyphosphate (TPP), sodium citrate (CT), and glutaraldehyde (GLU) for the physical properties, barrier properties, mechanical properties, and release kinetics of chitosan film incorporated with α-tocopherol	- Chitosan - Acetic acid glacial - Glycerol - Tween 80 - α-tocopherol	- Color and light transmission - SEM - WVP - Elongation at break, Tensile strength, Young's Modulus - FTIR - Contact angle - Release of α-tocopherol and estimation of the diffusion coefficient	The cross-linking emulsification process in situ was successful and demonstrated the influence of the cross-linking agent on the properties and release kinetics of chitosan incorporated with α-tocopherol, in addition, the cross-linking agent decreased the film luminosity, barrier properties to light and increased the green and yellow of the film, in addition to reducing the EB and TS values. But, the hydrophobicity and roughness of the film increased and there was no significant difference in the water vapor barrier	Yeamsuksawat, Liang (2019)
18	Characterization of whey protein-based films incorporated with natamycin and nanoemulsion of α-tocopherol	Films	Evaluate the effect of adding natamycin, α-tocopherol nanoemulsion and a mixture of them, on chemical, physical, mechanical, antioxidant and antimicrobial properties of whey protein-based films	- Natamycin - Whey protein concentrate - Glycerol - α-tocopherol	- Thickness - Water content - Solubility in water - Tensile strength, elongation at break and modulus of elasticity - Color, opacity and UV-Vis light barrier - WVP - SEM - FTIR - DPPH, ABTS and FRAP - Antimicrobial activity	The addition of natamycin, nanoemulsified α-tocopherol or both did not change the water content of the whey protein-based films. They led to a significant reduction in tensile strength and modulus of elasticity, while showing growth in elongation at break, film opacity, total color difference, UV-Vis light barrier, and water vapor, with the addition of the compounds there was an increase in permeability values. The film showed uniform porosity. The activity of the α-tocopherol nanoemulsion remained during its addition to the films	Agudelo-Cuartas et al. (2020)
19	Release of α-tocopherol from chitosan/pectin polyelectrolyte complex film into fatty food simulant for the design of antioxidant active food package	Films	Use as packaging material and α-tocopherol (α-TOH) as antioxidant and polyelectrolyte complex (PEC) of chitosan (CS) and pectin (PE) to develop an antioxidant active packaging with the addition of Tween-80 to facilitate incorporation of hydrophobic α -TOH into hydrophilic PEC CS/PE solution	- Chitosan - Pectina - Tween 80 - Acetic acid - α-TOH	- FTIR - Tensile strength - Solubility in water - Release Study - DPPH	PEC CS/PE composition, Tween–80 concentration and α-tocopherol concentration affected the α-TOH release profile. The hydrophilicity of the film increased with increasing pectin content in PEC and Tween-80 concentration, leading to an increase in the accumulated release of α-TOH. The increase in the concentration of incorporated α-TOH also promoted an increase in the release of α-TOH due to its plasticizing effect. The complex films exhibited high antioxidant activity of up to 90.60%. The release profile of all films exhibited an initial burst effect followed by sustained release over 10 d	Hapsari, Roto, Siswanta (2020)

20	Eco-friendly materials produced by blown-film extrusion as potential active food packaging	Films	To use the blown-extrusion technique to obtain fully biodegradable and low-cost starch/PBAT blends incorporated with α-tocopherol as antioxidant	- poly (butylene adipate-co-terephthalate) (PBAT) - Native cassava starch - Glycerol - α-tocopherol	- Thickness - Tensile strength, Elongation at break, Young's modulus - WVP - Color and opacity - Weight loss in water (WLW) - SEM - TGA - Wide angle X-ray diffraction (WAXD) - Release profile of α-tocopherol from pellets and films - Degradation efficiency of the films by composting	The processability of the films was adequate, even with the inclusion of α-tocopherol. The hydrophobic character of α-TOC starch probably destabilized the matrix/PBAT, which was demonstrated by SEM images. This increases water vapor permeability and reduces performance, regardless of antioxidant concentration. X-ray patterns offer the diffusion complexity crystallization of amyl. The formulation containing the lowest concentration of α-TOC was almost complete, favoring its application as food packaging. The assets offered biodegradability. Demonstrating that active films based on starch/PBAT with low α-tocopherol added (0.25 g.100 g^{-1}) are an alternative to non-degradable food packaging materials	Lopes et al. (2020)
21	Biodegradable Poly(ε-Caprolactone) Active Films Loaded with MSU-X Mesoporous Silica for the Release of α -Tocopherol	Films	Develop and characterize new films active PCL-based containing α-tocopherol and MSU-X mesoporous silica	- PCL - MSU-X - α-tocopherol	- TGA and DSC - WVP - Oxygen transmission rate (OTR) - Optical Properties - Release Tests - DPPH and ABTS - Antimicrobial Activity	Both PCL-AD (direct addition of TOC and MSU-X) and PCL-IMP (MSU-X impregnated with TOC silica) films demonstrated good thermal stability and showed no significant changes in oxygen and water vapor barrier properties. The increase in the values of the oxidation onset parameters (oxidative onset temperature-OOT and oxidative induction time-OIT) obtained for these formulations indicated the effectiveness of the addition of mesoporous silica and antioxidant TOC to protect the final material from oxidation and thermal degradation, favoring its processing at high temperatures and later use. PCL-IMP showed a slower antioxidant release in 50% ethanol (v/v) than the other films (PCL-TOC) and (PCL-AD). The antioxidant diffusivity of PCL-IMP films decreased 10-fold compared to films containing free α-tocopherol. PCL-IMP and PCL-AD films exhibited greater antibacterial activity against Gram-positive strains (S. aureus) and PCL-TOC film against Gram-negative bacteria (E. coli)	Mellinas et al. (2020)

22	Physical, mechanical, thermal and structural characteristics of nanoencapsulated vitamin E loaded carboxymethyl cellulose (CMC) films	Films	To study the effect of nanoencapsulation of α-tocopherol (TOCNPS) in CMC films on film properties to understand whether this useful change can improve film characteristics as it is very important to deliver food products in packages which can meet customers' expectations e.g. to be resistant against environmental changes (mechanical, thermal, humidity, etc.) and fluctuations	- CMC - Glycerol - Lechitin - α -tocopherol	- Thickness - Transmittance - Color properties - Contact angle - WVP - Tensile strength, Elongation at break, Young's modulus - DSC - FTIR - SEM	The properties of carboxymethyl cellulose films form improved with the addition of α-tocopherol nanocapsules, the nanoparticles may be the cause of porosity and changes in the structure of the film matrix, which according to the research results, these films can influence mainly, with regard to water vapor permeation	Mirzaei-Mohkam et al. (2020)
23	Hydroxypropyl methylcellulose or soy protein isolate-based edible, water-soluble, and antioxidant films for safflower oil packaging	Films	To develop edible, antioxidant, heat-sealable, oil-resistant, and water-soluble packaging	- 1hydroxypropyl methylcellulose (HPMC) - oleic acid (OA) - Soy protein isolate (SPI) - Cellulose nanocrystals (CNC) - Glycerol - DL-α-tocopherol acetate (VE)	- Color - Transparency - Opacity - SEM - WVP - Water solubility - Film disintegration - Oil permeability - Contact angle - Tensile strength, Elongation at break, Young's modulus In safflower oil: - Peroxide value	Packages were developed based on hydroxypropylmethyl cellulose (HPMC) and soy protein isolate (SPI), with combinations of DL-α-tocopherol acetate, oleic acid and CNCs. The HPMC-derived films showed good strength and were highly water soluble at 20 to 40 °C. Low concentration of CNCs improved the film barrier and mechanical properties. SPI films showed highly elastic characteristics, disintegrated in water over a wide temperature range (20 to 90 °C), and maintained superior antioxidant protection of safflower oil compared to HPMC films and a polypropylene control, with an estimated lifetime of more than one year based on lipid oxidation	Rosenbloom, Zhao (2020)
24	Characterization of α-tocopherol-loaded MCM-41 mesoporous silica with different pore sizes and	Films	To investigate the influence of pore size and morphology of MCM-41 on physical properties of controlled release LDPE films, and the effect of these factors on the release profiles of α-tocopherol from controlled	- LDPE - MCM-41 - α-tocopherol	- DSC - Tensile strength, Elongation at break, Young's modulus - SEM - Migration test	Was investigated the influence of mesoporous silica MCM-41 loaded with α-tocopherol, with different pore sizes and antioxidant active packaging films, the main result found was that the pore size and particle size of the antioxidant used in the controlled release packaging films should be comparable for a good controlled release effect	Sun et al. (2020)

25	Effect of α-tocopherol antioxidant on rheological and physicochemical properties of chitosan/zein edible films	Edible films	To fabricate edible film containing α-tocopherol as an antioxidant packaging for food applications	- Chitosan - Zein - α-tocopherol	- Rheological analysis - Particle size and zeta potential - Thickness - Tensile strength, Elongation at break - WVP - Opacity - XRD - SEM - DPPH	Was produced of a chitosan/zein-based edible film incorporating α-tocopherol as antioxidant packaging for food applications, the results showed that all solutions forming the composite film showed excellent stability, with good barrier properties, opacity. Evidencing the compatibility of α-tocopherol and chitosan/zein in edible films	Zhang et al. (2020a)
26	Combined antioxidant and sensory effects of active chitosan/zein film containing α-tocopherol on *Agaricus bisporus*	Films	To prepare active packaging films which were incorporated with α-tocopherol and evaluate its effect on the postharvest quality, antioxidant enzymatic system and bioactive compounds contents of *Agaricus bisporus*	- Chitosan (C) - Zein (Z) - Glycerol - α-tocopherol	- Package atmosphere composition In Mushroom: - Weight loss - Firmness - Membrane permeability - Respiration rate - Browning degree - Polyphenol oxidase (PPO) and peroxidase (POD) activity - Malondialdehyde (MDA) content - Total phenolic content - Catalase (CAT) activity - Superoxide dismutase (SOD) activity - DPPH	The active packaging film composed of chitosan/zein containing α-tocopherol proved to be efficient in reducing the postharvest quality of mushrooms at 4 °C. Where in all treatments the mushroom treated with the film showed the highest firmness, catalase, superoxide dismutase activities, total phenolic content and DPPH radical scavenging activity, showing that the film could improve antioxidant properties and maintain mushroom quality	Zhang et al. (2020b)

27	Chitosan-nanocomposites as a food active packaging: Effect of addition of tocopherol and modified montmorillonite	Films	To evaluate the effect of tocopherol concentration (0, 5, 10, and 20%) and modified montmorillonite clay (MMT15A) nanoparticles (0 and 1%) on the properties of chitosan (CS) films	- Chitosan (CS) - Montmorillonite (MMT) - DL-α-tocopherol acetate	- Transmission electron microscopy - SEM - Colorimetric parameters - Transparency - Tensile strength, elastic modulus - DPPH - Contact angle - Moisture sorption - WVP - TGA and DSC	The application of tocopherol provided antioxidant activity, increased the thermal stability of the film. This resulted in the development of antioxidant bionanocomposites with improved properties both for packaging and for foods that had their nutritional properties enriched by the addition of tocopherol	Dias et al. (2021)
28	Optimization of PCL Polymeric Films as Potential Matrices for the Loading of α-Tocopherol by a Combination of Innovative Green Processes	Films	To compare two different polymeric structures: nanofibrous films obtained by electrospinning and continuous films obtained by solvent casting, to identify the best solution and process conditions for subjecting the samples to the supercritical fluids impregnation process (SFI)	- Polycaprolactone (PCL) - Polyethylene glycol (PEG) - α-tocopherol	- FESEM. - Migration tests	The polymeric support was produced both by electrospinning, by pouring solvent, and then it was loaded with alpha-tocopherol by impregnation with $SCCO_2$.The authors noted that the optimal operating conditions must be properly selected to obtain an active package	Drago et al. (2021)
29	Antioxidant edible film based on a carrot pectin-enriched fraction as an active packaging of a vegan cashew ripened cheese	Edible films	To determine the filmogenic performance of CPEF, the capability of the film network to stabilise at 25 °C the orange color and hence the carotenoids responsible for it, and finally evaluate the antioxidant capacity of the edible film for the preservation of a vegan cashew ripened cheese during storage	- Commercial pectin (CP) - Pectin-enriched fraction from carrots (CPEF) - Glycerol - α-carotene - β-carotene - Lutein - α-tocopherol	- Moisture content, water activity and pH - DSC - Color - Thickness - Water solubility - Contact angle - WVP - Tensile strength, Elongation at break - FTIR - Determination of carotenoids - In vegan ripened cheese made of cashew nuts	Evaluated antioxidant edible film based on carrot pectin enriched fraction for the preservation of a vegan matured cashew cheese during storage. As main results, it was evidenced that 100% CPEF films stabilized orange color even under light storage at 25 °C and 57.7% RH, and carotenoids were lost according to a first-order kinetics. In addition, films containing CPEF showed high resistance to dissolution in water. These properties made the 100% CPEF film an effective material to preserve, during 60 days of storage at 7 °C, foods with high aW (0.952) and vulnerable to oxidation such as vegan cured cashew cheese	Encalada et al. (2021)

					- TBARS - MDA		
30	Active coatings of thermoplastic starch and chitosan with α-tocopherol/bentonite for special green coffee beans	Coatings and films	To incorporate α-tocopherol, a powerful antioxidant, in thermoplastic starch (TPS) and chitosan (TPC) and determined the best cavitation energy (960–3840 J·mL^{-1}) using an ultrasonic probe	- TPS - TPC - Soy lecithin - Bentonite (BNT) - α-tocopherol	- DPPH - Contact angle and surface energy - XRD - FTIR - WVP - Puncture strength (PS) - TGA and DSC - Compressive load supported by coated green coffee beans	Was developed active coatings of thermoplastic starch and chitosan with α-tocopherol/bentonite for specialty green coffee beans, was observed that the combination chitosan/ α-tocopherol/ bentonite, dispersed with energy of 960 J-mL^{-1}, is effective in developing biopolymeric coatings for green coffee beans. These coatings provided antioxidant activity, lowered water vapor permeability and increased compressive loading of the beans, thereby protecting them from oxidation, moisture and compression during storage conditions	Ferreira et al. (2021)
31	Enhanced mechanical and antioxidant properties of biodegradable poly (lactic) acid-poly(3-hydroxybutyrate-co-4-hydroxybutyrate) film utilizing α-tocopherol for peach storage	Films	To develop biodegradable and active films that could match petroleum-based films both in antioxidant and mechanical properties	- Poly (lactic) acid (PLA) - Poly(3-hydroxybutyrate-co-4-hydroxybutyrate) (PHB) - α-tocopherol	- Thickness - Tensile strength, Elongation at break - GP - WVP - Contact angle - TGA and DSC - SEM - FTIR - DPPH In peach - Determination of firmness and Total soluble sugar (TSS) - MDA - Measurement of weight loss - relaxation time and magnetic resonance imaging (MRI) detection	The incorporation of α-tocopherol in PLA-based films increased the mechanical properties, WVP and gas permeability compared to the pure PLA film. Some intermolecular gaps were found in the PLA-PHB-α-tocopherol film, with higher gas permeability. The firmness of the peach sample packaged with PLA-PHB-α-tocopherol film effectively delayed the aging of the fruit, the active substances increased the gas permeability and WVP of the film, improved the external gas and moisture exchange, and further maintained the quality of peaches. The PLA-PHB-α-tocopherol film showed the highest DPPH value, inhibited the increase in MDA content and protected the fruit cell wall structure	Jiang et al. (2021)

32	Development of active low-density polyethylene (LDPE) antioxidant packaging films: Controlled release effect of modified mesoporous silicas	Films	To develop active LDPE antioxidant packaging films with modified MCM-41and study the controlled release effects and mechanisms of modified MCM-41 on α-tocopherol in active LDPE packaging films	- LDPE - MCM-41 - α-tocopherol	- Migration tests - FTIR - isotherms of N2 adsorption/desorption of mesoporous materials - TGA - XRD	The main results obtained were that active low-density polyethylene films with enhanced slow-release effect were developed by incorporating modified mesoporous silicas loaded with α-tocopherol, which has potential application in the protection of fatty foods. Furthermore, modification with different organic groups can attribute to different textural properties and active loading capabilities of mesoporous silica compounds. And strong interaction energies between adsorbates and adsorbents caused by organic groups lead to slow release effects of mesoporous silicas	Sun et al. (2021)
33	Effect of α-dl tocopherol acetate (antioxidant) enriched edible coating on the physicochemical, functional properties and shelf life of minimally processed carrots (*Daucus carota* subsp. sativus)	Edible coatings	Evaluate the effect of antioxidant-enriched edible coating on shelf life and shelf life and nutritional quality retention of minimally processed carrots	- Sodium Alginate - Glycerol - Calcium chloride - Tocopherol Acetate	In minimally processed carrots - Weight loss - Total soluble solids (TSS), pH, reducing sugar, total sugar and Ascorbic acid estimation - Color - DPPH and ABTS - Total phenolic content - Carotenoid content and provitamin A activity - Firmness - Microbiological quality	The alginate-based coating supplemented with tocopheryl acetate showed potential application in extending the shelf life of minimally processed carrots during refrigerated storage, maintaining quality, acceptability, and nutritional value of the tested product	Keshari et al. (2022)

WVP- water vapor permeability, GP - Gas pemeability, FTIR - Fourier transform infrared spectroscopy, XRD - X-Ray Diffraction, DSC - Differential Scanning Calorimetry, SEM - Scanning Electron Microscope, TGA - Thermogravimetric analysis, - FESEM -Field emission scanning electron microscopy.

5.1.1 Properties of films and coatings added of tocopherol for food packing

The properties of tocopherol-added films and coatings have been extensively studied in food packaging, as demonstrated by the works of Dias et al. (2018), Hamid et al. (2019a), Hamid et al. (2019b), Jiang et al. (2021), Rosenbloom and Zhao (2020), Yan et al. (2019), Zhang et al. (2020a) which will be further commented in the item 5.3 Possible foods for application of tocopherol films and coatings. A highlight is the application of film or coating with food preservation by the action of the antioxidant in the polymer matrix. We also observed that this action was enhanced when tocopherol was added to various polymers, such as low-density polyethylene (LDPE), where the addition of the antioxidant promoted a synergistic controlled release in the active films (LI et al., 2019), chitosan and pectin in which the films exhibited high antioxidant activity up to 90.60% and high initial release profile followed by extended release for 10 days (HAPSARI et al., 2020), and poly(3-hydroxybutyrate-co-4-hydroxybutyrate) (PLA-PHB) films added of α-tocopherol showed less oxidative deterioration in packaged peaches (JIANG et al., 2021).

In the last five years, in addition to the well-established antioxidant property of tocopherol, other properties have been studied for films and coatings added with tocopherol for use in food packaging, such as thickness, optical properties, microstructure, barrier properties to water and gases, mechanical properties, thermal properties (TGA/DSC), X-ray diffraction (XRD), hydrophobicity, α-tocopherol migration, more details of these properties will be described below.

From this point on, for a better understanding of the subject, it was decided to separate the discussion topic from the articles that dealt with the properties of films and the properties of coatings added of tocopherol for use in food packaging. However, when analyzing Table 1, it can be seen that of the articles that developed coatings, only the work by Ferreira et al. (2021) evaluated the properties of the material developed, while the articles by Shokri and Ehsani (2017) and Keshari et al. (2022) evaluated the properties of the food systems to which the coatings were applied, only Ferreira's work will be discussed together with the articles that determined the properties of the tocopherol films, precisely because in this work a film was also developed to carry out the evaluations, while the articles by Shokri and Ehsani (2017) and Keshari et al. (2022) will be discussed in item 5.2.2 Properties of coatings added of tocopherol for food packing.

5.1.2 Properties of films added of tocopherol for food packing

5.1.2.1 Thickness

Thickness can influence other film properties such as mechanical, barrier properties (MIRZAEI-MOHKAM et al., 2020) and optics. The increase in film thickness attributed to the application of α-tocopherol was reported, mainly in film formulations with increasing concentrations of α-tocopherol (HAMID et al., 2019a; LOPES et al., 2020; MIRZAEI-MOHKAM et al., 2020; ENCALADA et al., 2021). Film thickness can be influenced by high concentrations (PIÑEROS-HERNANDEZ et al., 2017). Therefore, changes in the concentration of the polymer and even the antioxidant added to the film can change this parameter.

5.1.2.2 Optical properties

Regarding the effects of tocopherol on the optical properties, films with a yellowish color were reported (YEAMSUKSAWAT; LIANG, 2019; AGUDELO-CUARTAS et al., 2020; MIRZAEI-MOHKAM et al., 2020), and reddish-yellow (MELLINAS et al., 2020). The color of films and packaging can influence consumer acceptance and the commercial success of the final product, so it is considered an important parameter to be evaluated for packaging with the purpose of application in food (MELLINAS et al., 2020).

In the analyzed works, the increase in the opacity of the films was also widely reported (DARBASI et al., 2017; SUN et al., 2017a; PÉREZ-CÓRDOBA et al., 2018; HAMID et al., 2019a; YEAMSUKSAWAT; LIANG, 2019; AGUDELO-CUARTAS et al., 2020; ZHANG et al., 2020b, 2020a; DIAS et al., 2021; ROSENBLOOM; ZHAO, 2020); however, two articles reported a decrease in this parameter (LOPES et al., 2020; MIRZAEI-MOHKAM et al., 2020). The transparency was attributed to interference in the organization of the matrix, creating irregularities and favoring the transmission of light with an effect on the compaction of the polymeric network, increasing the free spaces.

Opacity is an essential parameter for food packaging films and coatings, as reduced light transmission can promote protection from photosensitive compounds. However, transparent films are also used in food to present the food inside the package better. Thus, the food industry guarantees both transparent and opaque packaging.

5.1.2.3 Barrier properties to water and gases

Barrier properties have also been extensively studied. Some articles have reported increased water vapor permeability (WVP) (DARBASI et al., 2017; MARTELLI et al., 2017; SUN et al., 2017a, 2017b; DIAS et al., 2018; AGUDELO-CUARTAS et al., 2020; LOPES et al., 2020; FERREIRA et al., 2021; JIANG et al., 2021; YEAMSUKSAWAT; LIANG, 2019) others the decrease of this parameter, attributed to the tocopherol applied to the polymer (LI et al., 2019; MELLINAS et al., 2020; MIRZAEI-MOHKAM et al., 2020; ROSENBLOOM; ZHAO, 2020; YAN et al., 2019; ZHANG et al., 2020b).

The WVP is one of the most critical parameters for the characterization of a film because it provides an idea of whether the film will contribute to the neutralization of water loss from the packaged product (SANDOVAL et al., 2019). It depends on the polymer/water interaction (KOCIRA et al., 2021). It is noted that many articles reported increased permeability to water vapor, which ends up being an obstacle to the industrial use of some films or coatings. So it is necessary to overcome this challenge so that this type of packaging is adopted industrially, specifically in the food sector, because vegetables, in general, are necessary an adequate barrier to the passage of water into the package to keep the fruit fresh, where the hydration must be maintained. While for dry foods such as bread and flour, it is necessary to prevent water from entering the film or coating.

Another barrier property evaluated was the permeability to O_2 (SUN et al., 2017a, 2017b) for the low-density polyethylene films with mesoporous silica nanoparticles added tocopherol developed in the two articles. The increase in permeability was attributed to the uneven dispersion of the nanoparticles in the films, while in the article by Jiang et al. (2021) with poly (lactic) acid-poly (3-hydroxybutyrate-co-4-hydroxybutyrate) (PLA-PHB) films, increased oxygen permeability and also increased CO_2 permeability were reported. Films and coatings need to function as a barrier to gases because if the film or coating involves an oxidation-sensitive product, it must remain protected so that it does not suffer the action of oxygen. "Thus, when a polymeric film package has low oxygen permeability coefficients, the oxygen pressure inside the container drops to the point where oxidation is delayed, prolonging the product's shelf life" (SIRACUSA, 2012). Meanwhile, CO_2 permeation must remain within the desired levels inside the package to not harm the food.

