

**UNIVERSIDADE FEDERAL DE PERNAMBUCO  
CENTRO DE BIOCIÊNCIAS  
PROGRAMA DE PÓS-GRADUAÇÃO EM BIOQUÍMICA E FISIOLOGIA**

KATHARINA MARQUES DINIZ

**AVALIAÇÃO DO POTENCIAL BIOTECNOLÓGICO DE FRUTOS DE *Syagrus schizophylla* M. E SUAS APLICAÇÕES**

Recife  
2021

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Tese de Doutorado apresentado como um dos requisitos para o cumprimento parcial das exigências para obtenção do título de Doutor em Bioquímica e Fisiologia pela Universidade Federal de Pernambuco.

**Área de concentração:** Bioquímica e Fisiologia

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**Co-orientador (a):** Dra. Márcia Vanusa da Silva

Recife  
2021

Catalogação na Fonte:  
Elaine C Barroso, CRB-4/1728

Diniz, Katharina Marques

Avaliação do potencial biotecnológico de frutos de *Syagrus schizophylla* M. e suas aplicações / Katharina Marques Diniz – 2021.

90 f.: il., fig., tab.

Orientadora: Maria Tereza dos Santos Correia

Coorientadora: Márcia Vanusa da Silva

Tese (doutorado) – Universidade Federal de Pernambuco. Centro Biociências. Programa de Pós-graduação em Bioquímica e Fisiologia, Recife, 2021.

Inclui referências e apêndice.

1. Resíduos 2. Sustentabilidade 3. Biodiesel I. Correia, Maria Tereza dos Santos (orient.). II. Silva, Márcia Vanusa da (coorient.) III. Título

363.728

CDD (22.ed)

UFPE/CB-2021-315

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Aprovada em: **23/08/2021**

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Dedico este trabalho a todos que contribuíram com a minha trajetória acadêmica durante esses 11 anos de Universidade Federal de Pernambuco.

## **AGRADECIMENTOS**

Primeiramente a Deus e a espiritualidade amiga que nos mais variados momentos me instruíram com bons pensamentos, vibrações e direcionamentos quanto ao desenvolvimento deste projeto e planos pessoais.

À minha mãe que caminhou durante estes dez anos trazendo leveza nos mais árduos momentos de indecisões, inseguranças e ânsias e vibrando positivamente a cada etapa.

Às professoras Tereza Correia e Márcia Vanusa que me receberam no grupo de pesquisa confiando a mim o desenvolvimento de um projeto. Agradeço imensamente a oportunidade de hoje ter a chance de concluir mais uma importante etapa da minha carreira, que foi pensada e desejada ainda na graduação. Meus sinceros agradecimentos.

Às minhas amigas, Beatriz Coutinho, Gabriela Calixto, Tássia Luana, Rhaissa Ribeiro, Raimunda Eduarda, que em muitos momentos desenvolveram o papel de irmãs, escutaram, motivaram e acolheram não só as dificuldades, mas também as conquistas vibrando comigo e almejando futuros melhores.

Aos meus colegas de laboratório Bruno Veras e Wilka Nascimento, que sem eles esse projeto não existira, me ajudando a solucionar a caminho pelo qual poderia traçar a pesquisa, desenvolvendo debates e ideias, colaboradores diretos e ativos me dando suporte em momentos cruciais.

Aos técnicos e colaboradores Júlio Cesar (Química fundamental) e Camilo (LEAAL-Nutrição) que me receberam com paciência e simpatia para desenvolver etapas cruciais dos experimentos. Indo comigo para a bancada, explicando teoria e prática envolvida nos experimentos. Ao lado deles tirei lições além da rotina laboratorial que levarei comigo em futuras colaborações.

A todos os alunos do laboratório de Produtos Naturais. Os ICs, mestrandos e doutorandos que propiciaram momentos de descontração, leveza, compartilhamento de ideias e conhecimentos, momentos que foram fundamentais no dia a dia no laboratório. Agradeço também ao Sr. João que vibrava comigo a tão esperada quinta-feira e sexta-feira.

À Usina Coruripe - Alagoas, a gerência administrativa e ambiental e demais funcionários pelo fornecimento do material orgânico possibilitando o desenvolvimento do projeto.

Aos grupos colaboradores da Central Analítica da UFPE, CETENE, CRCN-PE e ITEPE que contribuíram não só com a estrutura física, oferecimento de reagentes e equipamentos, mas principalmente com o grupo de técnicos que nos auxiliaram com os mais variados experimentos.

A CAPES pelo suporte financeiro oferecido durante os três últimos anos de doutorado.

Por fim agradeço a todos que estiveram comigo durante os onze anos de UFPE, que contribuíram diretamente e indiretamente no meu desenvolvimento.

A todos meu muito obrigada.

“Todo o Processo deve ser acompanhado de amadurecimento psicológico, de autocompreensão e de entrega em forma consciente.” (Pelo espírito Joanna de Angélis, livro *Em busca da Verdade* por Divaldo Franco)

## RESUMO

Os recursos naturais são considerados os principais insumos para geração de produtos em diferentes setores industriais, desde a produção de combustíveis com o uso do petróleo ou biocombustíveis a partir de biomassas vegetais, assim como extração de bioativos para compor a base de produtos farmacológicos, médicos e alimentícios. O crescente aumento populacional com a continuada exigência na extração dos recursos naturais tem exigido novos redirecionamentos sobre o uso sustentável da matéria prima ambiental. A valorização integral da matéria prima bruta visa o reaproveitamento dos seus insumos dentro do mesmo setor produtivos ou em setores diversos de forma a contribuir para a minimização da extração dos recursos ambientais e aproveitamento integral da matéria prima. As indústrias energéticas, alimentícias e farmacêuticas se destacam entre os setores da indústria de transformação na utilização do meio ambiente para a geração de novos produtos. Os frutos de *Syagrus schizophylla*, também conhecido como Aricurioba, foram empregados neste trabalho como modelo para implementação da valorização integral, considerando as três principais indústrias da transformação no âmbito brasileiro; a indústria energética, a alimentícia e a farmacêutica. Como proposta os frutos foram fracionados em dois principais produtos primários, o óleo vegetal e o resíduo sólido após extração. Ambas as matérias primas foram estudadas quanto as características físico químicas. A partir do óleo foram elaborados dois produtos, o biodiesel B100 e um fotoprotetor solar, o resíduo sólido foi estudo quanto ao seu potencial nutricional. Os resultados observados evidenciam o potencial biotecnológico inerente aos recursos naturais com contribuição em diferentes escalas produtivas que contribuem com o desenvolvimento sócio-econômico-ambiental.