5.1.2.4 Microestruture

The inclusion of tocopherol at different concentrations in starch and PBAT (poly (butylene adipate-co-terephthalate)) films altered the microstructure, causing heterogeneity of the polymer matrix regardless of the concentration used (LOPES et al., 2020). The films developed with carboxymethyl cellulose and higher concentrations of polycaprolactone nanocapsules suffered cracks in the structure. However, the films were more uniform, containing 30 and 50% concentrations of nanocapsules (MIRZAEI-MOHKAM et al., 2020). In monolayer and multilayer polyethylene terephthalate (PET)/polypropylene (PP) films, the films impregnated with tocopherol showed discontinuity of the film surface (Franco et al., 2019).

The surface of the gelatin and chitosan films became roughened with the addition of nano-encapsulated active agents (α-tocopherol+cinnamaldehyde+garlic oil) (PÉREZ-CÓRDOBA et al., 2018), while in chitosan films, the surface was rough, with irregular spots, due to incorporation with α-tocopherol (YEAMSUKSAWAT; LIANG, 2019). Chitosan and zein films added with tocopherol showed cracks, heterogeneities, or uniform spots (ZHANG et al., 2020b). However, chitosan films developed with different concentrations of montmorillonite nanocomposites added with 20% tocopherol showed heterogeneous characteristics (DIAS et al., 2021).

In semi-refined carrageenan films with different α-tocopherol concentrations, oil droplets were observed that increased with increasing tocopherol concentration (HAMID et al., 2019a), as well as with hydroxypropylmethylcellulose (HPMC) or soy protein isolate (SPI) films that showed oil droplets attributed to the lipid phase used in the study (ROSENBLOOM; ZHAO, 2020). Several structural behaviors of films and coatings are added with tocopherol. These are closely linked to the technique of preparing the films, the material used, and the amount of each material in forming the polymer. What was possible to perceive in this study was that there were articles that reported a smooth, homogeneous, or compact structure of the films (DE CARVALHO et al., 2019; AGUDELO-CUARTAS et al., 2020; JIANG et al., 2021). The microstructure of a polymer can influence several other properties, such as mechanical, optical, and barrier properties.

5.1.2.5 Mechanical properties

As for the tensile strength (TS), it was reported that after the addition of tocopherol to the polymeric matrix, the increase (DARBASI et al., 2017; LI et al., 2019; SUN et al., 2017b, 2020) attributed to a good distribution of tocopherol in the film, to the fluidity and viscosity of α-tocopherol for being similar to a plasticizer, and in the case of the study by Sun et al. (2020), this increase was attributed not only to the presence of α-tocopherol but the application of mesoporous silica nanoparticles with α-tocopherol in low-density polyethylene films.

As reported in other articles, the reduction of TS (SUN et al., 2017a; PÉREZ-CÓRDOBA et al., 2018; AGUDELO-CUARTAS et al., 2020; HAPSARI et al., 2020; LOPES et al., 2020; MIRZAEI-MOHKAM et al., 2020; ZHANG et al., 2020b; DIAS et al., 2021; JIANG et al., 2021; ROSENBLOOM; ZHAO, 2020), also attributed to the similarity of α-tocopherol with a plasticizer improving the mobility of the polymer chains. However, the microstructure was primarily associated with the behavior of the films for TS; that is, most of the films or coatings added with tocopherol for food presented as less rigid and less resistant than their respective controls.

The elongation at break (EB) of the films increased in the studies of Darbasi et al. (2017), Pérez-Córdoba et al. (2018), Li et al. (2019), Agudelo-Cuartas et al. (2020), Mellinas et al. (2020), Mirzaei-Mohkam et al. (2020), Jiang et al. (2021), and Rosenbloom and Zhao (2020), the addition of α-tocopherol to the films increased the mobility of the polymeric chains. It generates more flexible films, contrary to the studies of Lopes et al. (2020), Martelli et al. (2017), Sun et al. (2017a, 2017b), Yeamsuksawat and Liang (2019) and Zhang et al. (2020b), in which the reduction of EB and less flexibility of the films added with α-tocopherol were demonstrated.

The elasticity modulus (EM) increased, increasing the stiffness of the films (MARTELLI et al., 2017; MIRZAEI-MOHKAM et al., 2020; SUN et al., 2020; YEAMSUKSAWAT; LIANG, 2019; ZHANG et al., 2020b). However, some articles reported a reduction in EM (LOPES et al., 2020; AGUDELO-CUARTAS et al., 2020), and the plasticizing effect of α-tocopherol was considered a determining factor in the modification of the antioxidant/polymer interaction, which increased the polymer mobility and influenced EM, reducing the rigidity of the films.

In the case of food packaging, flexibility is an important factor because if the intention is to manufacture trays, for example, the material must have little flexibility; however, for the manufacture of films, the flexibility must be adequate to levels that can actually be involved the food and seal it, or wrap the food product as a flexible film.

5.1.2.6 Thermal properties (TGA/DSC)

It is essential to know TGA/DSC properties not to overheat or even reach the polymeric melting point when manufacturing the film or coating. In general, the works that evaluated the thermogravimetry of the films (TGA) (PÉREZ-CÓRDOBA et al., 2018; DE CARVALHO et al., 2019; FRANCO et al., 2019; HAMID et al., 2019b; VILLASANTE et al., 2019; LOPES et al., 2020; MELLINAS et al., 2020; SUN et al., 2020, 2021; ENCALADA et al., 2021; FERREIRA et al., 2021; JIANG et al., 2021) do, not observe thermal alterations attributed to the addition of tocoferol. However, Villasante et al. (2019) reported improved thermal stability of poly (α-Dodecyl-Glutamate) (PAAG-12) films added with α-tocopherol and stated that this allowed processability at higher film temperatures.

Ferreira et al. (2021), who produced thermoplastic starch and chitosan films with α-tocopherol/bentonite, observed a strong link between tocopherol and the other ingredients of the formulations that contained tocopherol and attributed it to the changes in the mass loss peaks. At higher temperatures, they observed a peak close to 437°C, which was attributed to a break in the aromatic ring of the α-tocopherol structure, corroborating the article by De Carvalho et al. (2019) who developed poly (vinyl alcohol) (PVA) films added with α-tocopherol nanoparticles, which observed mass loss peaks near 430°C.

On the other hand, studies evaluated differential scanning calorimetry (DSC) (PÉREZ-CÓRDOBA et al., 2018; DE CARVALHO et al., 2019; FRANCO et al., 2019; VILLASANTE et al., 2019; MELLINAS et al., 2020; MIRZAEI-MOHKAM et al., 2020; SUN et al., 2017a, 2020; JIANG et al., 2021; FERREIRA et al., 2021). In general, adding tocopherol to the polymer matrix showed few changes in the melting point. Several articles observed a reduction in the crystallinity of the films as the inclusion or increase in the concentration of tocopherol in the films occurred; however, Jiang et al. (2021) observed an increase in crystallinity, attributed to the interaction between the plasticized poly(lactic) acid-poly(3-hydroxybutyrate-co-4-hydroxybutyrate-(PLA-PHB)) and α-tocopherol interface, corroborating Sun et al. (2020) who justified the increase of this parameter to the surface area of mesoporous silica nanoparticles added with tocopherol.

5.1.2.7 X-Ray Diffraction (XRD)

As for the XRD, standards evaluated by the articles (DIAS et al., 2018; PÉREZ-CÓRDOBA et al., 2018; DE CARVALHO et al., 2019; LOPES et al., 2020; SUN et al., 2017a, 2017b, 2021; ZHANG et al., 2020b; FERREIRA et al., 2021), in the article by De Carvalho et al. (2019) the poly (vinyl) alcohol (PVA) films added with α-tocopherol at different concentration 30, 50 and 70 %, showed better miscibility than the control sample and concentration of 50 % presented more amorphous character. While in the article by Lopes et al. (2020) with starch/poly (butylene adipate-co-terephthalate) (PBAT) films that processing conditions were similar for all formulations concluded that α-tocopherol induces crystallization of amylose complexes, producing semicrystalline materials. Sun et al. (2017a, 2017b) described that the XRD standards for the formulation of the film included mesoporous silica nanoparticles added with α-tocopherol presented a reduction of the peak intensity after the use of the α-tocopherol, indicating the adsorption efficiency of the additive.

According to Pappas (2006), the X-ray diffraction technique or diffraction patterns is based on information from the atomic structures of materials, which can be examined and characterized through the position of atoms, their arrangement in each unit cell, and the spacing between the atomic planes. Knowing this property and the other properties mentioned here can contribute to designing a polymeric film or coating. However, from the analysis of the articles that evaluated this parameter, it is worth mentioning that the incorporation of α-tocopherol can contribute to obtaining more crystalline polymers.

5.1.2.8 Hydrophobicity

When developing a film or coating, it is necessary to evaluate the contact angle to know the hydrophobicity or hydrophilicity of the material. Freitas et al. (2022) stated that a contact angle below 90° denotes a low surface tension, so the lower the wettability, the more hydrophobic the surface. It is noted in the articles that the addition of α-tocopherol to the films or coatings produced polymers with greater hydrophobicity (DE CARVALHO et al., 2019; DIAS et al., 2021) and hydrophilicity (FERREIRA et al., 2021; JIANG et al., 2021). For use in food packaging, the film or coating must be more hydrophobic to function as a barrier to water, as greater humidity can cause deterioration of the food product the incorporation of α-tocopherol precisely to combine a hydrophobic compound with the polymer matrix to increase hydrophobicity. However, the α-tocopherol molecule also has hydroxyl groups, which gives this antioxidant a hydrophilic character, so when applying it to a polymer, the condition of this interaction must be evaluated because, at the time of joining with the other assembly materials of the formulation, it can attribute a hydrophilic character to the material.

5.1.2.9 α-tocopherol migration

According to the Brazilian Health Regulatory Agency (Anvisa), migration is "the transfer of material components in contact with food to these products, due to physical-chemical phenomena." Components used in materials intended to come into contact with food must be included in positive lists, which are lists of "substances that have been proven to be physiologically innocuous in animal tests and whose use is authorized for the manufacture of materials that will come into contact with food" (BRASIL, 2001).

The advent of active packaging has become an indispensable assessment, as the incorporation of agents in films or coatings must be safe. Plastic packaging materials and articles must not transfer their constituents to food simulants in amounts more significant than 10 milligrams of total constituents released per dm^2 of the food contact surface. In addition, this regulation defines the use of α-tocopherol as an additive for the production of polymeric packaging and does not establish restrictions according to European Regulamentation (REGULATION-Nº 10/2011).

With the addition of tocopherol, the intention is precise that it is controlled release into the headspace around the product and generates a protective effect on the food at an adequate migration limit. The vast number of articles that evaluated the stability and migration properties of α-tocopherol added to films or coatings for use in food (MARTELLI et al., 2017; DE CARVALHO et al., 2019; LI et al., 2019; MIRZAEI-MOHKAM et al., 2019; VILLASANTE et al., 2019; YEAMSUKSAWAT; LIANG, 2019; HAPSARI et al., 2020; LOPES et al., 2020; MELLINAS et al., 2020; SUN et al., 2017a, 2017b, 2020, 2021; DRAGO et al., 2021).

Many behaviors have been observed from short-term releases to long-term releases, but what drew much attention were the films in which mesoporous silica nanoparticles were added with α-tocopherol. Compared to films incorporated directly with this active, the controlled release was mainly attributed to the mesoporous silica that prolonged the migration period of α-tocopherol due to its incorporation into the pore channel, making this release difficult (LI et al., 2019; MELLINAS et al., 2020; SUN et al., 2017a, 2017b, 2020, 2021). Furthermore, it can be attributed to the adequate pore size of the mesoporous silica that controlled the release rate (SUN et al., 2017a, 2021).

A controlled release package, added with an active compound, aims to delay spoilage and prolong the shelf life of the food. However, the concentration of the active agent can be released at different controlled levels (VASILE; BAICAN, 2021); this agent retained in the packaging must be properly released to the food product because when it occurs initially, soon after the food product is packaged, it can contribute to inhibiting the oxidation induction period (DE CARVALHO et al., 2019), while if it is released at a slow rate, it may not delay the deterioration of the product (VASILE; BAICAN, 2021). Therefore, there is a need to evaluate this property in a film or coating added with an antioxidant such as tocopherol.

5.1.3 Properties of coatings added of tocopherol for food packing

As mentioned in section 5.2 Properties of tocopherol-added films and coatings for food packaging and observed in Table 1, only the articles by Ferreira et al. (2021), Shokri and Ehsani (2017) and Keshari et al. (2022) developed coatings; in Ferreira's work, only the compressive load borne by coated and uncoated green coffee beans was evaluated, while in Shokri and Keshari's work, the properties of the food systems to which the coatings were applied were evaluated.

Ferreira's results showed greater protection of coffee beans against breakage for beans coated with tocopherol-added coatings, preventing breakage in cases of large-scale storage. For uncoated beans, a force of 375.5N was needed for breakage, while for beans coated with thermoplastic chitosan-based coatings with tocopherol, 496.9N was needed.

In the case of the work of Shokri and Ehsani (2017), whey protein coating was applied to fish fillets conditioned at 4°C for 16 days, and the coatings added of tocopherol showed antioxidant and antimicrobial action in the product. Keshari et al. (2022) applied sodium alginate coating on minimally processed carrots packaged at 10°C for 15 days. They stated that the tocopherol-incorporated film maintained quality and nutritional value and minimized mass loss.

5.2 POSSIBLE FOODS FOR APPLICATION OF TOCOPHEROL FILMS AND COATINGS

According to the results (Table 1), some works that studied the application of films or coatings on food products, as is the case of the work with films: Dias et al. (2018) studied chitosan films on salmon fillet packaged at 4°C for 8 days and reported that product oxidation was minimized by films added of tocopherol. Hamid et al. (2019a) developed semi-refined carrageenan films applied to beef hamburgers conditioned at 4°C for 12 days. They observed that the film with tocopherol retained the pH and delayed the formation of methioglobin and browning of the meat. Also, working with beef burgers conditioned at 4°C for 14 days, Hamid et al. (2019b) applied semi-refined carrageenan films and reported it contributed to the delay of lipid oxidation and browning formation. Yan et al. (2019) applied chitosan nanocapsules with tocopherol in chitosan/montmorillonite films to sliced cured ham conditioned at 4°C for 120 days and observed that the film containing tocopherol was antioxidant.

Rosenbloom and Zhao (2020) applied films of soy protein isolate (SPI) or hydroxypropyl methylcellulose (HPMC) to soybean oil packaged at 35°C for 60 days. They observed that SPI films containing tocopherol minimized product oxidation. Zhang et al. (2020a) applied chitosan or chitosan/zein films to mushrooms packaged at 4°C for 12 days and observed less mass loss, browning, and higher firmness of mushrooms that were packaged with chitosan/zein films added of tocopherol. Jiang et al. (2021) applied Poly (lactic) acid (PLA) and Poly (3-hydroxybutyrate-co-4-hydroxybutyrate) (PHB) films on peaches packaged at 1°C for 30 days and reported that PLA/PHB films incorporated of tocopherol extended product shelf life. In the case of the work with coatings: Shokri and Ehsani (2017) and Keshari et al. (2022) were mentioned in section 5.2.2 Properties of coatings added of tocopherol for food packing.

All the films or coatings applied demonstrated antioxidant action in the products they applied. Demonstrating a promising advantage in the application of films and coatings added of tocopherol for use as food packaging material, it is therefore highly necessary to evaluate the properties of films and coatings because depending on the connection between tocopherol and the polymer matrix, these properties are altered. Figure 4 shows some food systems that can be applied with coatings or films with tocopherol and the various properties that can be evaluated in these materials.

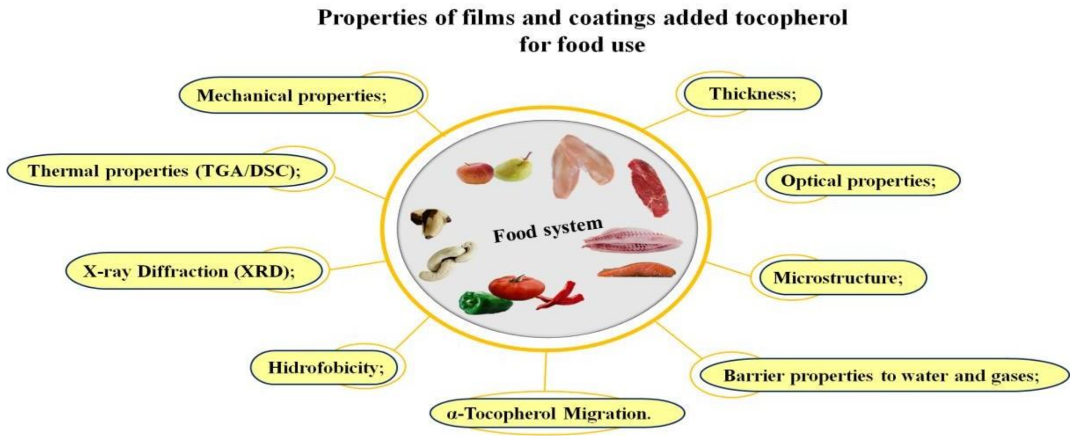

Figure 4. Properties of the films and coatings added of tocopherol for food systems application.

5.3 KEYWORDS CO-OCCURRENCE NETWORK

The data from the searched databases were extracted and analyzed by the VOSviewer tool, which allows the creation, visualization, and exploration of maps based on network data, resulting in different map configurations (VAN ECK; WALTMAN, 2021). Figure 5 shows the visualization maps in Co-occurrence networks of the keywords for the different bases studied.

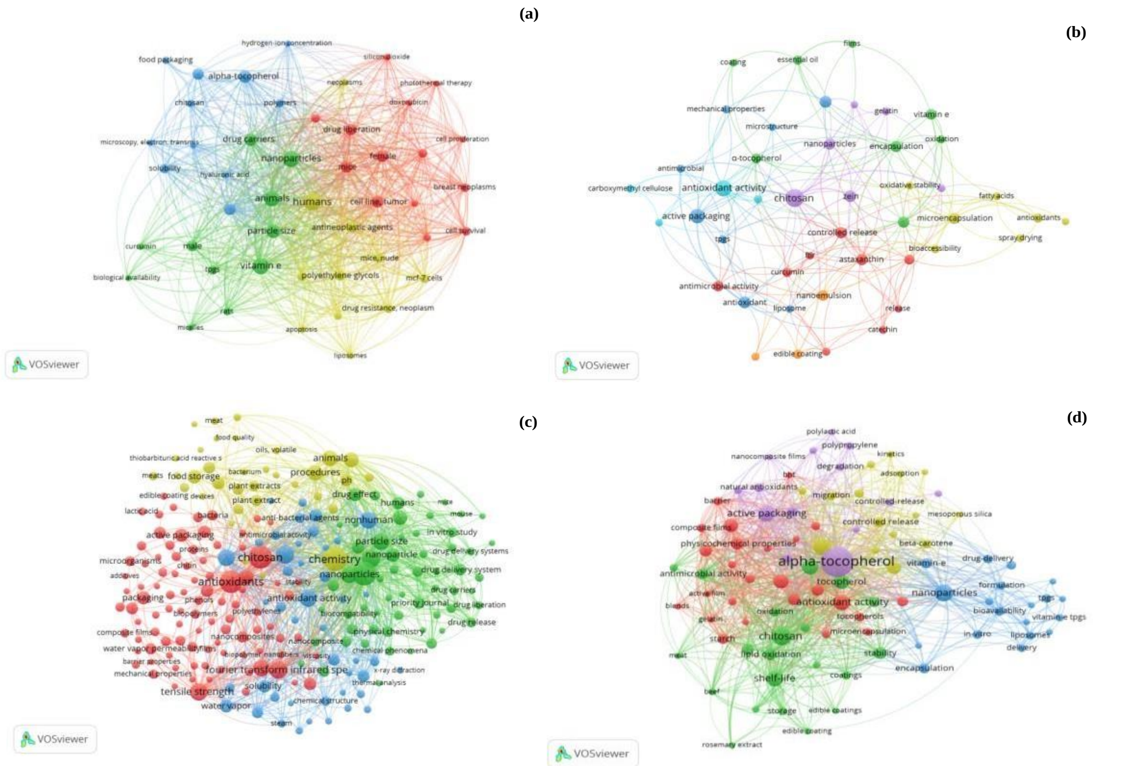

Figure 5. Network view of keyword co-occurrence, (a) PubMed, (b) Science direct, (c) Scopus and (d) Web of Science databases.

Figure 5 (a) presents the results for PubMed, where it is possible to observe 47 keywords, with at least 5 occurrences, forming 4 clusters, with 782 links or Co-occurrence relations between the terms. Figure 5 (b) presents the visualization map for Science Direct, where 45 keywords were obtained, with at least 5 occurrences grouped in 7 clusters. In this database, the term "chitosan" presented a Strength value of 34, evidencing the strength of Co-occurrence links relationship with other terms. For the results in Scopus, Figure 5 (c), 234 keywords can be observed, with at least 20 occurrences, due to the high number of terms in the network, and grouped into 4 clusters, the terms "chitosan," "antioxidants" and "chemistry" set link value of 233, 233 and 232, respectively, showing a co-occurrence connection between the other terms of published research in this database. In the WOS data visualization, for the results in Web of Science Figure 5 (d), we have network formation for 94 keywords with the formation of 5 clusters among them, and the term "alpha- tocopherol" appeared with 88 links, showing the importance of this term within the researches published in WOS. Therefore, when analyzing the network visualization in the different databases, one can notice the main search terms, their weights, and clusters. The different colors and connections of the keywords in each database studied are shown in the graphs.

Figure 6 presents the keyword density visualization; the items are represented by their labels, similar to the network visualization.

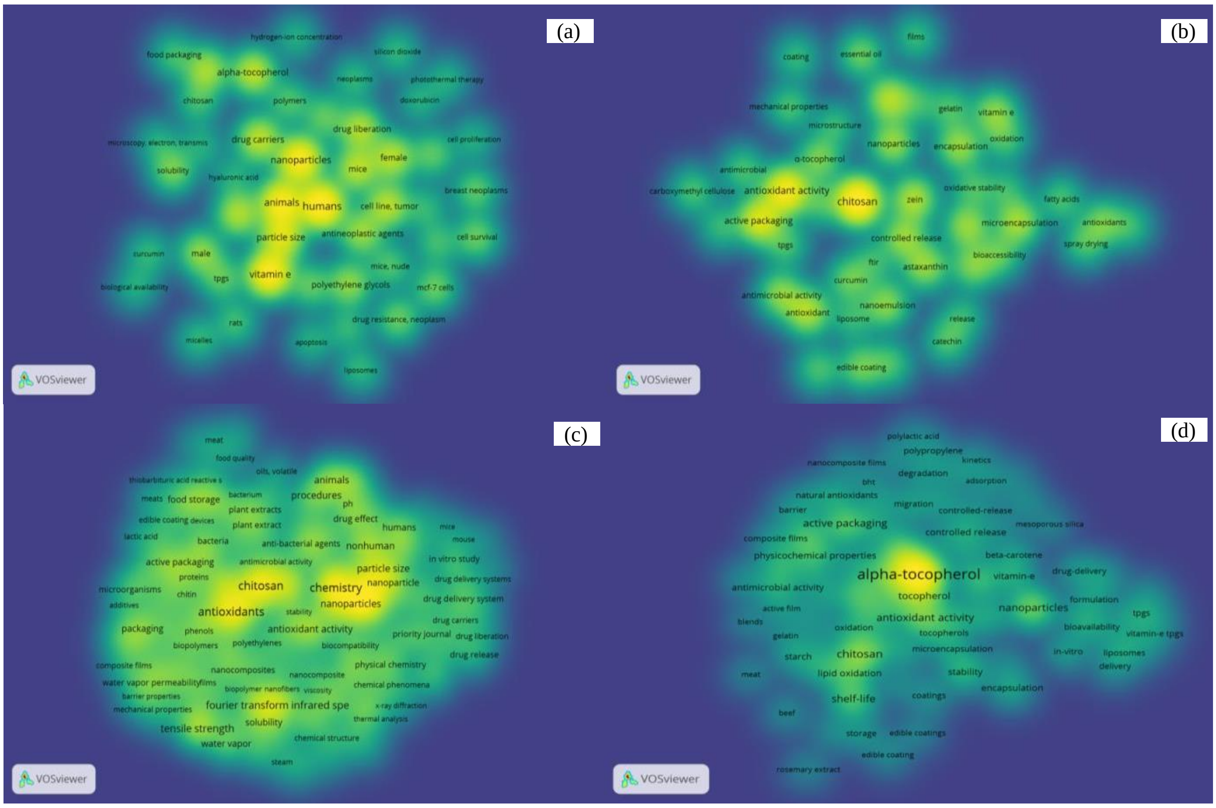

Figure 6. Mapping density visualization of keywords (a) PubMed, (b) Science direct, (c) Scopus and (d) Web of Science databases.

The color indicates the item density, so the greater the number of items in the vicinity of a point and the greater the weights of neighboring items, the closer the point's color is to yellow (VAN ECK; WALTMAN, 2021). Thus, it is possible to observe that the keywords with the highest density in the PubMed database searches, Figure 6 (a), are "nanoparticles," "animals," "humans," "particle size," vitamin E," which is consistent, as the PubMed publishes references and abstracts on life sciences and biomedical topics. For Science Direct, Figure 6 (b), the items with the highest density were "chitosan," "active packaging" and antioxidant activity. For Scopus, the keywords with higher density "chitosan," "antioxidants", "chemistry" and "nanoparticles" and "alpha–tocopherol" and "tocopherol" for WOS. Therefore, the terms with the highest density align with the results presented in the network visualization graph, showing the main keywords and their connections between publications in the period studied for the databases shown in this study.

6 CONCLUSION

In the area of food science and technology, it is sought to develop polymers capable of promoting the extension of the shelf life of the food product, so knowing the properties is vital for this area of research since combining a biodegradable polymeric material with a natural antioxidant active is of great interest to modern society, as they associate environmental preservation with food preservation.

When carrying out this review, it was possible to find 33 articles published in the last five years on films and coatings added of tocopherol for use in food packaging. The main properties have been addressed. Thus, it was possible to observe that the properties, together or separately, can direct the application and the product for which it is intended. This review also made it possible to survey the co-occurrence networks of keywords related to this topic in each investigated database. Data analysis using the VOSviewer tool enabled a better visualization and exploration of these words and the development of maps that showed the primary connections between the publications.

Conducting this review provided the synthesis of knowledge about the properties of these polymers, which can contribute to further research on the desired technological properties of films and coatings added of tocopherol for use in the food industry.

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CAPÍTULO 2

TIPO DE CAPÍTULO: EXPERIMENTAL.

TECHNOLOGICAL PARAMETERS OF CASSAVA STARCH/CARBOXYMETHYL CELLULOSE BLEND-BASED FILMS ADDED OF SOY LECITHIN AND TOCOPHEROL MIX

Manuscrito publicado na *Polymer Testing*

Comprovante de aceite e cópia do artigo (Anexo B)

O artigo pode ser acessado por meio do seguinte link:
https://doi.org/10.1016/j.polymertesting.2023.108245

Resumo do impacto do capítulo para a Tese: The results showed that the films containing soy lecithin (L, LT, and LT2) were more hydrophilic and had a denser structure than film B, but the LT and LT2 films were slightly yellow-green, malleable and showed few bubbles or cracks. In addition, all the films developed are biodegradable. It was also found that the antioxidant remained in the films to which it was added (LT and LT2) and that the LT2 film showed the highest antioxidant activity. It is strongly recommended for application in active food packaging.

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Technological parameters of cassava starch/carboxymethyl cellulose blend-based films added of soy lecithin and tocopherol mix

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ABSTRACT

The aim was to evaluate the technological properties of films based on cassava starch/carboxymethyl cellulose added to soy lecithin and tocopherol mix. The films were prepared by casting from a cassava starch/carboxymethyl cellulose mixture and identified as control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and LT and LT2 (with soy lecithin and tocopherol mix). Thickness, water solubility, water content, film weight, contact angle, permeability test, mechanical tests, antioxidant activity, biodegradability, color, thermogravimetry, and FTIR tests were performed. The results showed that the films containing soy lecithin (L, LT, and LT2) were more hydrophilic and had a denser structure than film B, but the LT and LT2 films were slightly yellow-green, malleable and showed few bubbles or cracks. In addition, all the films developed are biodegradable. It was also found that the antioxidant remained in the films to which it was added (LT and LT2) and that the LT2 film showed the highest antioxidant activity. It is strongly recommended for application in active food packaging.

1. Introduction

The interest in the food packaging industry is increasing regarding environmentally friendly materials [1]. Hydrocolloid-based polymers are a strategy to replace petroleum-based materials. These polymers may have better protection against the passage of oxygen than synthetic plastics due to the polymer network having a complex pathway throughout the polymer [2]. The high oxygen barrier of hydrocolloid-based polymers can be seen in the study by Poulose et al. [3], who studied the oxygen barrier properties of potato fruit juice-based films and observed a ratio of 0.025–0.042 cm^3 per (m^2 day atm).

Starch has become a very promising natural polymer for replacing synthetic polymers being renewable, widely available, and attractively priced. Several botanical sources of starch have been studied for their film-forming properties; some examples are corn, yam, and cassava [4]. One concern with starch-based packaging materials focuses on the hydrophilicity of this polysaccharide, as they cause weakness in the water barrier of polymers [5].

Carboxymethyl cellulose (CMC) is formed in an alkaline medium by the reaction of sodium monochloroacetate with cellulose, one of the most relevant products; it has significant economic importance, as it is widely used as a thickening and emulsifying agent in food products [6]. This polysaccharide has high water solubility and generally provides transparent polymers; water sensitivity is not inconvenient; it also provides good mechanical and barrier characteristics [7,8].

The advantage of mixing polymers seeking to combine attributes can often be a disadvantage, as some assemblies may interfere with polymer recycling [9]. One strategy to enhance the properties and applicability of biopolymers for use in the packaging of food products is the use of lipids in the composition of these polymers because, due to their hydrophobic nature, in conjunction with the polymer matrix, can minimize the hydrophilicity [10].

An example of the application of a lipid in a polymeric base is the study by Costa et al. [11] which aimed to develop and evaluate the properties of a lipid compound, buriti oil, in cassava starch films, seeking to add the bioactivity of buriti oil, which the authors proved

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CAPÍTULO 3

TIPO DE CAPÍTULO: EXPERIMENTAL

CASSAVA STARCH/CARBOXYMETHYL CELLULOSE BLEND BASED EDIBLE COATING ADDED OF TOCOPHEROL MIX: A STRATEGY TO MINIMIZE THE LIPIDIC OXIDATION OF BRAZIL NUTS

Manuscrito integra a TESE e não foi, até o momento (29 de fevereiro de 2024), divulgado em outro meio.

CASSAVA STARCH/CARBOXYMETHYL CELLULOSE BLEND BASED EDIBLE COATING ADDED OF TOCOPHEROL MIX: A STRATEGY TO MINIMIZE THE LIPIDIC OXIDATION OF BRAZIL NUTS

ABSTRACT

The aim was to apply cassava starch/carboxymethyl cellulose blend-based edible coating added tocopherol mixed in Brazil nuts and evaluate oxidative levels during storage. The edible coatings were prepared by casting from a cassava starch/carboxymethyl cellulose mixture and identified as control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and LT and LT2 (with soy lecithin and tocopherol mix). Volumes of the forming solutions of the coatings were reserved for stability, viscosity, pH, and color evaluations. The Brazil nuts were immersed in the solutions for the 30s, placed in an incubator at 25ºC, and evaluated at 1, 7, 15, 30, 45, 60, 90, and 120 days of storage. Tests were conducted for mass loss, the browning index, conjugated dienes and trienes, the oxidative state by official methods, and the accelerated oxidation index. The blend-forming solutions B, L, LT, and LT2 showed non-Newtonian and pseudoplastic behavior, excellent resistance to flow, and stability. The diene, triene, iodine value, peroxide value, p-anisidine value, and total oxidation indices showed that the application of the cassava starch/carboxymethyl cellulose blend-based edible coating added tocopherol mix, LT, and LT2 preserved the Brazil nuts up to 90 days of storage at 25°C. PCA shows that all coated applied to Brazil nuts promoted oil preservation in some evaluation periods, especially those added with a tocopherol mix.

Keywords: Antioxidant coating, Amazonian fruit, Lipid oxidation protection.

1 INTRODUCTION

A coating is a thin layer formed by one or several layers spread over the surface of a substrate that imparts to the substrate esthetic and physical properties derived from the coating material. The conventional process for producing an active coating is based on dissolving or dispersing an active compound in a solvent or matrix that is then applied to the surface of a substrate and dried by evaporation or crosslinking (AZEVEDO et al., 2022).

The active packaging interacts with the packaging environment (i.e., headspace) or directly with the food product. Thus, the active agent can be a scavenger, which absorbs residual oxygen from the headspace, moisture or water, and ethylene resulting from food maturation. Alternatively, the active ingredient can be released/emitted over time in a controlled manner from the package into the headspace or into the food (HAN et al., 2018; YILDIRIM et al., 2017).

Antioxidant agents are used in food packaging to inhibit or slow down oxidation reactions that affect food quality. They react with reactive oxidant species (e.g., peroxides, superoxides, and hydroxyl radicals) to slow down or block oxidation reactions of food products. The active agents are released from the packaging material into the headspace by vaporization or diffuse or migrate into the food (CARRIZO et al., 2016; OUDJEDI et al., 2019).

Tocopherols are used as powerful antioxidants (ZALAK, 2022) applied in polymers in various research areas, for example, in cosmetics (HELENAS et al., 2023), medicine (SONG et al., 2021), as well as in the food sector (de CARVALHO et al., 2019; FERREIRA et al., 2021; HAPSARI et al., 2020). Tocopherol, generally known as vitamin E, is an excellent radical-chain breaker in unsaturated fatty foods and is a lipid-soluble antioxidant that may be derived from food sources such as palm oil, sunflower, and soybeans. Furthermore, tocopherols are employed as antioxidants in food products in four different forms (α, β, γ, and δ), and their effectiveness decreases as follows: $\delta > \gamma > \beta > \alpha$ (BAROUH et al., 2022; MOURE et al., 2001).

The Brazil nut is one of the most important non-timber forest products, and the livelihood of thousands of traditional families depends on its commercialization (RIBEIRO et al., 2014; SILVA et al., 2016). Nuts and seeds are sources of fatty acids, phytosterols, fiber, and polyphenols and are low in sodium (ZEC; GLIBETIC, 2018). The Brazil nut is composed of proteins (16.03 g 100 g^{-1}), lipids (58.52 g 100 g^{-1}), carbohydrates (19.61 g 100 g^{-1}), ash (3.35 g 100 g^{-1}) (VASQUEZ-ROJAS et al., 2021), besides 5.24 µg g^{-1} of Selenium (BOTELHO et al., 2019). According to the Instituto Brasileiro de Geografia e Estatística - IBGE, the production of Brazil nuts in 2021/2022 was 38,160 tons, predominantly in the North region, whose production was 35,964 tons, and the state of Para was responsible for the production of 8,807 tons in this period (IBGE, 2024).

The high concentration of unsaturated fatty acids in the Brazil nut, it becomes quite perishable, especially when exposed to trading for an extended period, because it is subjected to high temperature and relative humidity conditions, being exposed to oxidative processes that can contribute to the loss of nutrients and occurrence of rancid odor and flavor in Brazil nuts (RIBEIRO et al., 1993; SILVA et al., 2010).

Some studies have evaluated the effect of storage conditions on oxidative changes in Brazil nuts, for example the studies by (RIBEIRO et al., 1993), who packed Brazil nuts in kraft paper bags and stored them at temperatures of -15, 2 and ambient - ranging from 18 to 25°C, and Casagrande et al. (2019) have stored Brazil nuts in transparent and opaque glass and plastic containers, at temperatures of 11 and 24°C.

In the study by Leme et al. (2023), developed thermoplastic starch/poly(butylene adipate-co-terephthalate) (TPS/PBAT) packaging containing curcumin and water-soluble pine nuts, Brazil nuts were packaged and packed in airtight jars stored at 10, 25 and 50ºC, lipid oxidation was determined at 0, 5, 10, 15 and 30 days of storage. However, no studies have been found on monitoring the lipid oxidation profile of Brazil nuts packed with an edible coating based on cassava starch/carboxymethylcellulose incorporated with tocopherols and stored for different lengths of time.

Therefore, developing an active edible coating that contains antioxidants may be a protective strategy for Brazil nuts and minimize lipid oxidation levels. This work aimed to apply a cassava starch/carboxymethyl cellulose blend-based edible coating with a tocopherol mix to Brazil nuts and evaluate oxidative levels during storage.

2 METHODOLOGY

The Brazil nuts (kindly provided by the Mutran export company, Belém, Pará, Brazil) were registered at Sisgen - National System for Managing Genetic Heritage and Associated Traditional Knowledge, nº A9659A8/2023. The Brazil nuts, according to information provided by the exporting Mutran Company, were from the 2022 harvest, collected from January to June, and had been dried at 50 °C for 8 hours and dehydrated at 70 °C for 18 to 20 hours until the donation, which occurred in December 2022. The Brazil nuts went through biometry and weighing for classification, according to criteria of Brasil (1976), were classified as peeled or processed nuts, small, measuring on average 27.10 cm in height, 12.85 cm wide, and 10.96 cm thick and weighing 2.07 g.

The material used to formulate the solutions for the formation of the coatings, all of which were food-grade: carboxymethyl cellulose (lot 25249 Arcolor, São Paulo, Brazil), cassava starch (lot HW294, Amafil, Cianorte, PR, Brazil), sorbitol in solid form (powder), (lot 250FU46, Ingredientes on-line, São Paulo, Brazil), Soy lecithin (lot 2021105, Ingredientes on-line, São Paulo, Brazil), and tocopherol mix (mix of tocopherols - α-tocopherol: 10-20 %; β -tocopherol: 1-3 %; γ-tocopherol: 45-65 %; δ-tocopherol: 12-26 %) (Vonplex E® L., São Paulo, Brazil).

2.1. FORMULATIONS AND PREPARATION OF THE BLEND-FORMING SOLUTIONS

The blend-forming solutions were prepared according to a methodology adapted from Tongdeesoontorn et al. (2011) e Wu et al. (2001). The formulations were defined using laboratory pre-tests. Four blends were prepared: B, L, LT, and LT2. Fixed amounts of cassava starch + carboxymethyl cellulose + sorbitol and water were used (3 g + 1.5 g + 0.2 g + 100 g, respectively), and the soy lecithin was added in the proportion of 20 % to tocopherol mix in the formulation L, LT, and LT2, the formulation L was prepared with the same amount of lecithin as LT2. The amount of tocopherol mix was added to the blends in the following proportions: B - 0, L - 0, LT - 0.0005 g, and LT2 - 0.010 g. The solution of cassava starch, sorbitol, and 70% of the total volume of water was stirred (Quimis Q261-22, Diadema, SP, Brazil), raising the temperature to 70 ºC, maintained for 10 minutes to induce the gelatinization of the starch, to form a paste. In another container, the carboxymethyl cellulose, the tocopherol mix, soy lecithin, and 30% of the total volume of water were mixed at 45 ºC, and the starch paste was added to this mixture under constant agitation until complete homogenization to form a blend. The blends were centrifuged in a centrifuge (Quimis Q222, Diadema-SP, Brazil) for 5 (five minutes) at 1000 G to remove air bubbles and stored until the moment of application, reserving part of the solution for the viscosity tests.

2.2 TESTS OF THE SOLUTIONS FORMATING EDIBLE COATINGS

2.2.1 Rheology

The rheological parameters of the solutions B, L, LT, and LT2 were obtained at the temperature of 45 ° C using a rheometer (Physica, MCR 101, Ostfildern, Germany), with a deformation rate of 1 to 500 s-1 in cone-plate geometry. The data were fitted to Newton's, Power Law and Herschel-Bulkley rheological models using Equations 1, 2, and 3, respectively. The coefficient of determination (R^2), the reduced chi-squared value (χ^2), and the root mean square error (RMSE) were the parameters used to evaluate the fits.

$$\sigma = \dot{y}.\mathrm{n} \tag{1}$$

$$\sigma = \mathrm{k}.\dot{y}^{\mathrm{n}} \tag{2}$$

$$\sigma = \sigma 0 + \mathrm{k}.\dot{y}^{\mathrm{n}} \tag{3}$$

Where: ζ - shear stress (Pa); $\dot{y}$ - shear rate (s); k - consistency index ($Pa.s^n$); n - fluid behavior index (dimensionless); ζ0- residual stress (Pa).

2.2.2 Centrifugation

In a centrifuge (Spinlab Scientific SL 5-GR, China), test tubes containing 2 g of each solution B, L, LT, and LT2 were submitted to 30 min cycles in rotation of 178, 1113, and 2183 G (ANSEL; POPOVICH; ALLEN, 1999). The volumes of the supernatant were quantified at each cycle.

2.2.3 Creaming index

Immediately after preparation, 50 mL of each of the B, L, LT, and LT2 solutions were transferred to closed 50 mL graduated beakers and kept at a controlled temperature of 25°C. If no sample showed phase separation in the first few hours, the volume of the aqueous phase was quantified every 24 hours until the seventh day of testing (COUTO, 2014). The stability was measured through the height of the upper phase, the creaming index described by Equation 4.

$$CI\ (\%) = (H/H_0) * 100 \qquad (4)$$

In which: H0 - initial height of the lower phase and H - initial height of the upper phase after 24 hours.

2.2.4 pH

The pH of solutions B, L, LT, and LT2 was measured using a bench pH meter (Luca 210P, Campinas-SP, Brazil) using method n°. 981.12 (AOAC, 1997).

2.2.5 Color parameters

The instrumental color parameters of solutions of the edible coating B, L, LT, and LT2 were measured using a colorimeter (Konica Minolta Sensing IC., CR-400, Sakai, Japan). The parameters L* lightness, a* chromaticity, b* chromaticity, C*- chroma, and Hue (h°) parameters were evaluated.

2.3 EVALUATION OF THE OXIDATION LEVEL OF BRAZIL NUTS

To evaluate the oxidation level of the Brazil nuts, the nuts were immersed for 30 s in the blend-forming solutions B, L, LT, and LT2, deposited in non-stick trays, and dried at 45 ºC in the oven (Lucadema Científica, 80/336, São José do Rio Preto/SP, Brazil) for 19 h. After drying, the nuts coated and uncoated were conditioned in a bacteriological incubator (347 CD, Fanem, Brazil).

To monitor the lipidic oxidation profile of the Brazil nuts, coated or not, in the periods 1, 7, 15, 30, 45, 60, 90, and 120 days of conditioning at 25±0.5°C, it was determined the mass loss, the browning index, conjugated dienes and trienes, the oxidative state by official methods and the accelerated oxidation index. For the analyses, the oil was extracted from the Brazil nuts by cold pressing using an ERT 60III press (N.S. 597, Vinhedo-SP, Brazil).

2.3.1 Loss of fresh mass

The mass loss of the Brazil nuts was carried out by gravimetry, the mass loss of the Brazil nuts expressed in % (REZAIYAN ATTAR et al., 2023).

2.3.2 Index of browning

The evaluation of the index of browning (IE) of the Brazil nuts uncoated and coated was according to Palou et al. (1999) and Equations 5 and 6.

$$\mathrm{IE} = [100(\mathrm{x} - 0{,}31)/0{,}172] \tag{5}$$

$$x = (a + 1{,}75 * L)/(5{,}645 * L + a - 3{,}012 * b) \tag{6}$$

In which: L= luminosity and the chromaticities a (-a: green and +a: red) and b (-b: blue a +b: yellow).

2.3.3 Determination of conjugated dienes and trienes

The determination was by UV light absorptivity at wavelengths 232 and 270 nm (conjugated dienes and trienes, respectively) according to method Ch 5-91 (AOCS, 2009). The calculations were performed using Equation 7.

$$E_{1cm}^{1\%} = (A/C) \quad (7)$$

In which: A - absorbance at λ of 232 nm for the conjugated dienes and λ of 270 nm for the conjugated trienes; C- concentration of the solution (g. 100 mL^{-1}).

2.3.4 Evaluation of oxidative status by official methods

The lipid oxidation degree of the Brazil nuts was evaluated using the free fatty acid (FFA - AOCS Ca 5a-40) and iodine value (IV - AOCS Cd 1-25) (AOCS, 2009). The formation of primary and secondary products (carbonyl products) of lipid oxidation was evaluated. The primary products (LOOH) were monitored using the peroxide value (PV - AOCS Cd 8b-90) (AOCS, 2009). The secondary products were determined using the p-anisidine value (p-AnV - Cd 18-90) (AOCS, 2004) which was determined spectrophotometrically at 350 nm. Based on p-AnV and PV, the overall oxidation rates of coated and uncoated Brazil nuts during storage were calculated as total oxidation (TOTOX = 2PV + p-AnV) (SYMONIUK et al., 2022).

2.3.5 Accelerated oxidation – Induction time

The accelerated oxidation test of Brazil nuts was by the AOCS Cd 12b-92 method (AOCS, 1997) using a Rancimat 743 equipment (Metrohm Schweiz AG, Zofigen, Switzerland). The test was carried out using 3.0 ± 0.1 g of the oil sample with an airflow of 15 L h^{-1} at 110 ºC. The induction time (IT) was expressed in hours.

2.3.5.1 Oxidation kinetics of Brazil nuts

The zero, first, and second-order kinetic constants for the oxidation of Brazil nut oil were calculated using the kinetic models shown in Table 1.

Table 1. Zero, first-order, and second-order kinetic models.

Reaction	Model	Equation number
Zero	$A = A_0 - k\theta$	(8)
First	$lnA = ln\ A_0 - k\theta$	(9)
Second	$1/A = k\theta + 1/A_0$	(10)

A - Concentration of the evaluated parameter after a time θ; A0- initial concentration of the evaluated parameter; k - reaction speed constant; θ - time.

For the adjustment of the mathematical models, non-linear regression analysis was performed, by the Gauss Newton method, using the Solver computer software for Microsoft Excel 2016. The performance of the model was evaluated using statistical parameters such as chi-square (χ^2), estimated mean error (SE), relative mean error (P), and coefficient of determination (R^2).

The half-life time (θ(1/2)) was calculated using Equation 11 for the zero order kinetic model, with Equation 12 for the first order kinetic model and with Equation 13 for the second order kinetics model. The value of k used was that of the kinetic model that best fits the experimental data of the kinetics.

$$\theta_{(1/2)} = A_0/2_k \quad (11)$$

$$\theta_{(1/2)} = ln2/k \quad (12)$$

$$\theta_{(1/2)} = 1/kA_0 \quad (13)$$

2.4 STATISTICAL TREATMENT OF DATA

All the tests were carried out in triplicate. For a better understanding, the statistical treatment of the data will be listed:

- The rheological data of the solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) were adjusted to Newton's, Power Law and Herschel-Bulkley models to evaluate the behavior of the emulsions and graphs was be generated using Oring Pro 9.6.5.169 software. The Levene test checked the homoscedasticity of the fit parameters, and the normality of the data was evaluated using the Shapiro-Wilk test; for the data was considered normal, the Tukey test was performed ($p \leq 0.05$) for variance analysis and comparison of means of the parameters using the statistical program Jamovi 2.3.26 (JAMOVI, 2022).