**Palavras-chaves:** Resíduos agroindustriais; Sustentabilidades; Aricurioba; Fotoprotetor; Bioeconomia circular.

## ABSTRACT

Natural resources are considered the main inputs for the generation of products in different industrial sectors, from the production of fuels using petroleum or biofuels from plant biomass, as well as the extraction of bioactives to compose the base of pharmacological, medical and food. The growing population increase with the continued demand in the extraction of natural resources has required new changes in the sustainable use of environmental raw materials. The integral valorization of the raw material aims at the reuse of its inputs within the same productive sector or in different sectors in order to contribute to the minimization of the extraction of environmental resources and full use of the raw material. The energy, food and pharmaceutical industries stand out among the sectors of the transformation industry in the use of the environment to generate new products. The fruits of *Syagrus schizophylla*, also known as Aricuriroba were used in this work as a model for the implementation of integral valorization, considering the three main transformation industries in the Brazilian scope; the energy, food and pharmaceutical industries. As proposed, the fruits were divided into two main primary products, vegetable oil and solid residue after extraction. Both raw materials were studied for physical chemical characteristics. Two products were prepared from the oil, biodiesel B100 and a sunscreen, the solid residue was studied as to its nutritional potential. The results observed show the biotechnological potential inherent to natural resources with contribution at different productive scales that contribute to socio-economic-environmental development.

**Key-words:** Agro-industrial residues; Sustainability; Aricuriroba; Photoprotector; Circular bioeconomy.

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## 1 INTRODUÇÃO

Com diferentes biomas e condições climáticas o Brasil é considerado um país de elevado poder biotecnológico natural (SÁNCHEZ *et al.*, 2018), uma vez que detém material gerador de economia local e nacional a serem aplicados pela indústria de transformação que é classificada como toda e qualquer operação que trabalha com a matéria prima bruta para realizar seu manufaturamento ao longo da cadeia produtiva gerando um produto primário. Com PIB anual de R\$ 6,8 trilhões em 2018 a indústria de transformação foi responsável por gerar ao governo brasileiro R\$ 2.705 bilhões em produtos (ABIA), sendo as indústrias de alimentos, biocombustíveis e farmacêutico os principais setores responsáveis pela contribuição ao PIB, segundo a Confederação Nacional da Indústria (CNI).

A atual processo de produção de novos produtos a partir dos recursos naturais é baseado na extração, processamento, manufaturamento, distribuição e consumo com descarte dos insumos gerados ao longo da cadeia, modelo conhecido como linear. Durante o procedimento de extração e obtenção da matéria prima de interesse, subprodutos considerados sem valor econômico não são devidamente reaproveitados resultando em perdas significativas de substâncias com potenciais bioatividades, como carboidratos, proteínas, lipídeos e elementos traços (DAHIYA *et al.*, 2018).

As sementes oleaginosas são uma das matérias primas que podem abranger esses três principais setores industriais. Estimasse que no ano de 2020 foram produzidos mundialmente 579,5 milhões em toneladas de oleaginosas, sendo o Brasil um dos principais exportadores destas sementes (USDA, 2019). As oleaginosas apresentam, aproximadamente, 50% a 70% da matéria seca em óleo vegetal (V. R. DE MOURA *et al.*, 2019), destinados ao consumo alimentar, mas também para a produção de biocombustíveis, enquanto o resíduo seco não é devidamente aproveitado, sendo perdido um percentual significativo de bioativos e elementos traços passíveis de reaproveitamento (OZSEZEN; CANAKCI, 2011; ZAFAR *et al.*, 2019).

O aproveitamento integral dos recursos naturais baseia-se na criação de um modelo circular de produção, visando uma menor taxa de extração dos recursos ambientais, assim como aumentar o valor agregado dos insumos gerados ao longo da linha produtiva. O fracionamento da matéria prima está compreendido dentro do modelo de bioeconomia circular sendo uma solução de abastecimento de matéria prima nos variados setores produtivos.

Para atingir essa mudança ideológica e de execução se faz necessário a expansão do conhecimento sobre o uso circular na utilização da matéria prima. Dessa forma espera-se que a introdução deste conceito não linear de produção consiga alcançar integralmente setores importantes indústria da transformação, como a energética, alimentícia e farmacêutica que podem se beneficiar mutuamente durante as etapas de fracionamento da matéria prima.

## **2 REVISÃO DE LITERATURA**

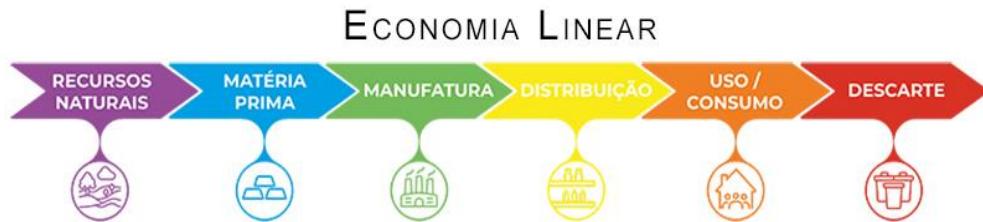
### **2.1 VALORIZAÇÃO INTEGRAL DOS RECURSOS AMBIENTAIS: CONCEITO DE BIOECONOMIA CIRCULAR**

O meio ambiente é reconhecido por oferecer bens essenciais à manutenção da subsistência humana e desenvolvimento local. A utilização de recursos naturais como matéria prima para geração de produtos farmacêuticos, químicos e alimentícios é reconhecido desde o início da história humana (DA SILVA; RODRIGUES, 2014). Entretanto, o desenvolvimento acelerado da população global tem gerado danos ambientais com perda de biodiversidade, exaustão de recursos naturais e intensas modificações climáticas (D'AMATO; KORHONEN; TOPPINEN, 2019). Esta atual situação tem sido trabalhada junto a ambientalistas, economistas e gestores governamentais na finalidade de reduzir os danos ambientais sem comprometer o desenvolvimento econômico e as necessidades do homem (D'AMATO *et al.*, 2019; NATIONS, 2018, 2015).