- The data from the centrifugation data, pH and color parameters of the solutions, and the results of the tests to evaluate the lipid oxidation of the Brazil nuts were checked for homoscedasticity using the Levene test, the normality of the data was evaluated using the Shapiro-Wilk test, for the data were considered normal, the Tukey test ($p \leq 0.05$) was performed for analysis of variance and comparison of the means of the parameters using the Jamovi 2.3.26 statistical program (JAMOVI, 2022).
- The Cremeation results were submitted to linear regression using Microsoft's Excel 2010® spreadsheet. The Rancimat results were adjusted to the mathematical models for evaluating oxidation kinetics, also using Excel 2010®.
- The physicochemical parameters and the induction time obtained in the Rancimat test to assess the level of oxidation of Brazil nuts with and without coating were subjected to a Principal Components Analysis (PCA), and a biplot was generated to explain the variability of the data, to analyze the similarity of the results obtained a Hierarchical Cluster Analysis (HCA) was carried out, a cluster dendogram graph was generated using the software Past4.03 (HAMMER; HARPER; RYAN, 2001).

3 RESULTS AND DISCUSSION

3.1 RHEOLOGICAL BEHAVIOR OF BLEND-FORMING SOLUTIONS

Flow curves were drawn to assess the rheological behavior of the blend-forming solutions. The data generated by the test makes it possible to test rheological models that make it possible to visualize information such as the consistency index, for example, which indicates the degree of flow, i.e., how much the fluid flows, which can reflect industrially how much the tested fluid resists flow if subjected to a pipe. In addition, assessing the viscosity of fluids allows them to be classified as Newtonian (constant viscosity) and non-Newtonian (viscosity varies with the magnitude of the shear rate). Figure 1 shows the flow curves of blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) at 45 ºC - shear stress vs. shear rate.

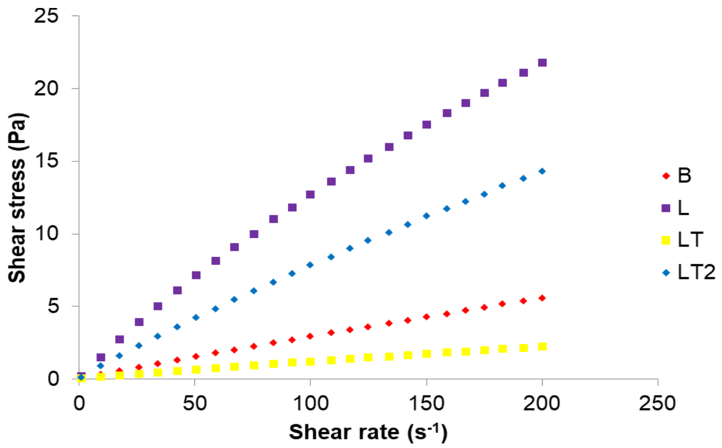


Figure 1. Flow curves of blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) at 45 ºC – shear stress vs. shear rate.

Figure 1 shows that the blend-forming solutions B, L, LT, and LT2 showed an increase in shear stress continuously according to the increase in the strain rate in all solutions; this behavior is characteristic of non-Newtonian fluids. The increase in the strain rate may have caused a collapse in the solutions' structure, leading to a more aligned arrangement of the molecules (ALPASLAN; HAYTA, 2002).

Figure 2 shows the flow curves of blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) at 45 ºC – apparent viscosity vs. shear rate.

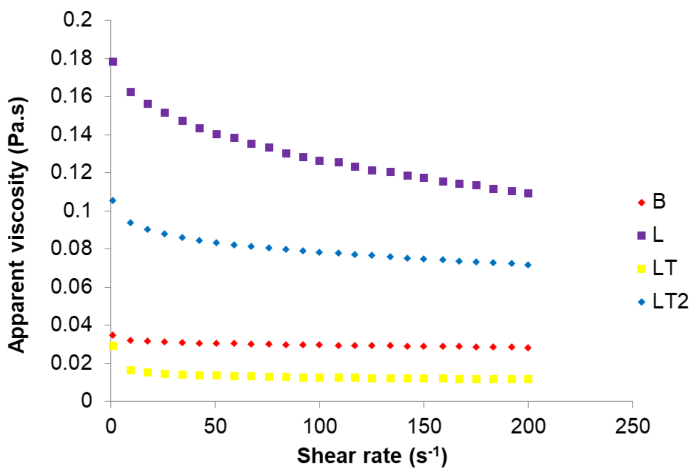


Figure 2. Flow curves of blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) at 45 ºC - apparent viscosity vs. shear rate.

In Figure 2, it is observed that in the blend-forming solutions B, L, LT, and LT2, the results showed apparent viscosity decreases with an increase in shear rate. This may have occurred because the shear rate's action disrupted the fluids' intermolecular junctions, preventing reformation and decreasing the viscosity (EL MIRI et al., 2015; ZHANG et al., 2020).

The blends L and LT2, with a higher concentration of lecithin in its formulation, showed higher viscosity; it is inferred that soy lecithin caused greater resistance to the flow of these fluids, the concentration of the emulsifier did not favour the flow in the blend LT2 in addition to the soy lecithin content also had the highest concentration of tocopherol mix forming intermolecular hydrogen bonds, causing the reinforcement in the intermolecular network, making the blend more viscous (WU et al., 2016; ZHANG et al., 2020). Various properties of film-forming solutions can be affected by fluid flow behavior, such as thickness, design sizing, application form, spreadability, and mechanical properties (CHEN et al., 2009; ZHANG et al., 2020).

The behavior of the flow curves can be attributed to the excellent interaction between the compounds in the solution forming the coatings, such as cassava starch + carboxymethyl cellulose + sorbitol + soy lecithin + tocopherol mix + water. In addition, the LT blend-forming solution had the lowest values throughout the test, so it can be said that at the minimum concentration of the tocopherol mix, the blend-forming solution had the lowest resistance to flow.

Table 2 shows the results of the fit parameters of the Newton, Power Law, and Herschel-Bulkley models to the rheological behavior of the blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Table 2. Mean±standard deviation of the fit parameters of the Newton, Power Law, and Herschel-Bulkley models to the rheological behavior of the blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Blend-forming solutions of the coatings	**Fit Parameters - Newton model**			
	n	R^2	χ^2	RMSE
B	0.028±0.0001c	0.998±0.0001a	0.004±0.0001c	0.310±0.004c
L	0.116±0.0004a	0.979±0.0004c	0.811±0.01a	4.461±0.10a
LT	0.011±0.0001d	0.992±0.0004b	0.003±0.00001c	0.273±0.01c
LT2	0.074±0.0001±b	0.993±0.00001b	0.117±0.001b	1.674±0.005b
p-Value homoscedasticity	<0.001	0.005	0.038	0.008
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001

Blend-forming solutions of the coatings	**Fit Parameters - Power Law model**				
	K ($mPa.s^n$)	n	R^2	χ^2	RMSE
B	0.151±0.19ab	0.945±0.00001a	0.999±0.00001a	0.012±0.00001c	0.052±0.00006c
L	0.282±0.002a	0.816±0.009c	0.999±0.00001a	0.024±0.00001b	0.766±0.002a
LT	0.021±0.00001b	0.883±0.00001b	0.999±0.00001a	0.134±0.001a	0.017±0.00002d
LT2	0.128±0.00001ab	0.890±0.001b	0.999±0.00001a	0.002±0.00001d	0.247±0.008b
p-Value homoscedasticity	<0.001	0.001	0.001	0.005	0.018
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001	<0.001

Blend-forming solutions of the coatings	**Fit Parameters - Herschel-Bulkley model**					
	ζ (Pa)	K ($mPa.s^n$)	n	R^2	χ^2	RMSE
B	0.00±0.008a	0.037±0.00001c	0.945±0.00001a	0.999±0.00001a	1.20±0.002c	0.002±0.001d
L	0.00±0.14a	0.285±0.003a	0.816±0.00001c	0.999±0.00001a	0.027±0.004a	0.58.004b
LT	0.00±0.003a	0.020±3.43d	0.878±0.00001b	0.999±0.001a	1.78±0.001b	3.07±0.016a
LT2	0.001±0.004a	0.125±0.00001b	0.890±0.00001b	0.999±0.00001a	0.003±0.00001d	0.078±0.001c
p-Value homoscedasticity	<0.001	<0.001	0.003	<0.001	0.026	0.012
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5% level. K = consistency index, n = fluid behavior index, R^2 = correlation coefficient, χ^2 = chi-squared, RMSE = root mean squared error, ζ (Pa) = residual stress.

The Power Law model generally showed a better fit for the rheological behavior of all blend-forming solutions. When using the Power Law model to describe the structural behavior of samples, a high correlation coefficient ($R^2 > 0.9999$) was obtained (Table 2). The n values of all samples were lower than 1, meaning that they were pseudoplastic fluids. However, all blend-forming solutions showed an n value close to 1, indicating closer to Newtonian fluids and a lower influence of shear rate on viscosity. Thus, the solution was more stable (LIN; KU, 2008; ZHANG et al., 2020).

Another essential factor to mention was the behavior index, which explains the results of the flow curves since the LT blend-forming solution had a lower K value than the other coating-forming blend solutions. It can be inferred that the concentration of the tocopherol mix added to this blend made it less resistant to flow due to the good interaction between the formation compounds in the blend-forming solution, especially at the lowest concentration of the tocopherol mix.

3.2 CENTRIFUGATION AND CREAMING INDEX

The centrifugation test imposes stress on the sample to simulate increased gravitational force, anticipating signs of sample instability, such as phase separation (BRASIL, 2008). The creaming index is important for checking the destabilization of emulsions, as phase separation can occur due to the difference in densities (SCHUSTER et al., 2012).

No phase separation occurred in the centrifugation test or the creaming test, and data was not shown. This behavior can be attributed to the good interaction of the ingredients, which promoted the stability of all the solutions. This is corroborated by the rheological behavior shown in the Power Law model (Table 2) since it showed behavior index values close to 1, demonstrating the high stability of all the blend-forming solutions.

3.3 pH AND COLOR PARAMETERS

Table 3 shows the pH and color parameters of the blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Table 3. Mean±standard deviation of the color parameters and pH of the blend-forming solutions B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Blend-forming solutions of the coatings	pH	Luminosity (L*)	Chromaticity (a*)	Chromaticity (b*)	Chroma (C*)	Hue (h°)
B	6.05±0.03c	12.93±0.58c	-1.34±0.01a	7.36±0.01ab	7.29±0.04ab	100.04±0.08c
L	6.22±0.005b	13.08±0.03c	-1.35±0.01a	7.65±0.16a	7.53±0.31a	99.32±0.41d
LT	6.22±0.005b	15.08±0.02b	-1.44±0.01b	6.72±0.03bc	6.88±0.03bc	102.37±0.005b
LT2	6.53±0.008a	16.61±0.07a	-1.63±0.02c	6.46±0.04c	6.66±0.03c	103.90±0.02a
p-Value homoscedasticity	0.045	0.002	0.424	0.009	0.008	0.005
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5% level.

Table 3 shows that the blend-forming solutions L and LT were similar according to Tukey's test and significantly different according to Tukey's test ($p \leq 0.05$) compared to B and LT2. The pH results showed that L, LT, and LT2 were superior to formulation B, demonstrating the influence of soy lecithin in these formulations. Meanwhile, the LT2 solution had the highest pH value, i.e., the higher concentration of the tocopherol mix caused an increase in the pH value. It can be said that this result did not adversely affect the polymer matrix or the stability of the solutions because, as already mentioned, all the solutions showed high stability in the creaming and centrifugation tests, demonstrating the good interaction of the cassava starch/carboxymethyl cellulose combination with sorbitol and water. Bai et al. (2017) reported that polysaccharides are more stable than proteins due to variations in pH, ionic strength, and temperature increase. So, the blend-forming solutions did not suffer so much change due to the pH results, as they all presented high stability without phase separation, as mentioned above.

Another important factor regarding the pH of an edible coating-forming solution is that the human body has extreme physiological pH variations throughout the gastrointestinal tract, ranging from pH 2 in the stomach to pH 7 in the colon and up to pH 8.2 in the lower duodenum (REYES-ORTEGA, 2014). So, the pH values found, which ranged from 6.05 to 6.53 when entering the gastrointestinal tract, could be digested without gastric damage by humans.

The color parameters (Table 3) showed that the luminosity parameter of all samples tended slightly toward white. The chroma values were low (achromatic), revealing weak or diluted coloration; it is clear when observing the chromaticity parameters a* and b*. The hue angle reveals that all the solutions had a slightly greenish-yellow tonality. Adding a tocopherol mix promoted a greater tonality in the blend-forming solutions LT and LT2.

3.4 EVALUATION OF THE OXIDATION LEVEL OF BRAZIL NUTS

3.4.1 Loss of fresh mass of the Brazil nuts uncoated and coated during storage time

The loss of fresh mass for nuts is a limiting factor for their preservation and marketing, as the nuts have a dry/shriveled appearance and consequently losses that can be in nutritional aspects, on the oxidative level of the nut. Figure 3 shows the loss of fresh mass of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

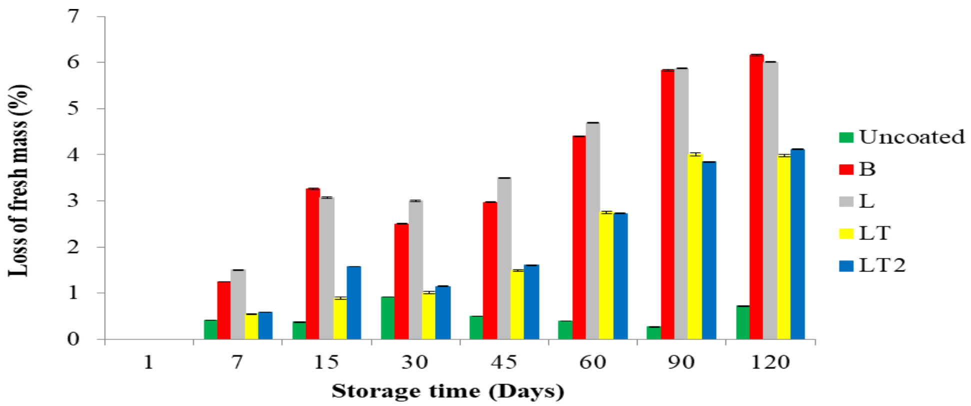


Figure 3. Loss of fresh mass of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

The loss of fresh mass of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) (Figure 3) showed that the uncoated Brazil nuts had the lowest weight loss when compared to the coated Brazil nuts, but even if this had happened, it was observed that the mass losses of the Brazil nuts coated with LT and LT2 (with soy lecithin and tocopherol mix) minimized the losses suffered by control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix). The loss of fresh mass presented by coated Brazil nuts is not a disadvantage because when a food product is exposed to a humid environment, it can become more susceptible to microbiological degradation.

3.4.2 Index of browning of the Brazil nuts uncoated and coated during storage time

Lipid oxidation generates volatile compounds that cause rancidity, while the Maillard reaction can alter the color of dried foods and decrease their nutritional value. These reactions are interrelated (ZAMORA; HIDALGO, 2011). Figure 4 shows the browning index of Brazil nuts uncoated and coated with the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

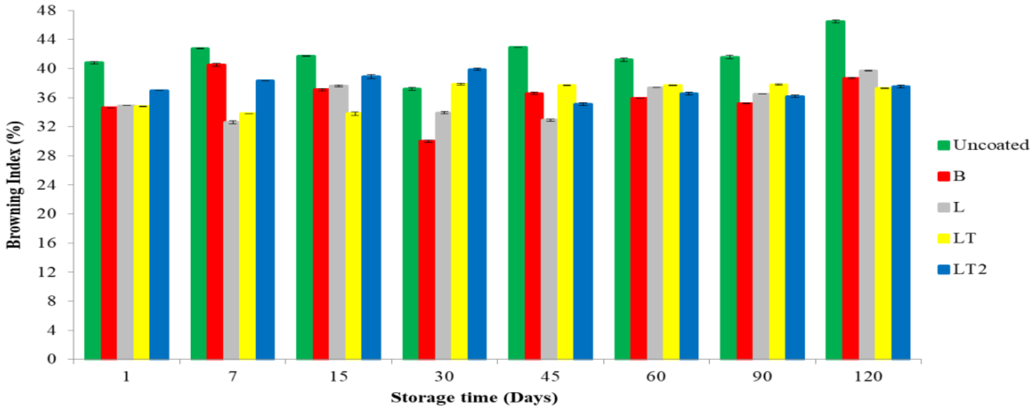


Figure 4. Browning index of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

The browning index of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) (Figure 4) showed that all the coated nuts showed a degree of preservation of the browning level on some of the storage days. It can also be seen that the nuts coated with LT showed more stable values after 30 days of storage. This result shows the excellent interaction of the cassava starch/carboxymethyl cellulose combined with sorbitol and water.

3.4.3 Physical and chemical parameters of oil of the Brazil nuts uncoated and coated during storage time

Table 4 shows the results of the quality parameters and induction time of the oil of the Brazil nuts uncoated and coated with the coatings of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

Table 4. Mean±standard deviation of the quality parameters and induction time of the oil of the Brazil nuts uncoated and coated with the coatings of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

Parameters	Brazil nuts coated and uncoated	STORAGE TIME (DAYS)							
		1	7	15	30	45	60	90	120
K232	Uncoated	0.12±0.005aE	2.16±0.03aD	2.45±0.03aC	2.67±0.003B	2.67±0.02aB	2.68±0.02aB	2.69±0.0001aB	4.46±0.0001aA
	B	0.12±0.005aB	0.12±0.006bB	0.11±0.01bB	0.12±0.0001BE	0.14±0.0001cA	0.12±0.005cB	0.12±0.0001eB	0.11±0.005dB
	L	0.12±0.0001aF	0.12±0.005bEF	0.10±0.0001bG	0.13±0.005	0.23±0.0001bB	0.18±0.0001bC	0.25±0.005bA	0.15±0.0001bD
	LT	0.12±0.0001aB	0.12±0.0001bB	0.10±0.005bC	0.12±0.01B	0.12±0.005cB	0.12±0.006cB	0.18±0.0001cA	0.12±0.0001cdB
	LT2	0.12±0.0001aB	0.12±0.0001bB	0.10±0.0001bD	0.13±0.0001A	0.13±0.005cA	0.13±0.0001cA	0.13±0.005dA	0.11±0.006dC
p-Value homoscedasticity		< 0.001	0.024	0.004	0.001	0.004	0.004	< 0.001	< 0.001
p-Value ANOVA/Welch		< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
K268	Uncoated	0.12±0.0001aG	0.29±0.0001aD	0.24±0.0001aF	0.25±0.005aE	0.28±0.01aD	0.35±0.0001aC	0.37±0.002aB	0.63±0.0001aA
	B	0.12±0.005aA	0.12±0.0001bA	0.11±0.0001bcB	0.12±0.0001bA	0.12±0.0001cA	0.11±0.0002eB	0.12±0.0001dA	0.12±0.005dA
	L	0.12±0.006aD	0.12±0.005bD	0.12±0.005bD	0.13±0.005bC	0.23±0.005bB	0.23±0.0001bB	0.25±0.005bA	0.25±0.0001bA
	LT	0.12±0.0001aA	0.12±0.0001bA	0.12±0.001bA	0.12±0.005bA	0.12±0.003cA	0.12±0.0001dA	0.12±0.0001dA	0.12±0.0001dA
	LT2	0.12±0.005aB	0.11±0.005bcC	0.12±0.001bB	0.12±0.005bB	0.12±0.0001cB	0.13±0.0001cA	0.13±0.0001cA	0.13±0.006cA
p-Value homoscedasticity		0.004	< 0.001	< 0.001	0.034	0.056	< 0.001	< 0.001	< 0.001
p-Value ANOVA/Welch		< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
FFA (mg KOH.g^{-1})	Uncoated	1.63±0.00001aA	1.63±0.00001cA	1.46±0.02dDE	1.51±0.02bC	1.56±0.01bB	1.50±0.03aCD	1.32±0.01dF	1.44±0.01cE
	B	1.63±0.001aB	3.38±0.01aA	3.17±0.02bA	3.55±0.8aA	3.71±0.8aA	1.51±0.06aB	1.48±0.01cdB	1.55±0.01cB
	L	1.63±0.001aB	3.25±0.01bA	3.28±0.02aA	3.38±0.14aA	3.27±0.19aA	1.93±0.8aB	1.42±0.02cB	1.57±0.02cB
	LT	1.63±0.001aE	3.26±0.08bBC	3.31±0.02aBC	3.32±0.07aB	3.14±0.04aC	1.55±0.03aE	2.89±0.01aD	4.20±0.0001aA
	LT2	1.63±0.008aB	1.64±0.03cB	1.52±0.01cB	1.56±0.14bB	1.59±0.03bB	1.51±0.05aB	1.61±0.09bCB	3.07±0.14bA
p-Value homoscedasticity		0.001	0.016	0.857	< 0.001	< 0.001	< 0.001	0.003	0.042
p-Value ANOVA/Welch		0.324	< 0.001	< 0.001	< 0.001	< 0.001	0.599	< 0.001	< 0.001
SV (mg KOH.g^{-1})	Uncoated	639±0.33aF	698±0.54dE	604±1.31eH	612±3.28eG	733±0.54dC	729±0.04dD	741±0.01cB	827±0.01aA
	B	639±0.09aH	663±0.52eF	709±0.33bE	725±0.05bD	744±0.03cC	808±0.19aA	772±0.005aB	651±0.40dG
	L	640±0.03aE	781±5.56aA	633±0.13dF	643±0.45dE	772±0.59bB	650±0.02eD	770±0.01bB	764±0.02bC
	LT	638±0.37aH	742±1.89bE	747±0.32aD	764±0.09aB	775±0.71aA	755±0.02cC	725±0.56dF	699±1.15cG
	LT2	639±0.35aG	726±0.80cD	695±1.39cF	708±0.06cE	744±0.28cC	793±0.15bA	770±0.001bB	631±0.09eH
p-Value homoscedasticity		0.026	0.003	0.005	< 0.001	0.035	0.001	< 0.001	< 0.001
p-Value ANOVA/Welch		0.011	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
IV (gI_2.100g^{-1})	Uncoated	59.1±0.03aE	54.6±0.03dG	82.8±0.01aB	102.6±0.08aA	81.8±0.02aB	74.5±0.03aD	78.2±0.04aC	56.6±0.04aF
	B	59.2±0.05aDE	58.8±0.03cDE	84.4±0.20aB	88.4±0.03bA	60.8±0.06bD	69.8±0.02bC	58.1±0.06cE	52.9±0.06bF
	L	59.2±0.10aDE	79.1±0.15aA	60.0±0.05bCDE	63.4±0.02cC	57.5±0.02cE	62.6±0.24cCD	66.4±0.23bB	41.0±0.02dF
	LT	59.3±0.09aBC	52.1±0.15eCD	47.8±0.60cDE	51.0±0.09dDE	48.1±0.005dDE	60.0±0.30cB	82.4±0.08aA	44.7±0.27cE
	LT2	59.5±0.11aAB	60.9±0.14bAB	53.9±0.05bcB	54.9±0.35dAB	57.2±0.16cAB	62.2±0.03cA	57.7±0.60cAB	25.4±0.04eC
p-Value homoscedasticity		0.188	0.002	0.001	0.007	0.007	0.004	0.059	0.002
p-Value ANOVA/Welch		0.079	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