A atual abordagem linear de produção (Figura 1) extração, processamento, manufaturamento, distribuição, consumo e descarte é reconhecido com um dos mais antigos modelos de produção aplicado aos setores industriais, principalmente na indústria de transformação. A indústria da transformação compreende todo e qualquer setor que envolva a utilização dos recursos primários para o desenvolvimento de novos produtos, e é reconhecida como um dos maiores produtores de insumos não tratados mas com possibilidade de reaproveitamento (D'AMATO *et al.*, 2015).

Novos modelos de extração, produção e consumo têm sido explorado, enfatizando o uso sustentável da matéria prima ambiental (GU *et al.*, 2020). O novo modelo tem como base a não linearidade da escala produtiva abordando uma teoria circular de reaproveitamento dos resíduos gerados ao longo da cadeia produtiva e do consumo.

Figura 1: Modelo de produção linear – Economia linear

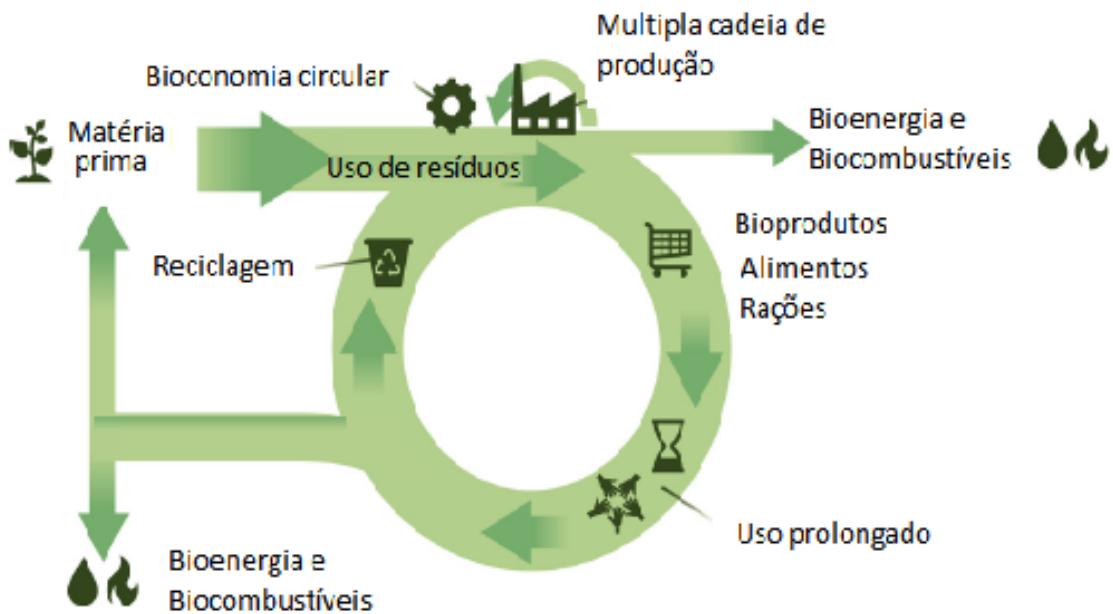


Fonte: A criação (<http://www.acriacao.com/>)

Em 2012 a Comissão Europeia adotou o conceito da bioeconomia circular (BC) como base fundamental para enfrentar os desafios mundiais nos setores ecológicos, ambientais, energéticos e alimentícios (NATIONS, 2018). A BC vem sendo adotada como referência a um sistema fechado de produção (economia circular) com utilização de matérias primas renováveis, baixa geração de resíduos, reutilização e reciclagem dos subprodutos gerados na cadeia produtiva (Figura 2) (CARUS; DAMMER, 2018; SHERWOOD, 2020) incorporando a ideia da eliminação de resíduos através do *design* superior de materiais e produtos (MICHELINI *et al.*, 2017) com aplicação biotecnológica em setores de alto impacto econômico, como a indústria alimentícia, de biocombustíveis e farmacêuticas (SCARLAT *et al.*, 2015).

D'Amato et al. (2019) descrevem a bioeconomia como um conjunto de soluções tecnológicas destinadas a substituição no uso de recursos não renováveis por alternativas de base biológicas renováveis (D'AMATO; KORHONEN; TOPPINEN, 2019). Estima-se que a utilização de recursos renováveis na produção de bioproductos (combustíveis, polímeros e químicos) alcance US\$ 478 bilhões em 2024 (KIRCHER, 2019). Os setores de bioenergia e biocombustíveis e bioproductos são reconhecidos como base fundamental para alcançar uma conversão gradual entre o modelo linear de produção e o modelo BC (KAMM *et al.*, 2008).

Figura 2: Modelo de Bioeconomia circular e seus elementos



Fonte: Adaptado de Stegmann, et al. (STEGMANN; LONDO; JUNGINGER, 2020)

Segundo a Organização para a Cooperação e Desenvolvimento Econômico (OECD), a bioeconomia impacta a economia mundial em aproximadamente 2 trilhões de Euros, com impacto direto nas ações propostas na lista da ODS - Objetivos de Desenvolvimento Sustentável da ONU (Figura 3) (NATIONS, 2015). Sendo assim a BC envolve a produção de inúmeros produtos, como: Biopolímeros, Biopesticidas, Pigmentos, Fragrâncias, Medicamentos e Alimentos funcionais e Nutracêuticos. No Brasil, país com uma das maiores biodiversidades, a BC demonstra potencial para impulsionar a multifuncionalidade da agricultura local, visando a produção de alimentos, fibras e de energia (biocombustíveis). Atualmente no país uma forte tendência na prática da BC é a utilização da biomassa integral ou residual como matéria prima para o desenvolvimento de bioproductos nos variados setores industriais (BRASIL-EMBRAPA).

Figura 3: Objetivos de Desenvolvimento Sustentável da ONU para a Agenda 2030



Fonte: Agenda 2030

## 2.2 BIOMASSA

Embora o termo *biomassa* seja associado diretamente à produção de biocombustíveis alguns estudos apontam seu uso como matéria prima para diferentes finalidades, como aplicação na construção civil (LIUZZI *et al.*, 2020; ROJAS *et al.*, 2019), tratamento de redes fluviais (ALOMÁ *et al.*, 2012; BRANDÃO *et al.*, 2010; DO NASCIMENTO; DE OLIVEIRA; LEITE, 2019), medicina diagnóstica (ABBAS; MARIANA; PHAN, 2018; PAPAIOANNOU *et al.*, 2018; SINGH, Sandip K., 2019), alimentos (ANDRADE, Amanda Cristina *et al.*, 2020; MACIEL *et al.*, 2020) e farmacêutica (BAYOUMI *et al.*, 2020; TAOKAEW; OFUCHI; KOBAYASHI, 2020).

O conceito de *biomassa* é aplicado para todo e qualquer material de origem orgânica derivado de frações vegetais ou animais, incluindo produtos sólidos, líquidos e gasosos recuperados de materiais orgânicos não fossilizados e biodegradáveis (BASU, 2018). Osman e colaboradores (2019) classificam a biomassa em três categorias sendo: (I) Biomassa primária, como resultado direto do cultivo, a exemplo da biomassa lenhosa juntamente com sementes oleaginosas e resíduos, (II) Biomassa secundária, resultado do processamento físico/ químico da biomassa primária e (III) Biomassa terciária onde estão inclusos, rejeitos e resíduos pós-consumo, vegetais (tubérculos), e gorduras animais e óleos vegetais usados (OSMAN *et al.*, 2019).

Em biomassas vegetais a fotossíntese é a principal rota para o desenvolvimento da biomassa, a interação entre dióxido de carbono, água e luz solar permite a produção de metabólitos primários, como carboidratos, lignocelulose e celulose além de

metabólitos secundários, extraídos em menor volume e identificados como classes de terpenos, tocoferóis, proteínas, triglicerídeos, compostos fenólicos e flavanóides (BASU, 2018; MAMVURA; DANHA, 2020). Sendo uma fonte para extração de conteúdos aplicáveis a outros setores além da produção de biocombustível, o fracionamento da biomassa permite a reutilização de resíduos orgânicos gerados no manufaturamento inicial da biomassa (Figura 2). O emprego de processos biotecnológicos, quando estrategicamente aplicados, permite o fracionamento da biomassa e a recuperação de compostos químicos e orgânicos, como ácidos carboxílicos, biopolímeros, antioxidantes, macroelementos (proteínas, fibras e carboidratos) e microelementos (minerais) (DHILLON; KAUR; BRAR, 2013).

Mundialmente estima-se que sejam produzidos anualmente 140 bilhões de toneladas de biomassa vegetal (WANG, Jianfeng *et al.*, 2017), gerando uma perda substancial de matéria prima que poderia ser convertida em possíveis produtos com manutenção do valor econômico. No Brasil a indústria da mamona produz, aproximadamente, 90.845 m<sup>3</sup> de resíduos de mamona, aproximadamente 43% do peso seco da semente, sendo óleo vegetal o principal produto de interesse econômico (SÁNCHEZ *et al.*, 2018).

A extração de óleo vegetal é de forte interesse pela indústria do setor energético, que redirecionam a essa biomassa primária para o uso na produção de biodiesel, como é observado no Brasil com o uso de óleo de soja em 70,39% de todo biodiesel produzido no país, segundo dados da Agência Nacional de Petróleo, Gás e Biocombustível (ANP,2018). O reaproveitamento da biomassa residual em suas variadas formas colabora com o ganho econômico na geração de novos produtos com valor agregado, sustentabilidade ambiental e benefícios sociais (GALLO-CORDOVA *et al.*, 2017; POSSO; SIGUENCIA; NARVÁEZ, 2020; RIJAL; GAUTAM; LEBEL, 2020; SAHA; BASAK, 2020).

## 2.3 APLICAÇÃO DA BIOMASSA VEGETAL E SEUS SUBPRODUTOS

A biomassa vegetal pode ser fracionada segundo sua composição química, seguindo a predominância encontrada. A depender da origem vegetal utilizada como matéria prima, em geral é possível encontrar como metabólitos primários polímeros como celulose, hemicelulose e lignina, e compostos oleaginosos quando o recurso vegetal for de origem oleaginosa, a exemplo do licuri, babaçu, macaúba. Por sua vez é possível definir como metabólitos secundários elementos encontrados em menor teor quantitativo

no caso de algumas classes de proteínas, açúcares, e compostos fitoquímicos (BASU, 2018; MAMVURA; DANHA, 2020).

Embora seja possível identificar limitações no uso tradicional de algumas espécies vegetais, por desconhecimento científico, a biomassa vegetal e seus subprodutos podem ser aplicados de acordo com o fracionamento dos metabólitos identificados. A indústria energética e de alimentos são os dois principais ramos da indústria de transformação que trabalham na extração dos recursos vegetais com produção de insumos passíveis de reutilização.