	Uncoated	2.33±0.01aG	3.04±0.03abF	3.07±0.005abEF	3.09±0.01dE	4.54±0.01bcD	5.78±0.01cC	11.21±0.005bB	11.93±0.02cA
	B	2.30±0.01aF	2.55±0.04abF	3.26±0.24abE	3.49±0.01bE	4.82±0.5bD	11.32±0.005aC	12.09±0.005aB	13.12±0.04bA
PV (meq. Kg^{-1})	**L**	2.28±0.03aE	2.54±0.42bE	3.01±0.01bD	3.21±0.005cD	3.81±0.01dC	4.67±0.01dB	10.61±0.07cA	10.95±0.04eA
	LT	2.32±0.03aH	3.02±0.01abG	3.21±0.07abF	3.54±0.06bE	4.21±0.14cdD	4.69±0.01dC	7.28±0.02eB	11.59±0.03dA
	LT2	2.32±0.03aH	3.06±0.01aG	3.33±0.005aF	3.83±0.01aE	5.55±0.12aD	6.10±0.01bC	9.64±0.01dB	17.70±0.02aA
p-Value homoscedasticity		0.107	< 0.001	0.024	0.013	0.002	0.584	0.008	0.641
p-Value ANOVA/Welch		0.265	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Uncoated	7.77±0.01aA	4.37±0.03dG	6.28±0.02bD	6.29±0.01bC	5.92±0.01bE	4.30±0.005dH	5.05±0.006cF	7.11±0.07aB
	B	7.66±1.16aA	5.38±0.25bE	6.27±0.02bB	6.28±0.02bcB	5.61±0.08cD	4.48±0.52cF	5.63±0.03aD	5.87±0.06bC
p-AnV	**L**	7.77±0.01aA	5.12±0.74cE	6.23±0.02cB	6.25±0.02cB	6.01±0.01aC	4.80±0.02bF	5.05±0.23cE	5.58±0.64cD
	LT	7.74±0.01aA	5.07±0.04cC	6.73±0.02aB	6.74±0.01aB	3.56±0.02eG	4.85±0.09bD	4.49±0.02dF	4.55±0.02eE
	LT2	7.77±0.57aA	5.74±0.03aC	6.11±0.01dB	6.09±0.28dB	4.11±0.01dF	5.41±0.27aD	5.17±0.01bE	5.47±0.17dD
p-Value homoscedasticity		< 0.001	< 0.001	0.010	< 0.001	0.064	0.002	< 0.001	0.002
p-Value ANOVA/Welch		< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
	Uncoated	12.43±0.02aE	10.46±0.06bF	12.41±0.02bE	12.47±0.03dE	15.00±0.02aD	15.86±0.01cC	27.47±0.01bB	30.96±0.10cA
	B	12.27±1.14aE	10.48±0.31bF	12.78±0.49abE	13.26±0.03bE	15.26±0.92aD	27.12±0.51aC	29.81±0.04aB	32.12±0.07bA
TOTOX	**L**	12.33±0.06aD	10.20±0.77bE	12.25±0.03bD	12.66±0.03cD	13.63±0.02bC	14.14±0.04eC	26.27±0.17cB	27.49±0.58eA
	LT	12.38±0.06aF	11.12±0.06abH	13.15±0.14aE	13.83±0.10aD	11.98±0.31cG	14.23±0.07dC	19.05±0.07eB	27.73±0.08dA
	LT2	12.40±0.49aG	11.85±0.01aH	12.78±0.09abF	13.76±0.26aE	15.22±0.23aD	17.62±0.26bC	24.45±0.01dB	40.87±0.16aA
p-Value homoscedasticity		< 0.001	0.002	0.029	0.003	0.002	0.002	0.061	0.002
p-Value ANOVA/Welch		0.023	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

**Means followed by the same uppercase letter in each row and lowercase letter in each column do not differ significantly by Tukey's test at the 5% level. K232 - Dienes, K268 - Trienes, FFA - free fatty acid, IV - iodine value, PV - peroxide value, p-AnV - p-anisidine value, TOTOX – Total oxidation.

It can be seen that the diene and triene indices (Table 4) of the oil of the Brazil nuts uncoated from day 7 of storage showed a sharper increase than the oil of the Brazil nuts coated, which indicates the presence of primary oxidation products and a high presence of secondary products in the sample. It can also be seen that the oil of the Brazil nuts coated with B, LT, and LT2 was the most preserved throughout the storage period, i.e., these coatings slowed down the oxidation reaction of the coated nuts. Sartori et al. (2018) found diene values much higher than those observed in the present study in Brazil nuts packaged in amber and transparent glass, stored for 5 months; all values were higher than 3.

As for free fatty acids (Table 4), in general, the oil of the uncoated Brazil nuts and those coated with LT2 had the smallest changes in value when compared to the others. However, at 120 days of storage, this FFA increased in the oil of the nuts coated with LT and LT2. This increase can be attributed to the content of the mixture of tocopherols present in the coatings, which may have oxidized during the 120 days of storage.

Even with these results, the maximum value recommended by the National Health Surveillance Agency and the Codex Alimentarium Commission for cold-pressed crude oils is a maximum of 4 mg $KOH.g^{-1}$ (BRASIL, 2021; CODEX ALIMENTARIUM, 2009), i.e., only the Brazil nut oil coated with LT was higher than this value on the last day of storage. (Machado et al., 2023) in a study with Brazil nut oil from the south of Amazonas without indicating the form of extraction or preservation, 5.56 mg $KOH.g^{-1}$ was higher than the values obtained in the present study on all evaluation days. The industry widely uses FFA to monitor the oxidative stability of oil. However, recent studies have shown that free fatty acids may not reflect oxidative stability, as bound fatty acids are the preferred substrate for oxidation (SHEN et al., 2021).

The iodine value results (Table 4) from the 15 days of storage onwards showed that the oil of uncoated and coated Brazil nuts B maintained higher indices than the oil of the coated Brazil nuts L, LT, and LT2, with the except on time 90 of storage when the oil of coated Brazil nuts B were lower than L and LT. However, the oil of the coated Brazil nuts B and LT2 were statistically similar by Tukey's test ($p \leq 0.05$). At 90 and 120 days of storage, the oil of the Brazil nuts coated by LT showed higher values. This index shows the degree of oil saturation, demonstrating that the oil from Brazil nuts coated L, LT, and LT2 was more saturated than the oil from Brazil nuts uncoated and coated B on the storage days mentioned here.

The IV is important in indicating the oxidative stability of edible oils since a high degree of unsaturation in the oil increases its susceptibility to lipid oxidation. On the other hand, a low iodine value in oils is associated with good quality and longer shelf life (AZLAN et al., 2010; ORTEGA-NIEBLAS et al., 2001). It can be seen that the values found in this study were low for Brazil nut oil, as Machado et al. (2023) found a value of 108.81 $g.100g^{-1}$ for the Brazil nut oil evaluated in their study, so it can be said that Brazil nut oil with and without coating showed good oxidative stability based on the iodine index over the storage period at 25°C.

In the peroxide value (Table 4), the uncoated and coated Brazil nut oil was within the limits recommended by ANVISA and Codex Alimentarium, in which the maximum value for cold-pressed crude oils is no more than 15 meq/kg (BRASIL, 2021; CODEX ALIMENTARIUM, 2009), throughout the storage period, except at 120 days when the LT2 coated nut oil was higher than established. The oil of the Brazil nuts was preserved uncoated and coated with B, L, and LT for the entire period evaluated without harming consumer health. Only the oil of the coated Brazil nuts with LT2 showed a value of 17.62 $meq.Kg^{-1}$, which is higher than that recommended by legislation.

Machado et al. (2023) found a peroxide value of 17.26 meq. Kg^{-1} for the Brazil nut oil evaluated in their study, so it can be said that Brazil nut oil with and without coating showed good oxidative stability based on PV for 90 days of storage at 25°C. Sartori et al. (2018) found PV values close to 3, 3.5, and 7 $meq.Kg^{-1}$ at 0, 30, and 60 days, respectively, for Brazil nut oil stored in amber and transparent glass, but the authors observed that after 90 days of storage, there was a differentiation since the PV was close to 8 $meq.Kg^{-1}$ for Brazil nut oil stored in amber glass at 90 and 120 days, while for Brazil nut oil stored in transparent glass, the values were close to 10.5 and 15 $meq.Kg^{-1}$ at 90 and 120 days, respectively. PVs close to those observed in this study for the same storage times.

The p-anisidine value (Table 4) measures secondary degradation products, measuring the amount of non-volatile aldehydes present in the oil (BEN HAMMOUDA et al., 2018). ANVISA and Codex Alimentarium recommend no limits for p-AnV. However, values were observed in the first storage time close to 8, which decreased after packaging in coatings and in an environment with controlled temperature (25±0.5°C).

The Totox oxidation (Table 4) of the oil of the uncoated and coated Brazil nuts showed values above 10 throughout the storage period. TOTOX is calculated by associating PV (primary oxidation products) with p-AnV (secondary oxidation products). Hydroperoxides (peroxides) are unstable and do not provide a reliable picture of an oil's oxidative stability. Therefore, TOTOX lower than 10 indicates better oil quality (TAVAKOLI et al., 2019). Values greater than 10 indicate the oil’s low stability (REGITANO-D’ARCE, 2010).

Oils with a high degree of unsaturation are more susceptible to oxidation (REGITANO-D'ARCE, 2010). Brazil nuts have a high content of polyunsaturated fatty acids (DA COSTA et al., 2011) evaluated the fatty acid profile of Brazil nuts and found that they contained $75.34g.100g^{-1}$ of polyunsaturated fatty acids. (KORNSTEINER-KRENN et al., 2013) evaluated the fatty acid composition of nuts and observed a polyunsaturated fatty acid content of $55.7g.100g^{-1}$ in Brazil nuts. Despite not having analyzed the fatty acid composition of the Brazil nuts used in this study, the fatty acids present in their composition contributed greatly to the oxidation state of the nuts. The nuts under study already had some degree of oxidation when the coatings were applied, which may have contributed to the results.

3.4.4 Induction time and Modeling Oxidation kinetics of oil of the Brazil nuts uncoated and coated during storage time

Figure 5 shows the induction time of oil of the Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

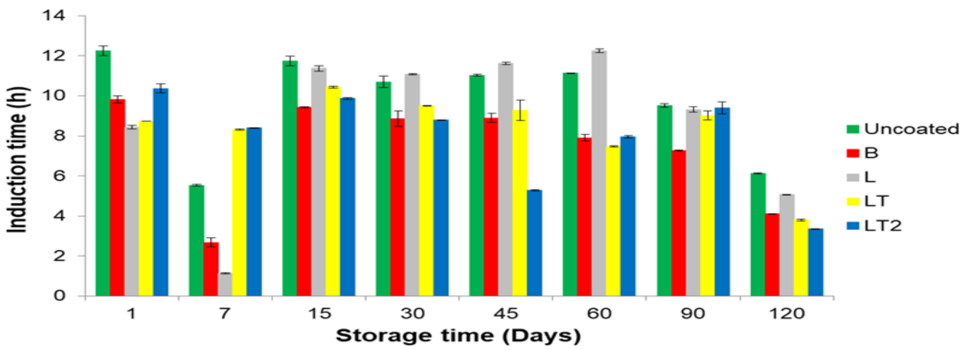


Figure 5. Induction time of oil of Brazil nuts uncoated and coated with a coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

The development of undesirable compounds resulting from lipid oxidation is an important problem that needs to be solved in order to prolong the shelf life of oils, fats, and fatty foods. In order to minimize the damage caused to food by oxidation and preserve the oxidative stability of oils, the oilseed industry uses antioxidants (COSTA et al., 2014); however, in this study, the oxidant was not added directly to the oil but the coatings that covered the oilseed. The antioxidant was only added to the LT and LT2 coatings.

It can be seen that during the induction time (Figure 5), the oil of the coated Brazil nuts underwent some changes in the oxidative state, although the use of the coatings largely preserved them. However, at 120 days, as the Brazil nuts coated with B, LT, and LT2 had the shortest induction times, corroborating the PV results at the same storage time.

The Rancimat test can be used to evaluate the oxidative stability phases of vegetable oils (GHARBY et al., 2016; MASZEWSKA et al., 2018). The Rancimat test by conductimetry determines the oil's resistance time to oxidation (FIGUEREDO et al., 2020; LE PRIOL et al., 2021). At 7 days of storage, the uncoated and B and L coated Brazil nut oil showed a sharp drop in IT values due to the absence of the antioxidant protecting the uncoated and B and L coated packaged Brazil nut oil. At 45 days of storage, the Brazil nut oil coated with LT2 (the highest concentration of the tocopherol mix) showed a sharp drop in induction time. This is probably because, at 45 days of storage, the oil analyzed was more susceptible than the oil in the other samples to the heating process undergone during the test since, throughout the test and storage time, the oil undergoes various changes in the degree of unsaturation in its composition, so it can be inferred that during the 45-day storage period, some change in the degree of unsaturation caused the decrease in IT, i.e., the induction time was shorter than the storage time. e., a decrease in the oxidative stability of Brazil nuts coated with LT2.

Table 5 shows the results of the fit parameters of the Zero, First, and Second Order Models of oil of the Brazil nuts uncoated and coated of the coatings of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

Table 5. Mean±standard deviation of the fit parameters of the Zero, First, and Second Order Models of oil of the Brazil nuts uncoated and coated of the coatings of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

Oil of Brazil nuts coated and uncoated	Fit Parameters – Zero-Order Model						
	A0	k	P	R^2	SE	χ^2	θ(1/2)
Uncoated	12.66	0.045	4.35	0.83	0.85	0.73	141.88
B	10.29	0.044	2.44	0.89	0.64	0.41	117.92
L	11.57	0.033	26.47	0.30	2.10	4.41	175.91
LT	9.89	0.034	14.66	0.49	1.45	2.10	145.36
LT2	9.69	0.038	22.82	0.44	1.80	3.26	126.67
	Fit Parameters – First-Order Model						
	A0	k	P	R^2	SE	χ^2	θ(1/2)
Uncoated	12.72	0.036	19.85	0.46	1.67	2.84	135.62
B	10.39	0.038	20.51	0.47	1.71	2.93	125.58
L	11.40	0.004	5.51	0.78	0.96	0.92	164.20
LT	9.85	0.005	3.65	0.84	0.78	0.61	129.86
LT2	9.80	0.003	28.22	0.26	2.17	4.70	240.09

	Fit Parameters – Second-Order Model						
	A0	k	P	R^2	SE	χ^2	θ(1/2)
Uncoated	13.96	0.0006	0.71	0.73	0.01	0.12	119.42
B	12.22	0.0010	0.93	0.74	0.02	0.16	81.95
L	12.84	0.0006	0.76	0.41	0.03	0.13	129.83
LT	11.35	0.0009	1.03	0.53	0.04	0.15	97.94
LT2	11.08	0.0012	1.16	0.50	0.05	0.17	75.21

A0 = initial concentration of the evaluated parameter; k = rate constants; P = relative mean error; R^2 = correlation coefficient, SE= estimated mean error, χ^2 = chi-squared, θ(1/2) = half-life time.

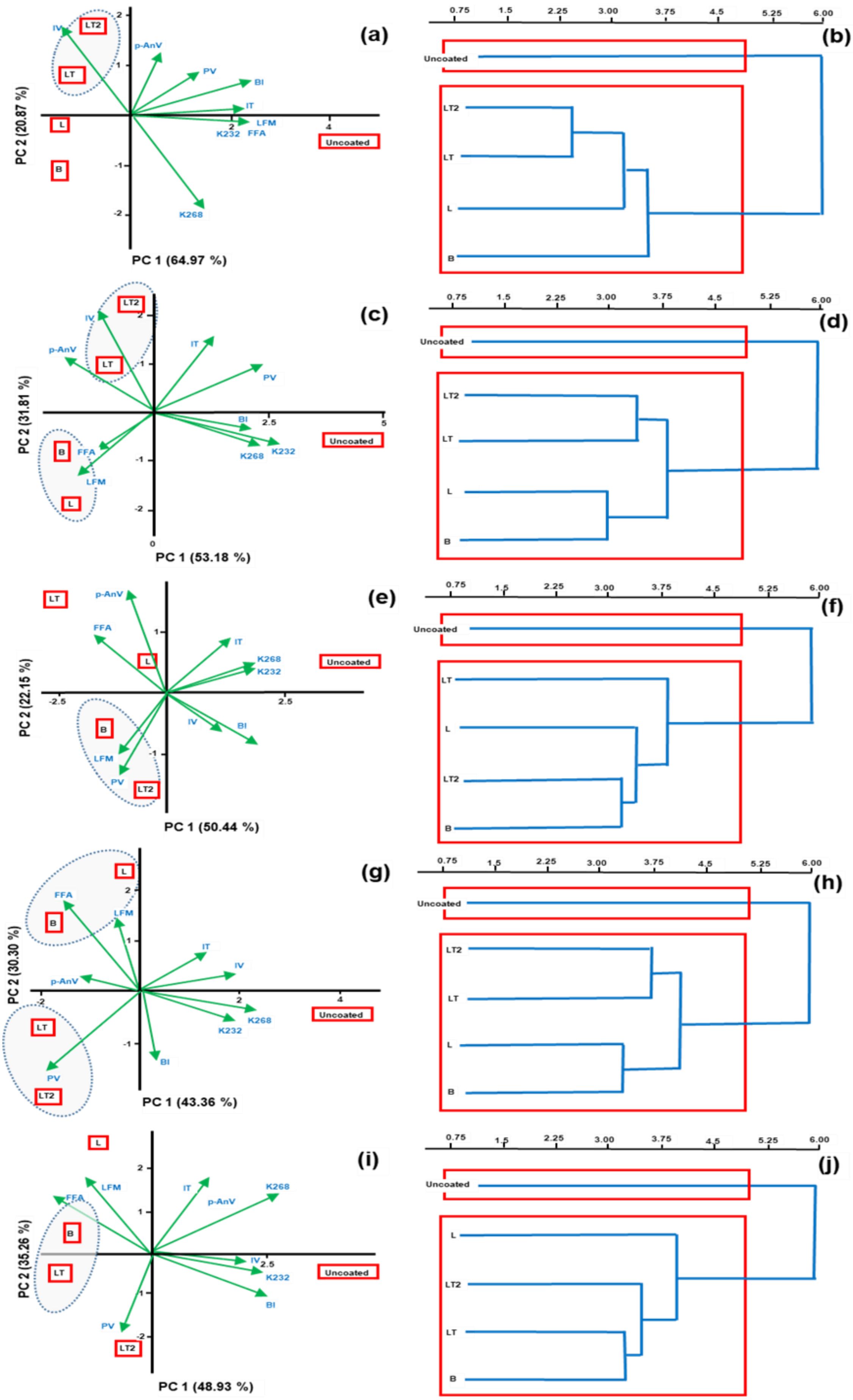
It can be seen that the zero-order model (Table 5) best fitted the experimental data for oil of Brazil nut uncoated and coated B. Meanwhile, the first-order model best fits the data for oil of the Brazil nut coated L and LT, generally showing better-fit coefficients. However, the oil of Brazil nut coated LT2 did not show a good fit for any of the models.

In addition, the models applied to the data from the accelerated oxidation test, in which the Brazil nut oil without coating and with coatings B, L, LT, and LT2 were subjected to a temperature of 110°C, made it possible to estimate the half-life of the oils. Checking the models that showed the best fit to the Brazil nut oil data showed that the oil from the nuts coated with L had the longest half-life - 164.20h. On the other hand, when considering the half-life of Brazil nut oil coated with LT and LT2 (added to the tocopherol mix) and considering that LT2 did not show a good fit with any of the models tested, it was found that the LT coating showed the highest value of 129.86h in the first-order model.

The greater the amount of unsaturation, the more susceptible the oil is to degradation (BODOIRA et al., 2017; FARHOOSH; MOOSAVI, 2007). Thus, it can be said that the increase in temperature and exposure time during the test caused a decrease in the level of unsaturations present in the uncoated and coated Brazil nut oil, which is closely linked to the half-life since the degree of degradation of an oil can dictate the shelf life of a lipid product (BODOIRA et al., 2017).

3.4.5 Principal component analysis (PCA) and, Hierarchical cluster (HCA) of the evaluation of the oxidation level of oil of the Brazil nuts uncoated and coated during storage time

Figure 6 shows the biplot graphic of principal component analysis (PCA) and hierarchical cluster (HCA) of properties of the evaluation of the oxidation level of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT, and LT2 (with soy lecithin and tocopherol mix) during storage time.



(a)
PC 2 (20.87 %)
PC 1 (64.97 %)
LT2
LT
L
B
Uncoated
IV
p-AnV
PV
BI
IT
LFM
K232
FFA
K268
(b)
0.75 1.5 2.25 3.00 3.75 4.5 5.25 6.00
Uncoated
LT2
LT
L
B
(c)
PC 2 (31.81 %)
PC 1 (53.18 %)
LT2
LT
B
L
Uncoated
IV
p-AnV
IT
PV
BI
K268
K232
FFA
LFM
(d)
0.75 1.5 2.25 3.00 3.75 4.5 5.25 6.00
Uncoated
LT2
LT
L
B
(e)
PC 2 (22.15 %)
PC 1 (50.44 %)
LT
L
B
LT2
Uncoated
p-AnV
FFA
IT
K268
K232
IV
BI
LFM
PV
(f)
0.75 1.5 2.25 3.00 3.75 4.5 5.25 6.00
Uncoated
LT
L
LT2
B
(g)
PC 2 (30.30 %)
PC 1 (43.36 %)
L
B
LT
LT2
Uncoated
FFA
LFM
IT
IV
p-AnV
K268
K232
BI
PV
(h)
0.75 1.5 2.25 3.00 3.75 4.5 5.25 6.00
Uncoated
LT2
LT
L
B
(i)
PC 2 (35.26 %)
PC 1 (48.93 %)
L
B
LT
LT2
Uncoated
LFM
FFA
IT
p-AnV
K268
IV
K232
BI
PV
(j)
0.75 1.5 2.25 3.00 3.75 4.5 5.25 6.00
Uncoated
L
LT2
LT
B

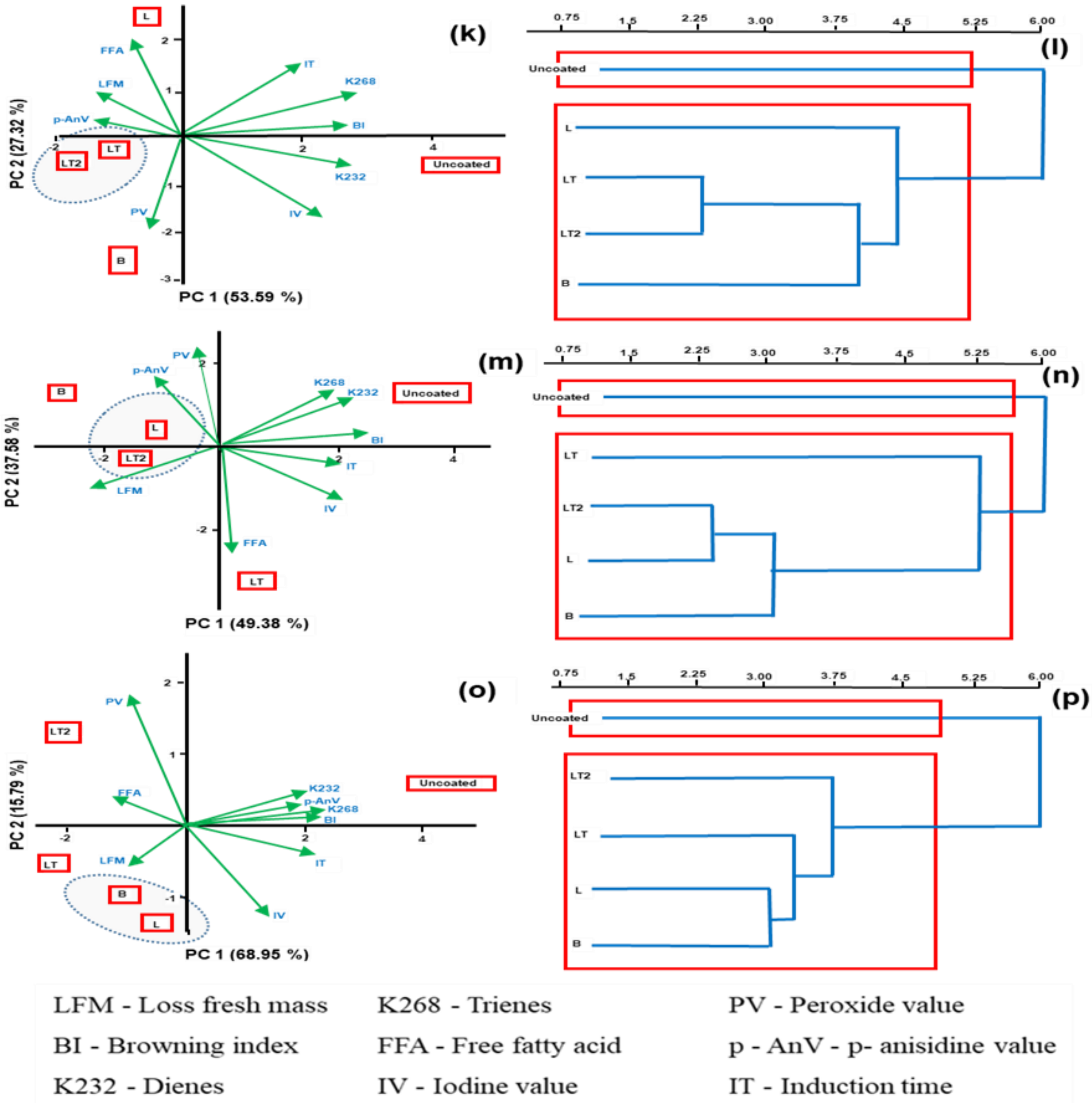


Figure 6. Biplot graphic of principal component analysis (a, c, e, g, i, k, m, and o) on each storage day (1, 7, 15, 30, 45, 60, 90, and 120 days, respectively), and hierarchical cluster (b, d, f, h, j, l, n and p) on each storage day (1, 7, 15, 30, 45, 60, 90, and 120 days, respectively) of properties of the evaluation of the oxidation level of Brazil nuts uncoated and coated with coating of the cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix) during storage time.