### **2.3.1 Recursos vegetais como fonte energética – Biodiesel**

O aumento da demanda energética global aliada aos agravos ambientais impulsionou o uso da biomassa vegetal para a substituição do petróleo como fonte de carbono, propondo uma mitigação das condições climáticas. (DA SILVA; RODRIGUES, 2014). A biomassa vegetal apresenta estrutura química semelhante ao petróleo, com diferenças na razão atômica dos elementos hidrogênio (H) e oxigênio (O) em relação ao teor do elemento carbono (C). O reduzido teor de carbono corrobora com baixas emissões de dióxido de carbono (CO<sub>2</sub>) (BASU, 2018; WANG, Aiguo G.; AUSTIN; SONG, 2017), sendo considerada, por muitos pesquisadores, uma emissão nula ou neutra. Muitos autores chegam a esta conclusão com a justificativa que o CO<sub>2</sub> liberado corresponde ao valor recuperado durante o processo de fotossíntese vegetal, diante deste entendimento a biomassa vegetal é denominada como *Greenhouse Gas neutral* ou *GHD neutral* (CHO *et al.*, 2019; DAIOGLOU *et al.*, 2019; FREDERICK *et al.*, 2019; SHERWOOD, 2020).

No Brasil, a principal biomassa vegetal ainda consumida para a produção de biocombustíveis é a cana de açúcar, que corresponde a 18% de todo o combustível produzido (DA SILVA; RODRIGUES, 2014). Entretanto, mediante a publicação da Lei nº 11.097/2005, que obriga a incorporação de biodiesel nos combustíveis a diesel, o uso de oleaginosas para obtenção da matéria prima (óleo fixo) e produção do biodiesel, tem crescido significativamente ao longo dos anos (BRASIL, 2005). Segundo a Agência Nacional de Petróleo, Gás e Biocombustível (ANP) em 2019, 70% do biodiesel produzido foi a partir de óleo vegetal da soja, seguido de gorduras animais 11% e outros materiais graxos 8,87% (ANP).

Os óleos vegetais apresentam grandes vantagens quando comparados ao petróleo, apresentam baixos conteúdos de compostos aromáticos e sulfúrico, são biodegradáveis, renováveis, com elevado poder calorífico e baixo índice de emissão de CO<sub>2</sub>

(DEMIRBAS, 2017). A conversão do óleo vegetal em biodiesel visa à redução de características físico-químicas inerentes ao óleo vegetal, responsáveis por danos em motores após o uso direto destes (GARCÍA-MARTÍN *et al.*, 2018).

Como um dos maiores setores econômicos a produção de biodiesel é responsável por gerar dois principais insumos ao longo da cadeia produtiva, (1) a biomassa vegetal residual, após extração do óleo vegetal e (2) glicerol produzido durante a etapa de transesterificação do óleo vegetal para biodiesel. Ambos os elementos podem ser devidamente destinados a dois principais ramos, o setor alimentício no caso da biomassa residual e o glicerol para a indústria farmacêutica. Dados na literatura sugerem a que a biomassa residual vegetal pode ser um importante recurso nutricional.

### 2.3.1.1 Transesterificação

O processo de conversão de óleos vegetais ou gordura animal baseia-se em sequências reacionais de transesterificação e esterificação que tem como princípio a troca entre um grupo alquil da função estér por um álcool como metanol e etanol, com auxílio de um a gente catalisador que converte os triglicerídeos (TGA) em ésteres metílicos (FAME) ou etílicos (FAEE) de ácidos graxos mais fração de glicerol, este como subproduto da reação (MOAZENI; CHEN; ZHANG, 2019).

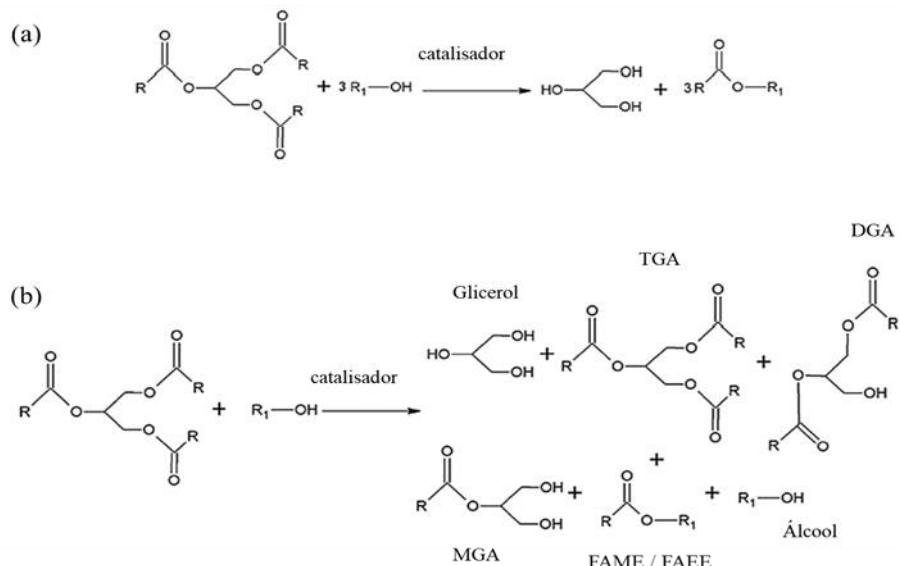
De maneira simplificada a transesterificação resultaria na conversão completa dos TGAs em três moléculas de ésteres e uma de glicerol (Figura 4a) com consumo completo do a gente catalisador empregado. Entretanto estima-se que o resultado final do processo reacional apresenta formação de co-produtos além dos ésteres metílicos/etílicos, como glicerol, triglicerídeo, diglycerídeos, monoglycerídeos, álcool e catalisador (Figura 4b), não apresentando 100% de conversão entre TGA e FAME / FAEE (AHMAD *et al.*, 2019).

A qualidade da matéria prima determina o tipo de catalisador necessário para produção do biodiesel que atenda as normas estabelecidas. Fatores como, razão molar entre o álcool e TGA (A:TGA), presença de ácidos graxos livres >2,5% e tipo de catalisador empregado são pré-determinantes na taxa de obtenção final do biodiesel (WAN GHAZALI *et al.*, 2015). Os catalisadores, atualmente, conhecidos podem ser divididos em três categorias, alcalinos, ácidos e enzimáticos. O elevado custo e maior tempo de reação tornam os catalisadores enzimáticos pouco atrativos comercialmente, sendo os catalisadores alcalinos e ácidos a principal escolha para produção de biodiesel comercial (LEUNG, Dennis Y.C.; WU; LEUNG, 2010).

A transesterificação alcalina, empregando catalisadores como hidróxido de sódio (NaOH) ou potássio (KOH), é considerada a mais econômica das opções, exigindo menor tempo de reação, baixas temperaturas e pressão ambiente que resultam em altas taxas de conversão dos TGAs em FAME/FAEE. Alguns óleos vegetais apresentam concentrações, consideráveis, de água e frações de ácidos graxos livres, nestas condições o uso de catalisadores alcalinos resulta em reações secundárias, como reação de saponificação (sabão) e água (Figura 5a). A água em presença de catalisadores alcalinos contribuem na hidrólise do biodiesel revertendo-o a ácido graxo livre e álcool (Figura 5b) (LEUNG, Dennis Y.C.; WU; LEUNG, 2010; MOAZENI; CHEN; ZHANG, 2019). As reações secundárias aumentam o tempo de reação e reduzem a taxa de conversão dos TGAs em FAME/FEE.

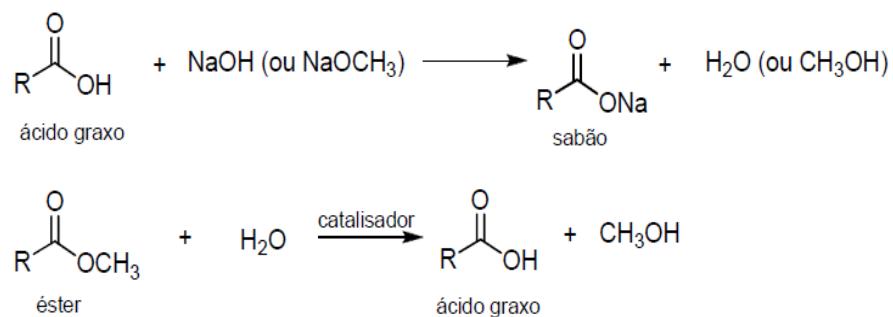
Em condições com teores de ácidos graxos livre >2,5% o uso de catalisadores ácidos (HCl, H<sub>2</sub>SO<sub>4</sub> e BF<sub>3</sub>) são escolhidos como pré-tratamento para redução da formação dos compostos secundários, uma vez que a reação não sofre interferência na presença de ácidos graxos livres. Entretanto, maiores concentrações na razão molar Álcool:TGA são requeridas, quando empregado catalisadores ácidos (SAMIOS *et al.*, 2009), aumentando assim o custo operacional.

Figura 4: Esquema do processo de transesterificação (a) condição ideal 100% de conversão (b) esquema com produtos secundários obtidos na produção do biodiesel



Fonte: Adaptado de (SAMIOS *et al.*, 2009)

Figura 5: Reação de transesterificação (a) com formação de sabão e (b) hidrólise do éster (biodiesel)



Fonte: Moraes, D.C. *et al.*, 2010

A concentração de álcool presente na reação é considerado fator importante para um bom rendimento e taxa de conversão. Excessos de álcool são empregados como garantia para uma conversão de TGAs mais significativa em menor tempo. Entretanto, em teoria são necessários 3 mol de álcool para converter 1 mol de TGA, produzindo 3 mol de éster e 1 mol de glicerol. Embora pesquisas apontem diferenças nas taxas de álcool ideais, a razão molar empregada na reação de transesterificação está associada ao tipo de catalisador envolvido e consequentemente a presença de ácidos graxos livres (LEUNG, D. Y.C.; GUO, 2006; LEUNG, Dennis Y.C.; WU; LEUNG, 2010).

Seguindo a estequiometria uma tonelada de óleo vegetal é capaz de produzir 103 kg de glicerina como co-produto do biodiesel (ALTITUDE; RESEARCH; REFINING, 1998; JAYED *et al.*, 2009). A produção de glicerol na reação de transesterificação é um dos problemas levantados por ambientalistas e pesquisadores da área. A contaminação pelo álcool utilizado na reação impede a utilização direta da glicerina pela indústria farmacêutica. Entretanto, embora represente um grande impacto ambiental quando não destinado de maneira adequada, a glicerina não é o único sub-produto produzido durante a obtenção do biodiesel.

Resíduos sólidos produzidos durante a extração dos óleos vegetais em biorefinarias são geralmente queimados em caldeiras ou com outras fidalidades de aquecimento, gerando fumaças e poluição (JAYED *et al.*, 2009). No Brasil a indústria de mamona produz, aproximadamente, 90.845 m<sup>3</sup> de resíduos de mamona, deixados a decompor como fertilizantes. Os resíduos gerados pela extração da mamona corresponde a 43% do peso da semente, apresentando nutrientes além como proteínas, carboidratos e alguns elementos minerais (SÁNCHEZ *et al.*, 2018).

### **2.3.2 Potencial uso da biomassa residual como recurso alimentício**

Com a mudança no padrão alimentar mundial, as doenças crônicas não transmissíveis (DCNTs) como diabetes, cardiopatias, hipertensão, e câncer estão entre as principais causas de morbi-mortalidades consideradas pela Organização Mundial de Saúde (OMS) um grave problema de saúde pública. Dessa forma fazendo-se necessário a busca por recursos alimentares capazes de promover mudanças no atual quadro instaurado, propondo benefícios e novas condutas terapêuticas, alimentos que demonstrem esse potencial podem ser debatidos como um recurso nutracêutico e funcional (JHAN *et al.*, 2020).