The variability explained by PCA (Figure 6 - a, c, e, g, i, k, m and o) was 85.84 %, 84.99 %, 72.59 %, 73.66 %, 84.19 %, 80.91 %, 86.96 % and 84.74 % on each storage day (1, 7, 15, 30, 45, 60, 90 and 120 days, respectively). In addition, it can be seen that on most of the storage days evaluated, there was the formation of the LT-LT2 group, indicating a similarity between the oxidation parameters of Brazil nuts for most of the storage days of these samples. There was also apparent isolation of the Uncoated group, corroborating the HCA (Figure 6 - b, d, f, h, j, l, n, and p), which showed that only on days 15, 45, 90 and 120 (Figure 6 - f, j, n and p, respectively) of storage was the LT-LT2 cluster not observed, on the other days of storage this cluster was formed, demonstrating the similarity between the parameters of LT and LT2. In addition, looking at the HCA, it can be seen that the clusters formed by the Euclidean distance show the formation of four clusters, and it can also be seen that the Brazil nuts uncoated are isolated at all storage times.

These results show that all the coatings applied to the Brazil nuts promoted the preservation of the samples in some of the evaluation periods. They also showed that most of the oxidative indices negatively influenced all the coated samples, especially the Brazil nuts coated with LT and LT2 coatings containing the tocopherol mix in their composition. That is an advantage, as it shows that the coatings generally protected the coated Brazil nuts, especially the Brazil nuts coated with LT and LT2.

4 CONCLUSÃO

In the rheological evaluation, the blend-forming solutions B, L, LT, and LT2 showed non-Newtonian and pseudoplastic behavior. All blend-forming solutions showed excellent resistance to flow and were stable. The pH of all the blend-forming solutions that were developed was close to neutral. The color of the blend-forming solutions tended slightly toward white, low (achromatic), revealing weak or diluted coloration and a slightly greenish-yellow tonality. Adding a tocopherol mix promoted a greater tonality in the blend-forming solutions LT and LT2.

The LT and LT2 coatings minimized the loss of fresh mass of Brazil nuts compared to B and L coatings but were higher when compared to uncoated Brazil nuts. The browning index of the coated Brazil nuts was lower than that of the uncoated Brazil nuts for almost the entire storage period; LT showed more stable values after 30 days of storage. Regarding the evaluation of the oxidative state of the uncoated and coated nuts, in general, the oil from the coated LT and LT2 Brazil nuts added to the tocopherol mix was preserved; the indices provided an insight into the possible minimization of oxidation of the Brazil nut oil under study. PCA showed that all coatings applied to Brazil nuts promoted oil preservation in some of the evaluation periods, especially the coatings added with a tocopherol mix. More research is needed. The first suggestion would be to determine the fatty acids in Brazil nut oil during the storage period to test the migration of the tocopherol mix into the headspace; the second suggestion is to apply it in the form of a film, and the third is to apply it to other foods.

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4 DISCUSSÃO INTEGRADA

Ao avaliar os resultados foi possível verificar que a partir da busca realizada no Capítulo 1 com os termos descritores e operadores booleanos citados anteriormente, as buscas retornaram 33 artigos referentes a filmes e revestimentos adicionados de tocoferol para uso em alimentos. No período avaliado não foi encontrado nenhum artigo nas bases de dados que se referisse a filmes ou revestimentos que associassem fécula de mandioca e carboximetil celulose em um polímero ativo contendo um mix de tocoferóis. E a partir a análise bibliométrica realizada também no Capítulo 1, foram observadas as palavras-chaves mais pesquisadas no período de 2017 a 2022, nas bases de dados PubMed, Science Direct, Scopus and Web of Science, entre elas: quitosana, antioxidante, química e alfa-tocoferol, mostrando uma lacuna frente aos polímeros escolhidos para o presente estudo, assim como para o mix de tocoferóis. O Capítulo 1 mostrou também diversos comportamentos das propriedades de filmes e revestimentos adicionados de tocoferol, a depender do polímero utilizado e composição selecionada para a montagem do filme ou revestimento, o que contribuiu como comparativo para alguns os resultados observados na determinação de propriedades avaliadas no Capítulo 2.

Ao avaliar o Capítulo 2 foi possível verificar o desenvolvimento das formulações e preparados filmes a partir de um blend de fécula de mandioca e carboximetil celulose adicionados de um mix de tocoferol, os quais foram testados para se chegar a características próximas ao que seriam as características de um revestimento, já que um revestimento, depois de aderido à superfície de um alimento dificilmente se solta, impossibilitando a realização de testes mecânicos por exemplo. Assim os resultados do Capítulo 2 mostraram que o uso de lecitina de soja nos filmes L, LT e LT2 tornou a estrutura mais homogênea e densa. Os filmes adicionados do mix de tocoferol (LT e LT2) apresentaram-se mais higroscópicos, apresentando permeabilidade ao vapor d’água de 0,833 e 0,873g.mm.h^{-1}.m^{-2}.kPa^{-1}, respectivamente e solubilidade em água de 29,03 e 45,59%, respectivamente, maiores que os filmes sem tocoferol em sua composição. Vasco et al. (2022) desenvolveram películas comestíveis de carboximetil celulose, amido de mandioca ou alginato e observaram 22,8 e 85% de solubilidade em água para as películas de amido de mandioca e CMC, respetivamente, resultados diferentes dos do presente estudo. Os filmes com maior concentração desse emulsificante, L e LT2 foram mais higroscópicos devido à interferência da concentração de lecitina de soja, pois o ângulo de contato apresentou menores valores sendo 41.6° e 40.0°, respectivamente.

Ainda no Capítulo 2 foi com a determinação da atividade antioxidante foi possível comprovar que após o processo de desenvolvimento dos filmes e as respectivas etapas de aquecimento a ação antioxidante foi preservada nos filmes LT e LT2. Esses filmes também apresentaram barreira acima de 30% à radiação ultravioleta e acima de 25% à radiação infravermelha. Além disso, no Capítulo 2 a PCA aplicada aos dados de caracterização dos filmes permitiu visualizar quais parâmetros tecnológicos tiveram maior peso para cada filme. Essa ferramenta foi importante para mostrar que os filmes adicionados de mix de tocoferóis (LT e LT2) poderiam ser indicados como filmes antioxidantes para proteção de alimentos, mas pela caraterística de ser muito solúvel, poderia ser aplicado como um filme comestível ou mesmo um revestimento comestível.

Ao avaliar os resultados do Capítulo 3, no qual foram realizados testes nas soluções formadoras dos revestimentos que possibilitaram caracterizar as soluções formadoras dos revestimentos B, L, LT e LT2 como fluido não-newtoniano e pseudoplástico, devido a composição das soluções blends formadoras dos revestimentos que proporcionou boa resistência ao escoamento, boa estabilidade. No capítulo 3 foram preparadas as soluções blend B, L, LT e LT2 e aplicadas em forma de revestimento nas castanhas-do-brasil, as quais foram acondicionadas a 25°C por 120 dias, castanhas-do-brasil sem revestimento foram acondicionadas nas mesmas condições como comparativo. De maneira geral todos os revestimentos aplicados promoveram a minimização da oxidação em alguma etapa do armazenamento, porém os filmes adicionados do mix de tocoferol promoveu a minimização de dienos, trienos, índice de iodo, índice de peróxido, p-anisidina e do índice de oxidação total apresentados na Tabela 4 do Capítulo 3, de acordo com os resultados pode-se afirmar que as castanhas já possuíam algum nível de oxidação desde o primeiro tempo de armazenamento, porém o estudo possibilitou verificar certo nível de proteção proporcionado pelos revestimentos adicionados de tocoferol.

Quanto a avaliação da oxidação acelerada o óleo das castanhas-do-brasil em estudo sofreu algumas alterações no estado oxidativo, embora tenham sido em grande parte, preservadas pelo uso dos revestimentos, provavelmente devido à mudança no grau de insaturações de sua composição ocorridas ao longo do período de armazenamento. A modelagem da cinética de oxidação mostrou que o modelo de ordem zero foi o que se ajustou melhor aos dados experimentais para o óleo de castanha-do-brasil sem revestimento e com revestimento B. Enquanto, o modelo de primeira ordem se ajustou melhor aos dados do óleo de castanha-do-brasil com revestimento L e LT, apresentando, em geral, coeficientes de melhor ajuste. No entanto, o óleo de castanha-do-brasil revestido por LT2 não apresentou um bom ajuste para nenhum dos modelos. Além disso, os modelos aplicados aos dados do teste de oxidação acelerada permitiram estimar a meia-vida dos óleos, assim verificou-se que o revestimento LT apresentou valor de 129,86h no modelo de primeira ordem.

Por fim, no Capítulo 3 a PCA aplicada aos dados de caracterização físico-química e ao tempo de indução revelou que o óleo das castanhas não revestidas apresentou-se isoladamente sendo extremamente influenciado pelos índices oxidativos avaliados, mas o que chamou bastante atenção é que em todos os dias de armazenamento foram os valores da variabilidade dos dados formada pelos PC1 e PC2, que na maioria dos tempos de armazenamento foram maiores que 75%. A clusterização mostrou a formação de quatro grupos, principalmente do grupo LT e LT2, demonstrando a similaridade entre os parâmetros apresentados pelo óleo das castanhas-do-brasil revestidas por esses revestimentos e nitidamente separado o grupo do óleo das castanhas não revestidas. Contudo, o óleo das castanhas-do-brasil revestidas com LT e LT2 foram de acordo com a PCA influenciados negativamente pela maioria dos índices, nesse caso isso é uma vantagem, pois demonstra menores índices de oxidação lipídica do óleo dessas amostras.

Em síntese, ao desenvolver um filme com propriedades antioxidantes, com características indicadas para uso como um filme ou revestimento comestível, o qual foi aplicado como revestimento em castanha-do-brasil possibilitou observar alguns níveis de proteção das castanhas-do-brasil, o que é um grande avanço para esse produto, já que é um produto que é exposto para venda em embalagens com partes transparentes, que possibilitam a passagem da luz diretamente sobre o produto, assim a aplicação de uma proteção contra as radiações infravermelha e ultravioleta é uma vantagem frente a algumas embalagens utilizadas atualmente para as castanhas-do-brasil.

Outra vantagem é a atividade antioxidante que foi comprovadamente visto que se manteve nos filmes, assim sendo o uso como filme ou revestimento é fortemente indicado para uso não somente em castanhas-do-brasil, mas em outros tipos de alimentos.

Corroborando com o trabalho de Dias et al. (2018) que estudaram filmes de quitosana contendo tocoferol e aplicaram em filé de salmão, o produto foi embalado e acondicionado a 4°C por 8 dias, os autores relataram que a oxidação do produto foi minimizada pelos filmes adicionados de tocoferol. Assim como no estudo de Shokri e Ehsani (2017), em que foi aplicado revestimento de proteína de soro de leite em filés de peixe, acondicionados a 4°C por 16 dias, e os revestimentos adicionados de tocoferol também apresentaram ação antioxidante no produto.

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SHOKRI, S.; EHSANI, A. **Efficacy of whey protein coating incorporated with lactoperoxidase and α-tocopherol in shelf life extension of *Pike-Perch* fillets during refrigeration.** LWT - Food Science and Technology, v. 85, p. 225–231, 2017.

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5 CONCLUSÃO DA TESE E PERSPECTIVAS

Na área da ciência e tecnologia de alimentos procura-se desenvolver polímeros capazes de promover o prolongamento da vida útil do produto alimentício, por isso conhecer as propriedades é vital para esta área de investigação, uma vez que combinar um material polimérico biodegradável com um antioxidante natural ativo é de grande interesse para a sociedade moderna, pois associa a preservação ambiental à preservação de alimentos.

Ao realizar a revisão foi possível encontrar 33 artigos publicados no período de 2017 a 2022 sobre filmes e revestimentos adicionados de tocoferol para uso em embalagens de alimentos. As principais propriedades foram abordadas. Assim, foi possível observar que as propriedades, em conjunto ou separadamente, podem direcionar a aplicação e o produto a que se destina. Esta revisão também possibilitou o levantamento das redes de coocorrência de palavras-chave relacionadas a esse tema em cada base de dados investigada. A análise dos dados por meio da ferramenta VOSviewer possibilitou uma melhor visualização e exploração dessas palavras e a elaboração de mapas que evidenciaram as principais conexões entre as publicações.

Dos filmes que foram desenvolvidos nesse estudo, os que continham lecitina de soja em sua composição (L, LT e LT2) foram fortemente influenciados pela presença do emulsificante, esses filmes tinham a microestrutura mais densa e também eram mais hidrofílicos que o filme controle B, mas os filmes LT e LT2 adicionados do mix de tocoferol eram ligeiramente verde-amarelados. Além disso, todos os filmes desenvolvidos são biodegradáveis e LT e LT2 mantiveram a atividade antioxidante. O filme LT apresentou maior barreira à radiação infravermelha, e o LT2 apresentou maior barreira à luz visível e à radiação ultravioleta, sendo mais opaco que os demais filmes. A PCA mostrou que os filmes LT e LT2 mantiveram sua bioatividade e são fortemente recomendados para uso em embalagens ativas de alimentos.

Na avaliação reológica todas as soluções formadoras dos revestimentos apresentaram comportamento não newtoniano e pseudoplástico. As soluções que apresentavam maior concentração de lecitina de soja (L e LT2) em sua composição apresentaram maior resistência ao escoamento, e todas as soluções foram estáveis, conforme comprovado pelos testes de cremeação e centrifugação, uma vez que nenhum das soluções apresentou separação de fases durante o período de avaliação. O pH de todas as soluções formadoras de blends desenvolvidas, mas principalmente do LT2, ficou próximo do neutro, beneficiando a estabilidade das soluções. A cor das soluções blends tenderam levemente ao branco, acromática, revelando coloração fraca ou diluída e tonalidade levemente amarelo-esverdeada, a adição do mix de tocoferóis promoveu maior tonalidade nas soluções formadoras dos revestimentos LT e LT2.

Os revestimentos LT e LT2 (adicionados do mix de tocoferóis) minimizaram a perda de massa fresca da castanha-do-brasil quando comparados aos revestimentos B e L, mas foram superiores quando comparados às castanhas-do-brasil não revestidas. O índice de escurecimento das castanhas-do-brasil revestidas foi inferior ao das castanhas-do-brasil não revestidas durante quase todo o período de armazenamento, o LT apresentou valores mais estáveis após 30 dias de armazenamento.

Quanto à avaliação do estado oxidativo das castanhas não revestidas e revestidas, de modo geral, o óleo das castanhas-do-brasil revestidas por LT e LT2 foi preservado, apesar dos altos índices de saponificação apresentados nas amostras desde o início do estudo, os demais índices forneceram uma visão sobre a possível minimização da oxidação do óleo da castanha-do-brasil em estudo. Assim, os índices de dienos, trienos, IV, PV, p- AnV e Totox mostraram que a aplicação do revestimento comestível à base do blend de fécula de mandioca/carboximetil celulose adicionado do mix de tocoferóis preservou a castanha-do-brasil até 90 dias de armazenamento a 25 °C. A PCA mostrou que todos os filmes aplicados nas castanhas-do-brasil promoveram a preservação do óleo em algum dos períodos de avaliação, especialmente os filmes adicionados com o mix de tocoferóis, pois preservou as castanhas-do-brasil em níveis aceitáveis até 90 dias de condicionamento a 25±0,5° C.

Como sugestões de perspectivas futuras indica-se a aplicação dos blends desenvolvidos na forma de filme ou revestimento ativo, para outros produtos alimentícios; A segunda sugestão seria determinar os ácidos graxos do óleo da castanha-do-brasil durante o período de armazenamento; A terceira sugestão, testar a migração do mix de tocoferóis para o headspace. Além disso, sugere-se ampla divulgação para o meio científico e público em geral, pois é um material altamente biodegradável e antioxidante, que pode ter resultados muito bons se aplicados a castanha-do-brasil e até em outros produtos alimentícios, respeitando-se é claro, testes de aplicação e avaliação de qualidade do produto.

ANEXO A – Artigo aceito para publicação na revista *British Food Journal.*

From: rob.hamlin@otago.ac.nz

To: danusa_silvacosta@hotmail.com, lucelynogueira@gmail.com, nelson.ufpa@gmail.com, katiucha@cpd.ufmt.br, alessalopes@ufpa.br

CC:

Subject:

British Food Journal - Decision on BFJ-04-2023-0292.R2

Body:

23-Jan-2024 Dear da Costa, Danusa; dos Santos, Lucely; Ferreira, Nelson Rosa; Takeuchi, Katiuchia; Lopes, Alessandra It is a pleasure to accept your manuscript BFJ-04-2023-0292.R2, entitled "PROPERTIES OF FILMS AND COATINGS ADDED OF TOCOPHEROL FOR FOOD PACKAGING: Tool-based review for systematic reviews and bibliometric analysis" in its current form for publication in British Food Journal. Please note, no further changes can be made to your manuscript. This email will be followed by a second message containing a copy of your author accepted manuscript (AAM) which is the version that we will typeset and publish in the journal. Your article will now go through editorial checks by Emerald's editorial team to ensure it meets our publication standards. These checks can take up to five days; we'll be in touch if we have any queries at this stage. Once this step has been completed you will receive an email directing you to Emerald Submit to select your publishing licence and submit your article to production. If you are publishing in one of our subscription journals and wish to make your article open access you can choose this option in Emerald Submit. If you have not received an email with editorial queries or an invitation to complete licensing on Emerald Submit within 10 working days of acceptance, please do contact the JEO (Journal Editorial Office), you can find their details on the journal homepage: https://www.emeraldgrouppublishing.com/journal/bfj Once you have completed licensing on Emerald Submit, your article will enter the production process and you'll be provided with a proof. You will need to approve your proof before your article is published. If you have any queries about the proofing system you can contact the journal's Supplier Project Manager (SPM) whose contact details are on the journal homepage: https://www.emeraldgrouppublishing.com/journal/bfj. By publishing in this journal your work will benefit from Emerald EarlyCite. Once the above steps are completed your article will be published online in EarlyCite. EarlyCite is the author proofed, typeset version of record, fully citable by DOI. The EarlyCite article sits outside of a journal issue and is paginated in isolation. The EarlyCite article will later be collated into a journal issue according to the journals' publication schedule. Thank you for your contribution. On behalf of the Editors of British Food Journal, we look forward to your continued contributions to the Journal. Yours sincerely, Dr. Robert Hamlin Editor, British Food Journal rob.hamlin@otago.ac.nz -- Tell us how we're doing! We'd love to hear your feedback on the submission and review process to help us to continue to support your needs on the publishing journey. Simply click this link https://eu.surveymonkey.com/r/F8GZ2XW to complete a short survey and as a thank you for taking part you have the option to be entered into a prize draw to win £100 in Amazon vouchers. To enter the prize draw you will need to provide your email address.

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ANEXO B – Comprovante de aceite do artigo para publicação na revista *Polymer Testing* e Cópia do artigo

em Polymer Testing

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Action	Manuscript Number	Title	Initial Date Submitted	Status Date	Current Status	Date Final Disposition Set	Final Disposition
View Submission View Decision Letter Publishing Options Send E-mail	POTE-D-23-00639	TECHNOLOGICAL PARAMETERS OF CASSAVA STARCH/CARBOXYMETHYL CELLULOSE BLEND-BASED FILMS ADDED OF SOY LECITHIN AND TOCOPHEROL MIX	Jul 04, 2023	Oct 19, 2023	Completed - Accept	Oct 19, 2023	Accept

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Technological parameters of cassava starch/carboxymethyl cellulose blend-based films added of soy lecithin and tocopherol mix

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ABSTRACT

The aim was to evaluate the technological properties of films based on cassava starch/carboxymethyl cellulose added to soy lecithin and tocopherol mix. The films were prepared by casting from a cassava starch/carboxymethyl cellulose mixture and identified as control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and LT and LT2 (with soy lecithin and tocopherol mix). Thickness, water solubility, water content, film weight, contact angle, permeability test, mechanical tests, antioxidant activity, biodegradability, color, thermogravimetry, and FTIR tests were performed. The results showed that the films containing soy lecithin (L, LT, and LT2) were more hydrophilic and had a denser structure than film B, but the LT and LT2 films were slightly yellow-green, malleable and showed few bubbles or cracks. In addition, all the films developed are biodegradable. It was also found that the antioxidant remained in the films to which it was added (LT and LT2) and that the LT2 film showed the highest antioxidant activity. It is strongly recommended for application in active food packaging.

1. Introduction

The interest in the food packaging industry is increasing regarding environmentally friendly materials [1]. Hydrocolloid-based polymers are a strategy to replace petroleum-based materials. These polymers may have better protection against the passage of oxygen than synthetic plastics due to the polymer network having a complex pathway throughout the polymer [2]. The high oxygen barrier of hydrocolloid-based polymers can be seen in the study by Poulose et al. [3], who studied the oxygen barrier properties of potato fruit juice-based films and observed a ratio of 0.025–0.042 cm^3 per (m^2 day atm).