O termo nutracêutico entre pesquisadores da área ainda não é concisa. Segundo alguns autores qualquer extrato ou agente fitoquímico que resultem em benefícios à saúde, com capacidade de prevenir o desenvolvimento de patologias pode ser enquadrado como elemento nutracêutico ou alimento com esta funcionalidade (LÓPEZ-GUTIÉRREZ *et al.*, 2015). As fontes vegetais têm sido alvo de intensas pesquisas para a obtenção, quantificação e descobertas de novos elementos nutracêuticos, sendo empregados como uma proposta terapêutica na conduta clínica de patologias variadas, resultantes dos padrões de alimentação nutricional inadequada (UDEH; NYILA; KANU, 2020).

Dentre estes possíveis elementos o fitoquímicos e compostos hidrocolóides encontram-se entre os ativos nutracêuticos mais pesquisados, juntamente com a busca de novos prébióticos e probióticos. São propostos como elementos bioativos na regulação de doenças cardiovasculares, da obesidade, controle insulínico em quadros de diabetes tipo 2, regulação da resposta glicêmica, imunológico e da função colôn retal com manutenção da microbiota (FENG *et al.*, 2020; MANZOOR *et al.*, 2020).

Compostos fenólicos estão entre os grupos de compostos fitoquímicos mais isolados e pesquisados entre variadas espécies vegetais e frações caules, folhas, frutos. Reconhecidos por sua ação antioxidante, os compostos fenólicos atuam removendo radicais livres, como quelantes de metais, na ativação de enzimas antioxidantes e inibição de oxidações (OBOH; ADEMOSUN, 2011). Alguns estudos apontam a atuação de compostos fenólicos no controle da hipertensão assim como na prevenção e controle da diabetes tipo 2, através da inibição de enzimas possivelmente correlacionadas com estas patologias, como  $\alpha$ -amilase,  $\alpha$ -glucosidase, e enzima conversora de angiotensina I (OBOH; ADEMOSUN, 2011; SEYOUN; ASRES; EL-FIKY, 2006), além de apresentar, também envolvimento na prevenção de outras patologias, como câncer de colôn retal, favorecendo a modulação da microbiota local (CIANCIOSI *et al.*, 2020).

O teor de compostos fenólicos extraídos é correlacionado diretamente com tipo de cultivar, estágio de maturação, fração trabalhada na extração, e demais constituintes químicos intrínsecos do material vegetal (ALAÑÓN *et al.*, 2021). Embora estes fitoquímicos sejam mais evidentes e reconhecidos, quantitativamente, em frutos e vegetais cítricos, espécies oleaginosas demonstram apresentar também grupos fenólicos envolvidos nos constituintes químicos.

Estudos a partir da caracterização dos resíduos sólidos das sementes de oliva apontam a permanência de 98% de compostos fenólicos na biomassa residual, sendo uma fonte considerável de agentes antioxidantes não reaproveitados (EC, 2008; SUÁREZ *et al.*, 2009). Rahaman et al. mostraram que após a extração de óleo vegetal de sementes da Camelina (*Camelina sativa*), empregados como ração animal, a biomassa residual das sementes apresentaram concentração de compostos fenólicos iguais a  $11.69 \pm 0.44$  mg GAE/g (RAHMAN; COSTA DE CAMARGO; SHAHIDI, 2018).

As fibras vegetais (celulose, hemicelulose, lignina) desempenham um fator crucial na biodisponibilidade dos compostos fitoquímicos de origem vegetal. Através de ligações fracas entre os grupos OH<sup>-</sup> (hidroxilos) dos compostos fenólicos e das fibras dietéticas o complexo formado permanece estável em pHs alcalinos (pH > 7), permitindo

a biodisponibilidade destes compostos onde há variação de pH entre 2 a 7, que corresponde, respectivamente, ao ambientes gástrico e colôn retal, disponibilidade nestas regiões permite uma maior absorção induzindo a efeitos benéficos locais e sistémicos (JAKOBÉK; MATIĆ, 2019; LIU, Chengzhen *et al.*, 2016; LIU, J. *et al.*, 2018; SAURA-CALIXTO, 2011).

As fibras dietéticas juntamente com algumas classes proteícas, compõem o grupo dos elementos reconhecidos hidrocolóides. Os hidrocolóides apresentam uma importância tecnológica à indústria de alimentos, porém atualmente têm-se demonstrado numerosas ações terapêuticas, incluindo anti-diabética, anti-hipertensiva, hipercolesterolêmica, antimicrobiana, com potencial atividade pré-biótica, em virtude a resistência a digestão gastrointestinal, a exemplo das fibras dietéticas que tem impacto na composição da microbiota intestinal (MANZOOR *et al.*, 2020).

### **2.3.3 Potencial uso na indústria farmacêutica**

Os óleos fixos vegetais são empregados, historicamente, como bases cosméticas e medicinais, tendo sido já descritos por trazer efeitos benéficos a fisiologia da pele, atuando como uma barreira protetora reduzindo a perda de água transepidermal (LIN; ZHONG; SANTIAGO, 2018). Em geral os óleos fixos vegetais são formados por ácidos graxos livres, triacilglicerol (TAG), e compostos quimicamente ativos a exemplo dos grupos fenólicos referidos por sua ação antioxidante (LERCKER; RODRIGUEZ-ESTRADA, 2000).

Uma das grandes vantagens no uso de óleos vegetais em formulações cosméticas é sua larga produção e biocompatibilidade com a pele humana, uma vez que o extrato córneo, responsável pela manutenção da barreira física da pele é constituído ceramidas (50%), colesterol (27%) e ácidos graxos livres saturados (10%) variando ante 14 a 22 carbonos (WERTZ, 2018), como ácido palmítico, ácido esteárico, ácido oleico e ácido linoleico (KANG; HO; CHAN, 2006). O emprego de fontes naturais, com os óleos fixos vegetais, tem-se demonstrado uma alternativa para o desenvolvimento tecnológico e sustentável, propondo desenvolvimento econômico local e manutenção da valorização das espécies vegetais.