Starch has become a very promising natural polymer for replacing synthetic polymers being renewable, widely available, and attractively priced. Several botanical sources of starch have been studied for their film-forming properties; some examples are corn, yam, and cassava [4]. One concern with starch-based packaging materials focuses on the hydrophilicity of this polysaccharide, as they cause weakness in the water barrier of polymers [5].

Carboxymethyl cellulose (CMC) is formed in an alkaline medium by the reaction of sodium monochloroacetate with cellulose, one of the most relevant products; it has significant economic importance, as it is widely used as a thickening and emulsifying agent in food products [6]. This polysaccharide has high water solubility and generally provides transparent polymers; water sensitivity is not inconvenient; it also provides good mechanical and barrier characteristics [7,8].

The advantage of mixing polymers seeking to combine attributes can often be a disadvantage, as some assemblies may interfere with polymer recycling [9]. One strategy to enhance the properties and applicability of biopolymers for use in the packaging of food products is the use of lipids in the composition of these polymers because, due to their hydrophobic nature, in conjunction with the polymer matrix, can minimize the hydrophilicity [10].

An example of the application of a lipid in a polymeric base is the study by Costa et al. [11] which aimed to develop and evaluate the properties of a lipid compound, buriti oil, in cassava starch films, seeking to add the bioactivity of buriti oil, which the authors proved

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throughout the paper. Another example of the application of lipids is the study by Santos et al. [12] which developed films of gum arabic, carboxymethyl cellulose, and sodium alginate added to anise essential oil; the authors observed that the evaluated properties of solubility, water vapor permeability and mechanical properties of CMC films with oil showed results that direct the polymer for use as an edible coating.

In the work of Oliveira Filho et al. [13] with arrowroot starch films added of the carnauba wax (lipid compound), they observed a decrease in water solubility (mean 60.7 % in control film and 22.5 % in film added of the 15 % carnauba wax) and water vapor permeability (mean 6.7 10^{-7} g H_2O/m h Pa in control film and 4.2 10^{-7} g H_2O/m h Pa in film added of the 15 % carnauba wax). In this study, an increase in the hydrophobicity of the films was also observed (contact angle mean 42° in control film and 84° in film of the 15 % carnauba wax added), as well as an increase in elongation at break (mean 119 % in control film and 221.7 % in films of the 15 % carnauba wax added).

"Vitamin E is a fat-soluble and plant-derived substance; it refers to a group of eight different compounds: α-, β-, γ-, and δ-tocopherols and the four corresponding tocotrienols" [14]. Tocopherol is an antioxidant widely applied in films and coatings for food packaging in several polymeric matrices – e.g., Agudelo-Cuartas et al. [15] in whey protein, Drago et al. [16] in polycaprolactone (PCL), Sun et al. [17] in low-density polyethylene (LDPE), Keshari et al. [18] in sodium alginate (SA). Standard in these studies is the feasibility of associating tocopherol to different polymeric matrices, generating active films/coatings.

The tocopherol mix was used in Wu et al. [19], which developed modified starch/sodium alginate films with tocopherol mix of 70 % concentration and was applied at 2 % concentration on filmogenic solution—however, no studies associated cassava starch/carboxymethyl cellulose with this antioxidant. Therefore, the aim was to evaluate the technological properties of cassava starch/carboxymethyl cellulose blend-based films added to the soy lecithin and tocopherol mix.

2. Materials and methods

The materials used in the formulation of the blend-forming solutions in this study were food grade: carboxymethyl cellulose (lot 25249 Arcolor, São Paulo, Brazil), cassava starch (lot HW294, Amafil, Cianorte, PR, Brazil), sorbitol in solid form (powder), (lot 250FU46, Ingredientes on-line, São Paulo, Brazil), Soy lecithin (lot 2021105, Ingredientes on-line, São Paulo, Brazil), and tocopherol mix (mix of tocopherols - α-tocopherol: 10–20 %; β -tocopherol: 1–3 %; γ-tocopherol: 45–65 %; δ-tocopherol: 12–26 %) (Vonplex E® L., SP, Brazil).

2.1. Formulations and preparation of the blend-forming solutions

The blend-forming solutions were prepared according to a methodology adapted from Wu et al. [19] and Tongdeesoontorn et al. [20]. Briefly, the cassava-starch solution and sorbitol were prepared by stirring (Quimis Q261-22, Diadema-SP, Brazil), raising the temperature from 20 to 70 °C gradually, and maintaining at 70 °C for 10 min to induce starch gelatinization. Separately, the carboxymethyl cellulose, tocopherol mix, and soy lecithin as an emulsifier were mixed at concentrations according to Table 1 at 45 °C, and the starch-solution was added to this mixture under constant stirring until complete homogenization. Then the formulations were centrifuged in a centrifuge (Quimis Q222, Diadema-SP, Brazil) for 5 (5 min) to remove air bubbles. The formulations were defined by laboratory pre-tests and were named B, L, LT, e LT2.

The film-forming dispersions (72 g) were cast on glass dishes (256 cm^2) and dried at 45 °C in an oven (Lucadema Científica, 80/336, São José do Rio Preto/SP, Brazil) for 24 h. After drying, the films were conditioned in a desiccator containing magnesium nitrate pentahydrate, with a relative humidity of 53 %.

2.2. Characterization tests of the developed films

2.2.1. Scanning electron microscopy (SEM)

The microstructure of the films B, L, LT e LT2, measuring 12 mm in diameter, was determined using a scanning electron microscope (TESCAN, VEGA3, France). The samples were sprayed with gold coating at 15 mA for 60 s. The digital images of the cross-sectional structure and film compatibility were observed at 5-kV acceleration voltage. Sequences of images were recorded at magnifications of 1 to 4x.

2.2.2. Thickness, water solubility, water content, film weight, contact angle

The thickness (mm) of films B, L, LT, and LT2 was measured with a digital pachymeter (MTX®, 316119, China) with an accuracy of 0.01 mm. Ten measurements were carried out, three in the center and six in opposite positions of the films [13].

The water solubility of films B, L, LT, and LT2 was determined according to Gontard et al. [21]. Film samples in triplicate measuring 2 cm in diameter with known weight were immersed in 50 mL of distilled water and subjected to agitation in an incubator shaker (Cienlab, CE-320, Campinas, SP, Brasil) at 60 rpm for 24 h at 25 °C. The film samples were dried in an oven (Lucadema Científica, 80/336, São José do Rio Preto, SP, Brazil) at 105 °C for 24 h to determine the resulting material (not solubilized), and water solubility was calculated using Equation (1).

$$Water\ solubility\ (\%) = [(Wi - Wf) / Wf] * 100 \quad (1)$$

where: Wi is the initial weight of the film sample before drying (g), and Wf is the final weight after drying (g).

The gravimetric method determined the water content (WC) of films B, L, LT, and LT2. The samples with 2 cm diameter were weighed in an analytical balance (Shimadzu, AUY 220, Japan). They were then heated at 105 °C in an oven (Lucadema Científica, 80/336, São José do Rio Preto/SP, Brazil) and weighed until constant mass. After cooling the samples, the final masses were recorded. WC values (%) were determined using four repetitions for each sample, according to Equation (2).

$$WC\ (\%) = [(Wi - Wf) / Wf] * 100 \quad (2)$$

where: Wi is the initial weight of the film sample before drying (g), and Wf is the final weight after drying (g).

The films B, L, LT, and LT2 weight was determined according to Sobral [22] using the film's weight and area and calculated using Equation (3).

$$Weight\ (g / m^2) = 10000 * (w / a) \quad (3)$$

where: w is the film weight (g), and a is the film area (cm^2).

A contact angle measuring instrument (Attension, Theta Lite Optical

Table 1
Blend-forming solution used for preparing cassava starch/CMC-based films added of soy lecithin and tocopherol mix.

Blend-Forming Solution	Cassava starch (g)	Carboxymethyl cellulose (g)	Sorbitol ($g.g^{-1}$ of the cassava starch)	Soy lecithin ($g.g^{-1}$ of the tocoferol mix)	Tocopherol mix ($g.g^{-1}$ of the cassava starch)	Water (g)
B	3	1.5	0.2	0	0	100
L	3	1.5	0.2	0.0030	0	100
LT	3	1.5	0.2	0.0015	0.005	100
LT2	3	1.5	0.2	0.0030	0.010	100

Tensiometer) and software for the calculation (Attension Theta (Versão 4.1.9.8) were used to measure the contact angle of water on the surface of the film B, L, LT, and LT2. 5 μl distilled water was dropped on the film's surface using a microsyringe. Once the water droplets touched the specified baseline, the image was collected at a rate of 25 frames per second for 10 s. Five points on the image were taken, and the angle was curved. Each sample was measured five times.

2.2.3. Permeability test

Water vapor permeability (WVP) of the films B, L, LT, and LT2 was measured using film cut into pieces and, later, fixed on polycarbonate permeation cells (8.0 × 2.7 cm) containing silica gel and sealing with paraffin after placing this film into a desiccator, where the relative humidity was maintained at 100 % using distilled water. This apparatus was kept in a bacteriological incubator (347 CD, Fanem, Brazil) at 25 ± 0.5 °C, according to the standard method E96/E96M − 16 [23]. The mass of films was followed daily for 7 days, and WVP was calculated using Equation (4).

$$WVP\left(g\ mm\ h^{-1}cm^{-2}Pa^{-1}\right) = (w/t) * (x/A.\Delta p) \quad (4)$$

where: w is the water loss from the permeation cup (g), t is the time of analysis (h), x is the thickness (mm), A is the film area (m^2), and Δp is the difference in water vapor pressure between the inside and outside of the cup (Pa).

The oil permeability, an indication of the lipid barrier property of the films, was determined according to Tang et al. [24]. Tubes with 5 mL of soybean oil were sealed with a layer of developed films B, L, LT, and LT2 (diameter = 4 cm), followed by filter paper, and conditioned at 40 % RH and 25 °C. The filter paper was weighed daily over 7 days, and the oil permeability coefficient (OPC) was calculated using Equation (5).

$$OPC\ \left(g.mm.d^{-1}\right) = (\Delta W.FT/A.t) \quad (5)$$

where: ΔW is the variation of weight with time (g), FT is the average of film thickness (mm), A is film permeation area (mm^{-2}), and t is the time (days). Curves were generated using linear regression to estimate the weight gain over time (W/T).

2.2.4. Mechanical tests

Film samples B, L, LT, and LT2 (1.5 × 15 cm) of each treatment (n = 15) were conditioned for 1 or 5 weeks at 25 °C and RH of 53 %. Their mechanical behavior was evaluated using equipment from Instron (EMIC DL 500, Brazil) to determine the tensile strength (TS) (MPa), the elasticity modulus (EM) (MPa), and the elongation until shear (ES) (%) of the films. The samples were fixed in the base, and a traction force was realized at least 50 mm until rupture. The puncture of the films samples measuring 6.5 cm diameter of each treatment (n = 15) was conditioned for 1 or 5 weeks at 25 °C and RH of 53 %. Their mechanical behavior was evaluated using equipment from Instron (Instron, 3367, Grove City, PA, USA) according to the standard method D882-12 [25].

2.2.5. Antioxidant activity of the films

Antioxidant activity was evaluated using DPPH• (1,1-diphenyl-2-picryl-hydrazyl) radicals of developed films B, L, LT, and LT2 was determined according to Mensor et al. [26]. The absorbance was measured using a UV/VIS spectrophotometer (Bel Engineering UV-M51, Monza, Italy) at 520 ηm, and the total antioxidant activity was calculated using Equation (6).

Ethanol was used as a blank, and the extraction solution was used as a control.

$$AA(\%) = 100 - [(ABSsample - ABSwhite)/ABScontrol]. * 100 \quad (6)$$

2.2.6. Biodegradable test

Biodegradation of films B, L, LT, and LT2 measuring 3 cm × 1 cm was evaluated by the composting system, according to the standard method G-160-03 [27] standard. The samples were placed at a depth of 5 cm under previously prepared soil (pH between 6.5 and 7.5 and moisture content between 20 and 30 %) and conditioned in a bacteriological incubator (347 CD, Fanem, Brazil) at 30 °C with 95–100 % relative humidity. They were visually observed until their decomposition.

2.2.7. Color parameters

The instrumental color parameters of films B, L, LT, and LT2 were measured using a colorimeter (Konica Minolta Sensing IC., CR-400, Sakai, Japan). Three films of each treatment were evaluated; the parameters considered were L* lightness (light to dark variation), a* chromaticity (chromaticity on the green to red color axis), b* chromaticity (chromaticity on the blue to yellow color axis) and the saturation (C*-chroma) and Hue (h°).

The transmittance of films B, L, LT, and LT2 was determined in Transmission Meter equipment (Linshang, LS-162, China). Measurements were performed in three positions for the same film, center, and two sides. The following were quantified: ultraviolet radiation rejection value, in the wavelength range of 100–400 nm; and of the infrared, >780 nm and, the visible light transmission value.

2.2.8. Thermogravimetry analysis – TGA

The TG curves of the B, L, LT, e LT2 films were obtained in a TGA-50 thermal analyzer (Shimadzu ®, Kyoto, Japan). One sample of each film with mass between 2 and 4 mg was heated from 25 °C to 500 °C using a heating rate of 10 °C.min^{-1}. The measurements were performed under nitrogen atmospheres (N_2) and a flow rate of 60 mL min^{-1}.

2.2.9. Fourier transform infrared spectroscopy

The functional groups in the films were determined by infrared spectroscopy with an Agilent Technologies Spectrophotometer (CARY 630 FTIR, Santa Clara, USA) with a resolution of 4 cm^{-1} over 32 scans. For the analysis, the samples of films B, L, LT, and LT2 were submitted to drying at 45 °C for 20 h and grinding individually to homogeneity. The spectral range was 4000 to 600 cm^{-1}.

2.2.10. Statistical treatment of data

Initially, the homoscedasticity of the data was checked by the Levene test and evaluated normality test Shapiro-Wilk. For variance analysis and comparison of means of thickness, water solubility, water content, film weight, contact angle, antioxidant activity, permeability test, mechanical tests, and color parameters of the films the Tukey's test was performed ($p \leq 0.05$) using the statistical program Jamovi 2.3.26 [28]. The TGA graph was generated using the OringinPro 2019b software. The FTIR data were processed using SpectraGryph 1.2.16.1 - spectroscopy software [29]. The Principal Component Analysis (PCA) was performed to analyze the interdependence and variability of the results obtained, a Principal Component Analysis (PCA) was performed, and to analyze the similarity of the results obtained, a Hierarchical cluster analysis (HCA) was performed to analyze the similarity of the results obtained. The software (Past4.03) was used for the analysis considering two principal components for PCA from the analysis performed generated the graph Biplot and a dendrogram of clusters graph was generated for HCA [30].

3. Results and discussion

Fig. 1 shows the surface micrographs of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Fig. 1 shows the surface images of the microstructure of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), LT and LT2 (with soy lecithin and tocopherol mix). Air bubble marks can be seen on the surface of film B. These bubbles were greatly minimized by the centrifugation process carried out during the production of the films. However, it is possible to

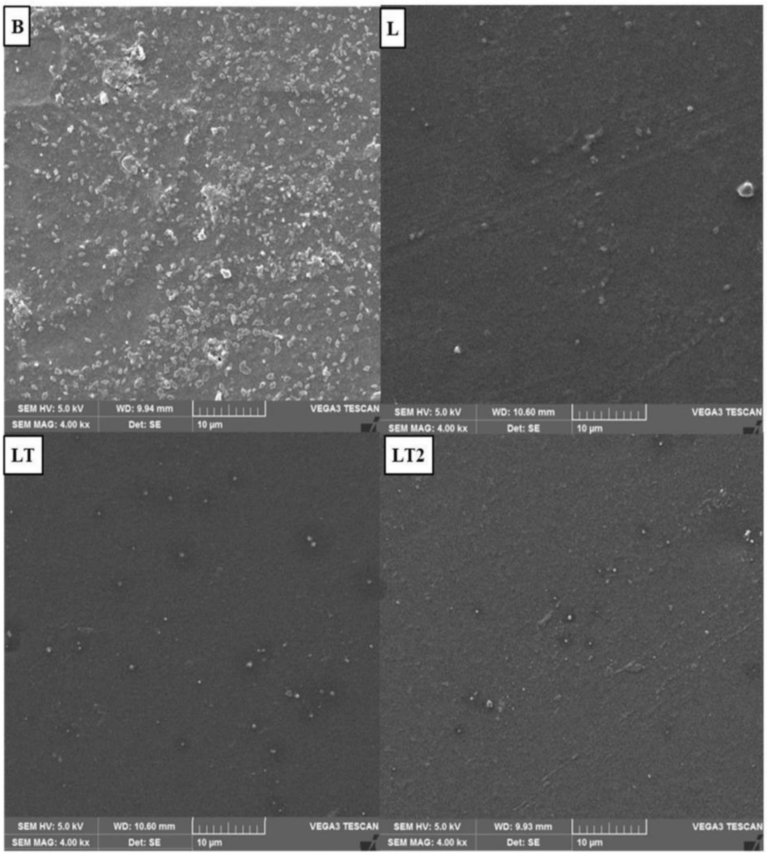

Fig. 1. Surface micrographs of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

see that many remained in the micrographs of this film. It is indicated that the addition of the soy lecithin mix contributed to the formation of a denser structure, as did the tocopherol mix added to the films, as the structure of the films was positively affected, with films L, LT, and LT2, showing a denser, smoother structure, with no cracks than the others, indicating the homogeneity of the polymer. This result was different from the surface in the work by Mirzaei-Mohkam et al. [31], who developed CMC films with α-tocopherol nanocapsules and obtained porosity in the samples after adding α-tocopherol nanocapsules.

Table 2 show mean ± standard deviation of the thickness, water solubility, water content, film weight, contact angle, water vapor permeability, and oil permeability coefficient of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

In Table 2, it was observed that all the parameters evaluated showed a significant difference by Tukey's test ($p \leq 0,05$), except the contact angle of films B and LT. The film with a higher concentration of

Table 2
Mean ± standard deviation of the thickness, water solubility, water content, film weight, contact angle, water vapor permeability, and oil permeability coefficient of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

Films	Thickness (mm)	Water solubility (%)	Water content (%)	Film weight (g/m^2)	Contact angle (θ) (°)	Water vapor permeability (WVP) ($g.mm.h^{-1}.m^{-2}.kPa^{-1}$)	Oil permeability coefficient (OPC) ($g.mm.d^{-1}$)
B	0.0887 ± 0.001d	27.69 ± 0.01c	14.15 ± 0.004a	179 ± 3.75a	56.2 ± 4.0a	0.735 ± 0.01c	21.3 ± 0.3d
L	0.0907 ± 0.001c	27.15 ± 0.004d	13.79 ± 0.002b	162 ± 3.25c	41.6 ± 6.6b	0.716 ± 0.02d	26.5 ± 0.4a
LT	0.1025 ± 0.002b	29.03 ± 0.002b	13.11 ± 0.002c	168 ± 2.00b	56.2 ± 5.5a	0.833 ± 0.02b	23.7 ± 0.5c
LT2	0.1057 ± 0.002a	45.59 ± 0.01a	10.33 ± 0.002d	150 ± 0.86d	40.0 ± 4.7c	0.873 ± 0.01a	24.2 ± 0.06b
p-Value homoscedasticity	0.461	0.142	0.318	0.208	0.266	0.206	0.726
p-Value ANOVA/ Welch	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5 % level.

tocopherol mix (LT2) showed higher water solubility, lower water content, and weight, making it more prone to disintegration when submerged in water and more water-soluble.

The Mirzaei-Mohkam et al. [31] that developed CMC films added nanocapsules of α-tocopherol obtained thicknesses ranging from 0.08 to 0.097 mm, resembling the results of the present study. Boonthod and Wacharawichanant [32] developed films of polyvinyl alcohol/-starch/carboxymethyl cellulose added citric acid and observed water solubility ranging from 33 to 50 %, values close to the present study. Vasco et al. [33] developed edible films of carboxymethyl cellulose, cassava starch, or alginate. They obtained 14.5 and 28 % water content for cassava starch and CMC films, respectively, and 22.8 and 85 % water solubility for cassava starch and CMC films, respectively. The films developed based on cassava starch had the closest results to the present study.

The contact angle of all films was below 60°, films L and LT2, which had a lecithin concentration of 0.0030 g g^{-1} in their composition, showed higher hydrophilicity, indicating that this surfactant concentration made these formulations more sensitive to environmental humidity. That is, the concentration of soy lecithin used in L and LT2 caused an amphiphilic character characteristic of this emulsifier [34], while the concentration of emulsifier used in LT was not so significant as to increase the hydrophilicity of this film. The results characterize all films as hydrophilic, but the L and LT2 were more hydrophilic than B and LT.

Tavares et al. [35] developed a blend of corn starch with CMC and obtained a 56–61° of contact angle, a value close to the present study's. Vasco et al. [33] developed edible films of carboxymethyl cellulose, cassava starch, or alginate and obtained 73.9 and 59.3° of contact angle for cassava starch and CMC films, respectively; the films developed based on CMC had the closest results to those of the present study.

The WVP of the LT and LT2 films added with the tocopherol mix showed the highest values; this is probably because, during the production process, the higher concentration of the antioxidant in the LT2 film may have broken the –OH group molecules present in this antioxidant, opening spaces in the molecule, leaving it more exposed to interaction with water, which may explain the higher value for water vapor permeability, which occurred not only in the sample with maximum tocopherol mix concentration but also in the one with the minimum concentration (LT). Santos et al. [12], in work about films of gum arabic, carboxymethyl cellulose, and sodium alginate added to anise essential oil, observed values of 0.259 for films only CMC and 0.629 g mm $h^{-1}.m^{-2}.kPa^{-1}$ for of gum arabic and carboxymethyl cellulose films added of anise essential oil, lower than the values found in this study.

For oil permeability (Table 2), the sample (L), which had lecithin in its composition, promoted greater exposure to oil permeability; this may have occurred due to the lecithin molecule having fatty acids and phospholipids that make this film more lipophilic. While the samples added of tocopherol mix (LT and LT2) (lipidic) and in its composition contained lecithin, this emulsifier interacted with the tocopherol mix, promoting some protection to the oil permeability, indicating that films added of tocopherol mix maintained a denser structure which minimized oil penetration.

Table 3 shows the puncture, tensile strength, elongation until shear, elasticity modulus, and antioxidant activity of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

For the mechanical properties (Table 3), it was observed that all the parameters evaluated showed significant differences by Tukey's test ($p \leq 0.05$), except for puncture, where there was no significant difference between films B and LT2 and L and LT. When evaluating the puncture results, it is observed that all films were sensitive to tearing, with films B and LT2 being slightly more resistant.

In TS, the films added with soy lecithin were superior to film B, but film L showed the highest value, demonstrating greater tensile strength in this parameter, i.e., the films added with the tocopherol mix were only more resistant than the control film. This can be attributed to the microstructure of the films, as it was shown that the films containing soy lecithin in their formulation were more cohesive than film B, because as was demonstrated in Fig. 1, these films showed a denser, smoother structure, with no cracks than film B, indicating the homogeneity of the polymer, which contributed to greater tensile strength.

Yao et al. [36], in the study with the synthesis of carboxymethyl cellulose extracted from various vegetable sources, developed films and observed TS values of 15.81–28.97 MPa, values close to those found in the present study. The same author determined ES in the films and obtained values ranging from 3.5 to 6.60 %, close to those found when evaluating cassava starch/CMC in films added to the tocopherol mix.

In the modulus of elasticity, the films L, LT, and LT2 were more malleable than the control film (B), as they showed a greater capacity to be stretched, possibly because they contain lecithin in their composition, which acted by promoting good interaction between the polymeric matrix and tocopherol mix. In the work of Mirzaei-Mohkam et al. [31] that developed CMC films added nanocapsules of α-tocopherol for the parameter of elongation until shear, the authors found 35–53 %, higher than those observed in the films under study. In contrast, for the parameter of elasticity modulus, the authors observed 40–80 MPa lower than those obtained in this study.

The work by Tavares et al. [35] obtained results of 4.7–32.6 MPa for tensile strength values close to those found in this work; for the parameter of elongation until shear, the authors found 21.2–64.8 %, higher than those observed in the films under study, while for the parameter of elasticity modulus, the authors observed 40.3–295.6 MPa lower than those obtained in this study.

Table 3
Mean ± standard deviation of the puncture, tensile strength, elongation until shear, elasticity modulus, and antioxidant activity of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

Films	Puncture (%)	Tensile strength (TS) (MPa)	Elongation until shear (ES) (%)	Elasticity modulus (EM) (MPa)	Antioxidant activity (by DPPH• method) (%)
B	0.09 ± 0.001a	24.5 ± 0.001d	4.01 ± 0.01d	118 ± 1.53d	0.37 ± 0.02d
L	0.08 ± 0.001b	43.3 ± 0.14a	5.28 ± 0.13a	519 ± 9.94a	8.48 ± 0.36c
LT	0.08 ± 0.001b	32.8 ± 1.0b	4.58 ± 0.17c	424 ± 27.10c	46.42 ± 0.46b
LT2	0.09 ± 0.001a	32.4 ± 0.43c	5.22 ± 0.006b	463 ± 26.42b	74.29 ± 1.2a
p-Value homoscedasticity	0.717	0.109	0.256	0.159	0.137
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5 % level.

When evaluating the antioxidant activity by the DPPH• method (Table 3), it can be observed that the films LT and LT2 showed good antioxidant activity, 46.42 and 74.29 %, respectively. This was expected since the tocopherol mix was added to the formulations of these films, but it was necessary to verify by testing whether the antioxidant was maintained after the heating process during the production process and the second heating during the oven drying. The results prove that the developed films added to the tocopherol mix have the potential for use in the active packaging of foods because they contain bioactive in their structure.

Fig. 2 shows the results of the biodegradability test of the films of cassava starch/CMC control B (no soy lecithin and tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

Rapid decay occurred in the films. Visually it seems a strange image, but images were selected that could provide an overview of what was observed in the biodegradability test of the films under study (Fig. 2). All samples on day zero were placed in the test condition; on first day it was observed that all had absorbed moisture from the material on which they were deposited, and presenting a gummy aspect, soft and with plenty of adhered material; on the second day it was observed that the samples began to disintegrate; on the third day, there was greater disintegration, probably due to the action of the microorganisms contained in the composting material and on the fourth day no traces of the films were found in the middle of the composting system. This indicates that all the cassava starch/CMC films developed in this study are highly biodegradable.

Table 4 shows the color parameters of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

When evaluating the color parameters (Table 4), it was observed that all the parameters evaluated showed significant differences by Tukey's test ($p \leq 0.05$). In the luminosity parameter, all samples tended toward white, i.e., clear. The chroma values were low (achromatic), revealing weak or diluted coloration; it is clear when observing the chromaticity parameters a* and b*, while the hue angle reveals that all the films had a slightly greenish-yellow tonality. In the work of Mirzaei-Mohkam et al. [31] that developed CMC films added nanocapsules of α-tocopherol obtained L*, a*, and b* ranging from 93.73 to 94.98, −0.59 to −0.23 and 2.55 to 4.88, respectively, approximate to the results of the present study.

Although there was a significant difference in all parameters, as shown by Tukey's test, the films containing the highest concentration of soy lecithin (L and LT2) showed close numerical values for most of the color parameters. In the article by Nilsuwan et al. [37] that evaluated the effects of soy lecithin levels and microfluidization conditions on the properties of fish gelatine-based films incorporated with palm oil observed a decrease in the L and a parameters, with samples containing 50 and 75 % (w/w, based on palm oil) obtaining ranges of 87.14–87.30 for the L* parameter in films containing 50 % soy lecithin and 85.01 to 85.18 in films containing 75 % soy lecithin, while for a* and b* there was an increase, in the a* parameter the values varied between −1.82 and −1.78 for the films with 50 % soy lecithin and −1.49 and −1.45 for the films with 75 % soy lecithin and in the b* parameter in the films with 50 % lecithin 13.09 to 13.24 and films with 75 % lecithin 15.04 to 15.11, different from the behavior observed for the films developed in the present study, since the films (L, LT and LT2) to which soy lecithin was added showed lower values for L*, a* and c* than the film without soy lecithin and tocopherol mix (B).

Table 5 shows the transmittance parameters of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

When evaluating the transmittance parameters (Table 5), it was observed that all the parameters evaluated showed significant differences by Tukey's test ($p \leq 0.05$). VLT shows that the LT and LT2 films showed about 21.3 and 21.1 % barrier, while the B and L films did not block even 20 % of the visible light passage. The same behavior occurred for UVR and IRR, with only the films added of tocopherol mix reaching barrier levels above 30 % for ultraviolet radiation and above 25 % for infrared radiation. The transmittance parameters indicate that the films added tocopherol mix (LT and LT2) were more opaque than films B and L.

Wang et al. [38] developed CMC-based films and observed approximately 10 % visible light barrier, lower than the results of cassava starch/CMC films added to the tocopherol mix. In the work of Mirzaei-Mohkam et al. [31], that developed CMC films, added nanocapsules of α-tocopherol obtained a 20 % barrier to ultraviolet light, lower than the results found in the present study. Agudelo-Cuartas et al. [14] developed films based on whey protein incorporated with natamycin and nanoemulsified α-tocopherol. They observed approximately

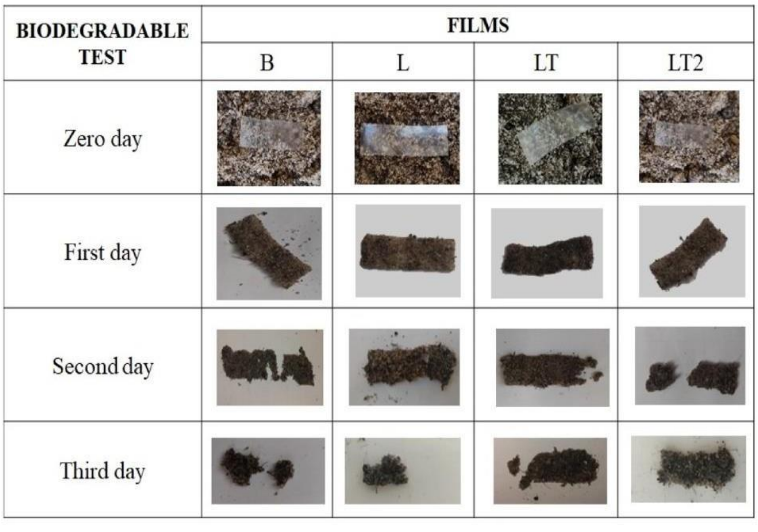

Fig. 2. Biodegradable test results of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

Table 4
Mean ± standard deviation of the color parameters of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Films	Luminosity (L*)	Chromaticity (a*)	Chromaticity (b*)	Chroma (C*)	Hue (h°)
B	92.8 ± 2.03a	−0.52 ± 0.02a	4.17 ± 0.55a	4.29 ± 0.01a	97.3 ± 0.24d
L	92.2 ± 1.52b	−0.57 ± 0.05b	4.06 ± 0.47b	4.01 ± 0.05c	98.5 ± 0.15c
LT	89 ± 3.39d	−0.73 ± 0.02d	3.54 ± 0.51c	3.68 ± 0.08d	102.2 ± 0.21a
LT2	91.3 ± 0.98c	−0.69 ± 0.02c	4.17 ± 0.45a	4.20 ± 0.01b	99.7 ± 0.11b
p-Value homoscedasticity	0.394	0.075	0.138	0.058	0.392
p-Value ANOVA/Welch	<0.001	<0.001	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5 % level.

Table 5
Mean ± standard deviation of the transmittance parameters of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), and LT and LT2 (with soy lecithin and tocopherol mix).

Films	Visible light transmission - VLT (%)	Ultraviolet radiation rejection - UVR (%)	Infrared radiation rejection - IRR (%)
B	82.8 ± 0.1a	19.1 ± 0.01d	20.3 ± 0.27d
L	81.1 ± 0.04b	28.6 ± 0.0001c	22.4 ± 0.16c
LT	78.7 ± 0.18d	31.8 ± 0.08b	27.6 ± 0.51a
LT2	78.9 ± 0.15c	33.4 ± 0.02a	25.4 ± 0.5b
p-Value homoscedasticity	0.078	0.188	0.289
p-Value ANOVA/Welch	<0.001	<0.001	<0.001

*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5 % level.

30 % barrier to ultraviolet light, similar to the results found in the present study.

Foods, in general, can suffer several alterations due to the incidence of light, such as loss of vitamins, unpleasant smell, and changes in coloration, besides lipid oxidation. Oxidative processes occur mainly in foods with high unsaturated lipid content; such processes can be initiated by free radicals driven by oxygen and/or light [39]. Opaque films can be used for the protection of photosensitive food products. Thus, the films added with a tocopherol mix of LT and LT2 may be applied to protect light-sensitive foods and be a great ally in preventing lipid oxidation.

Fig. 3 shows the TGA and DTGA thermograms of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix), LT and LT2 (with soy lecithin and tocopherol mix).

When evaluating the TGA and DTG, it was observed that all the parameters evaluated showed significant differences by Tukey's test ($p \leq 0.05$). The TGA curves (Fig. 3 - a) show the first stage of mass loss close to 95 °C, referring to the loss of water from the sample, the second stage from 140 to 160 °C, referring to sorbitol decomposition, and the third stage from 272.74 to 313.38 °C referring to the degradation of starch and carboxymethyl cellulose. The same behavior was observed in other studies, which observed losses of 250–300 °C [35,40,41].

This result (Fig. 3 - a) indicates the films that had lecithin in their composition, i.e., L, LT, and LT2, showed a more pronounced mass loss, as it is possible to verify that near 300 °C film B had lost approximately 5 %, while L, LT, and LT2 had already lost 10 % mass, a difference probably linked to the degradation of soy lecithin. Demir et al. [42] determined the TGA of soy lecithin and observed that the decomposition of lecithin started around 200 °C and continued up to 500 °C. It can be observed that lecithin played an important role in the thermal degradation that occurred in the films.

In addition, adding a tocopherol mixture in the formulations slightly altered the degradation temperature of the polymeric matrix of the LT film and strongly altered the degradation temperature of the LT2 film. Tocopherol is stable at room temperature but is easily oxidized at high temperatures, under light, or in an alkaline environment, and generally between 160 and 190 °C, vitamin E is oxidized [14]. In addition, it is a compound that contains a large number of hydroxyl groups, and possibly, with the increase in temperature, it decomposed and evaporated, explaining this behavior, which occurred mainly in the LT2 film. The same behavior was observed by Jiang et al. [43] in a study of Poly (lactic) acid-poly(3-hydroxybutyrate-*co*-4-hydroxybutyrate) (PLA-PHB) films; the film to which tocopherol was added showed a greater weight loss than the others.

The degradation temperatures of the films (Fig. 3 - b) were close to 290 °C, the films containing soy lecithin showed lower degradation temperatures than film B. Film LT2 showed the lowest temperature of

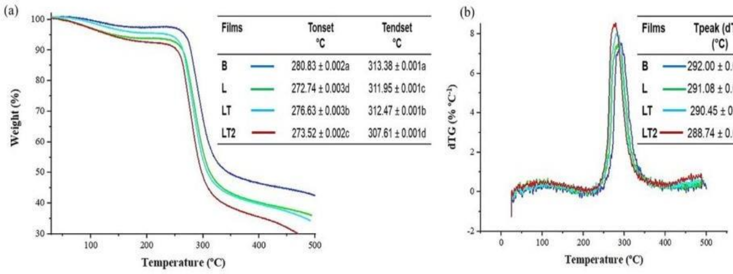

Fig. 3. (a) TGA and (b) DTGA thermograms of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).
*Means followed by the same lowercase letter in each column did not differ significantly by Tukey's test at the 5 % level.

288.74 °C, corroborating the immense contribution mentioned during the discussion of the TGA curve, which was attributed to the degradation and evaporation of the antioxidant. The values found in this study were lower than those observed by Zahiruddin et al. [41] in cassava starch films around 310 °C.

Fig. 4 shows the Fourier transform infrared spectroscopy of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

The FTIR curves (Fig. 4) indicate similarity in all films but differences in intensity. The FTIR spectra show that all films presented bands characteristic of polysaccharide films; one can observe the band 3243 cm^{-1}, typical of the stretching vibrations of the –OH group [44], occurs typically in the region between 3600 and 3200 cm^{-1}; one can also observe the band 2874 cm^{-1}, in the region between 3000 and 2800 cm^{-1}, represents the stretching vibration of the –CH group, other bands from 1370 to 1265, 1093 and 941 cm^{-1} observed in the carbohydrate region typically expressed by the bands in the region of 750–1300 cm^{-1} [8]. The distinct peak at 941 cm^{-1} was also related to $–CH_2$ ring vibration and C = C stretching vibration, respectively, proven by the results reported by Tongdeesoontorn et al. [20]. These bands are characteristic of starch, meaning that the functional groups of this polysaccharide have remained in the film.

In addition, the band was observed in the region of 1541 cm^{-1}, assigned vibrational bands related to the symmetric carboxylate (COO-) stretches (1547-1480 cm^{-1}) [45–47], characteristic carboxymethyl cellulose band. It can be observed that the addition of tocopherol mix did not cause changes in the functional groups nor the nature of the chemical bonds of the compounds but contributed to intensifying them, as in all samples, bands characteristic of the polymer matrix were observed.

Fig. 5 shows the hierarchical cluster (HCA) and biplot graphic of principal component analysis (PCA) of properties of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

It is observed in Fig. 5 (a) that the clusters formed by the Euclidean distance show the formation of three clusters and demonstrate the similarity, especially between LT and LT2 films, proven by the principal component analysis Fig. 5 (b). It is observed that the variability explained by means of PCA (Fig. 5 - b) (PC 1 = 59.66 % and PC 2 = 25.36 %, respectively) is 85.02 %; it is the variability of the whole data set.

The films showed well-differentiated results; CA and PCT more positively influenced film B, and this film had more weight from the values of these parameters; film L was positively influenced by the parameters VLT and Tpeak, indicating these parameters had more weight to explain the PCA. For the films added of tocopherol mix, the LT and LT2 films were more negatively influenced by the VLT and Tpeak and positively influenced by the WS, WVP, IRR, and AA parameters; this result shows that the films added with tocopherol mix enabled have higher results of these variables.

That is, the PCA was important to show that the films added of tocopherol mix (LT and LT2) could be indicated as antioxidant films for food protection, but by the characteristic of being very soluble, it could be applied as an edible film or even an edible coating. In addition, PCA makes it possible to visualize which technological parameters had more weight for each film; this tool also serves as an option to project future films with predetermined attributes.

4. Conclusions

The films that contained soy lecithin in their composition (L, LT, and LT2) were strongly influenced by the presence of this emulsifier; these films had a denser microstructure and were also more hydrophilic than the control film B, but the LT and LT2 films added with tocopherol mix were slightly yellow-green. In addition, these films are biodegradable and antioxidant. LT showed a greater barrier to infrared radiation, and LT2 showed a greater barrier to visible light and ultraviolet radiation, being more opaque than the other films. PCA showed that the LT and LT2 films maintained their bioactivity and are strongly recommended for use in active food packaging. Further research is needed. The first suggestion would be to apply the films with a mix of tocopherols as active packaging for food products, and a second suggestion would be to apply them as an active edible coating.

Author statement

Danusa Silva da Costa: Conceptualization, Methodology/Study design, Formal analysis, Investigation, Resources, Writing – original draft, Writing – review and editing, Visualization.

Roseane Maria Ribeiro Costa: , Formal analysis.

Katiuchia Pereira Takeuchi: Conceptualization, Methodology/Study design, Formal analysis, Investigation, Resources, Writing – original draft, Writing – review and editing, Visualization, Project administration.

Alessandra Santos Lopes: Conceptualization, Methodology/Study design, Formal analysis, Investigation, Resources, Writing – original draft, Writing – review and editing, Visualization, Project administration, Funding acquisition.

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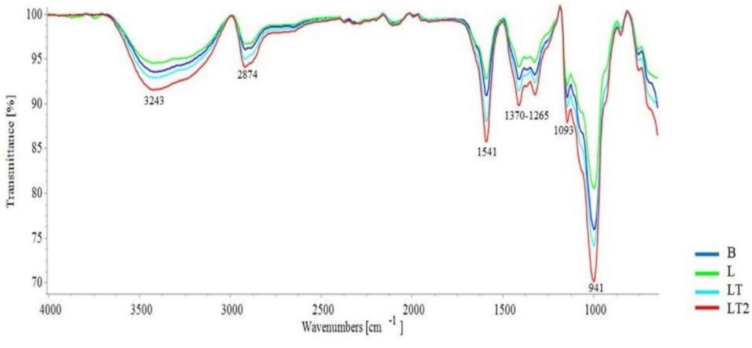

Fig. 4. Fourier transform infrared spectroscopy of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

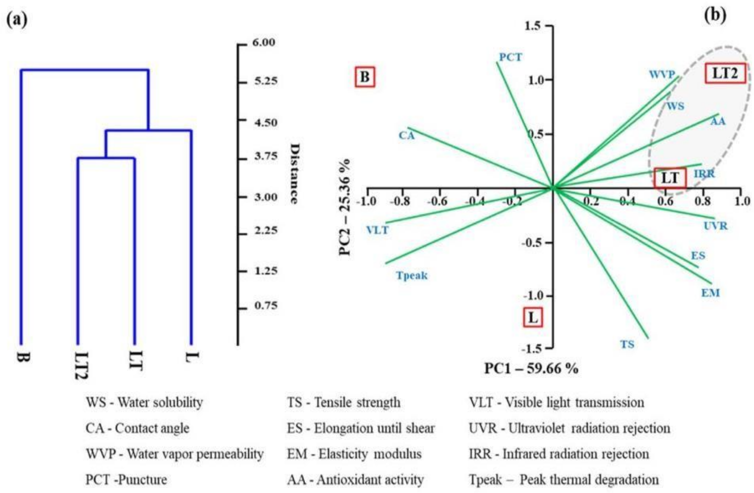

Fig. 5. (a) Hierarchical cluster analysis (HCA) and (b) Biplot graphic of principal component analysis (PCA) of the loadings and scores of PC1 and PC2 of properties of the films of cassava starch/CMC control B (no soy lecithin and no tocopherol mix), L (with soy lecithin and no tocopherol mix) and, LT and LT2 (with soy lecithin and tocopherol mix).

UFPA– Edital PAPQ 2023).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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ANEXO C – Atestado de regularidade de acesso emitido pelo sistema nacional de gestão do patrimônio genético e do conhecimento tradicional associado.

Ministério do Meio Ambiente
CONSELHO DE GESTÃO DO PATRIMÔNIO GENÉTICO

SISTEMA NACIONAL DE GESTÃO DO PATRIMÔNIO GENÉTICO E DO CONHECIMENTO TRADICIONAL ASSOCIADO

Atestado de Regularidade de Acesso

Cadastro nº A9659A8

O Conselho de Gestão do Patrimônio Genético, no exercício da competência conferida pelo inciso IV do § 1º do art. 6º da Lei nº 13.123/2015, atesta que o acesso ao Patrimônio Genético/CTA, referente ao cadastro abaixo identificado e resumido, cumpriu os requisitos da Lei nº 13.123/2015 e seus regulamentos até a data de emissão deste atestado.

Número do cadastro:	**A9659A8**
Usuário:	**Alessandra Santos Lopes**
CPF/CNPJ:	**423.897.122-15**
Objeto do Acesso:	**Patrimônio Genético/CTA**
Finalidade do Acesso:	**Pesquisa**

Espécie

Bertholletia excelsa

Fonte do CTA

CTA de origem não identificável

Título da Atividade: **Coleta, análise físico-química de castanha-do-Brasil**

Equipe

Alessandra Santos Lopes	**UFPA**
Danusa Silva da Costa	**UFPA**
Katiuchia Pereira Takeuchi	**UFMT**
Kalebe Ferreira Furtado	**UFPA**

Data do Cadastro:	**03/05/2023 12:48:29**
Situação do Cadastro:	**Concluído**

Conselho de Gestão do Patrimônio Genético
Situação cadastral conforme consulta ao SisGen em **17:14** de **25/06/2023**.

SISTEMA NACIONAL DE GESTÃO DO PATRIMÔNIO GENÉTICO E DO CONHECIMENTO TRADICIONAL ASSOCIADO - SISGEN

APÊNDICE A – Publicação obtida ao longo do curso da Tese - revista *Molecules*.
Link de acesso: https://www.mdpi.com/1420-3049/28/16/6069

molecules

MDPI

Systematic Review

Mairá-Potato (*Casimirella* sp.): Botanical, Food, Pharmacological, and Phytochemical Aspects

Danusa Silva da Costa [1,*], Lucely Nogueira dos Santos [1], Nelson Rosa Ferreira [1], Katiuchia Pereira Takeuchi [2] and Alessandra Santos Lopes [1]

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Abstract: Millions of people in the world live in food insecurity, so identifying a tuber with characteristics capable of meeting the demand for food and also identifying active compounds that can be used to minimize harm to human health is of great value. The aim was to carry out a review based on systematic review tools and the main objective was to seek information on botanical, food, pharmacological, and phytochemical aspects of *Casimirella* sp. and propose possible applications. This review showed papers that addressed botanical, food, pharmacological, and phytochemical aspects of the Mairá-potato and presented suggestions for using this tuber allied to the information described in the works found in the Google Academic, Scielo, Science Direct, Scopus, PubMed, and Web of Science databases. This review synthesized knowledge about the Mairá-potato that can contribute to the direction of further research on the suggested technological applications, both on the use of this tuber as a polymeric material and its use as biomaterial, encapsulation, bioactive use, and 3D printing, because this work collected information about this non-conventional food plant (PANC) that shows great potential for use in various areas of study.

Keywords: new starch; non-conventional starch; review; chemistry structure; application hints

check for updates

Citation: Costa, D.S.d.; Santos, L.N.d.; Ferreira, N.R.; Takeuchi, K.P.; Lopes, A.S. Mairá-Potato (*Casimirella* sp.): Botanical, Food, Pharmacological, and Phytochemical Aspects. *Molecules* **2023**, *28*, 6069. https://doi.org/10.3390/molecules28166069

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1. Introduction

One person out of nine does not have enough food for a healthy life. Around 805 million people worldwide are food insecure according to the Food and Agriculture Organization of the United Nations (FAO). This insecurity is due to the poorer classes being unable to obtain enough food to meet an adequate diet [1]. But, in 2022, the FAO reported that in 2021 about 828 million people in the world, approximately 10.5% of the world's population, faced hunger and estimated that by 2030 almost 670 million people are expected to remain undernourished [2]. The discussion around the inability to meet human food demand is increasingly present due to population projections that indicate increased consumption, growth of cities, and restrictions on land exploitation [3].

Root and tuber crops are tropical countries' second largest cultivated species after cereals. They occupy a privileged position in food security because of their high carbohydrate content and calorific value [4]. They can contain various medicinally important bioactive principles found in different parts such as tubers, stems, and leaves; furthermore, tubers serve as different carbohydrate depots [5].

Identifying a plant with constituents that promote health benefits is of great interest. Foods of plant origin have a wide variety of non-nutritive phytochemical compounds. They are synthesized as secondary metabolites and serve various ecological functions in plants [6]. Tubers are significant sources of several compounds, such as saponins,