Alguns estudos científicos apontam o uso de óleos vegetais, como óleo de soja (ARIANTO; CELLA; BANGUN, 2019) e óleo de girassol (ARIANTO; CINDY, 2019) na formulação de protetor solares em substituição ao emprego de filtros solares sintéticos.

A presença de compostos antioxidantes na composição natural dos óleos vegetais corrobora com a substituição proposta pela indústria farmacêutica e cosmética. A incorporação de óleos fixos vegetais contribui com o aumento da fotoestabilidade do filtro diminuindo a absorção de raios ultravioletas (UV) pela pele, as micelas ocasionais geradas no processo de homogeneização em um sistema óleo/água ou água/óleo torna-se um carreador promissor dos agentes fotoprotetores presente na formulação (TEERANACHAIDEEKUL *et al.*, 2020).

A eficácia do protetor solar deve garantir um fator de proteção solar (FPS) atuante nas faixas da radiação ultravioleta A (UVA) na faixa de comprimento de onda de 320-400 nm e B (UVB) na faixa entre 290-320 (ARIANTO; CELLA; BANGUN, 2019). Segundo a Agência Nacional de Vigilância Sanitária o menor nível de proteção solar aceitável corresponde a FPS 6 (BRASIL, 2010).

A exposição aos raios ultravioletas compreende uma das principais causas de fotoenvelhecimento da pele, queimaduras, eritemas e aumenta as chances para o desenvolvimento de câncer de pele. O Brasil é listado entre os países com uma das maiores incidências em câncer de pele, que entre os tumores malignos corresponde a 27% de todos os registros de câncer no Brasil, sendo a região Norte e Nordeste maiores acometidas (INCA).

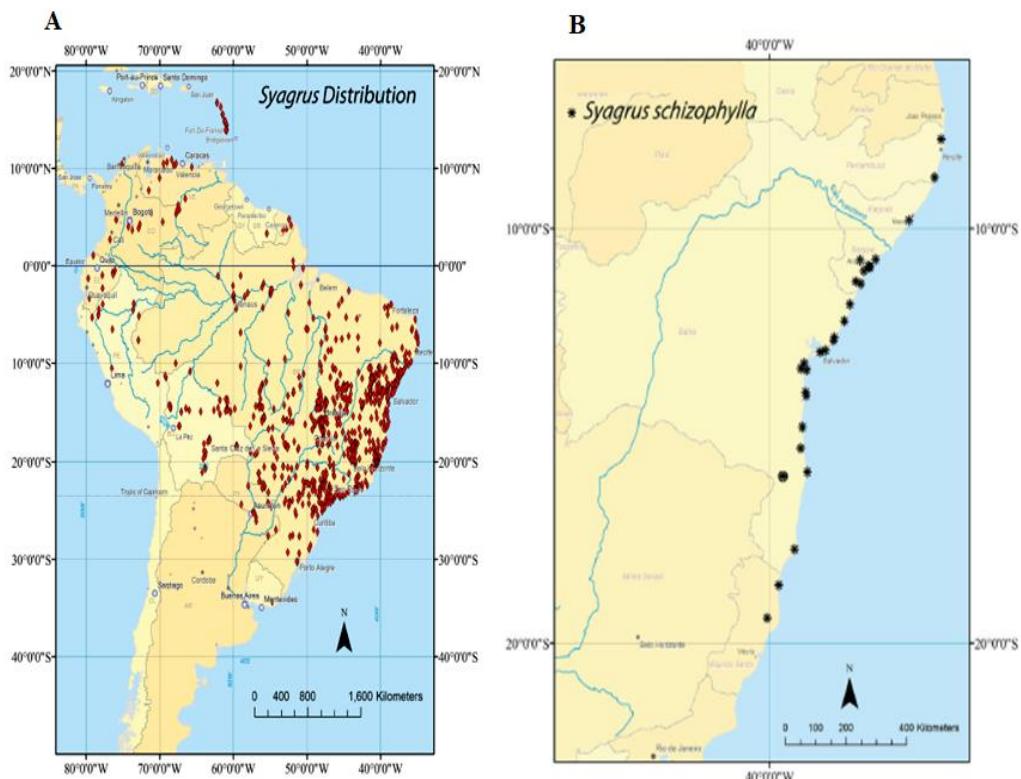
#### 2.4 *SYAGRUS SCHIZOPHYLLA* (MART.) GLASSMAN

As palmeiras são plantas de morfologia variada, pertencentes a família Arecaceae, antigamente denominada como Palmae. Os padrões de diversidade e abundância de palmeiras nas regiões neotropicais, como América Central, Sul do México, Caribe e América do Sul, estão relacionados com a sazonalidade, temperatura e condições edáficas destas regiões (SVENING, 2001). O gênero *Syagrus* encontra-se distribuído ao longo das regiões no Sul do México, América Central, Caribe e América do Sul. No Brasil este gênero é representado por 57 espécies nativas, sendo a região Central e Leste do país as de maior concentração de palmeiras deste gênero (Figura 6a). Dentre as 57 espécies apenas algumas delas apresentam maior conhecimento popular e de interesse científico, a exemplo do Licuri (*Syagrus coronata*) de amplo reconhecimento regional (SVENING, 2001). A carência de informações e detalhamento entre as espécies deste gênero redireciona erroneamente a sua identificação aplicação biotecnológica.

Encontrada ao longo do Estado de Pernambuco até região norte do Espírito Santo, a espécie *Syagrus schizophylla*, popularmente identificada como Aricuriroba, é observada

ao longo da restinga costeira, costa litorânea e florestas adjacentes com solos arenosos (Figura 6) (PRESS; ZEALAND, 2017., LEITMAN, P.; et al., 2015). As palmeiras de Aricuriroba são caracterizadas por serem de baixo porte, solitárias e de crescimento folhoso disperso. A Aricuriroba é distinguida de outras espécies do gênero *Syagrus* pela presença de pecíolos armados, pedúnculo incomumente longo, fruta amarela-alaranjada, bases de pecíolos foliares persistentes e bainhas foliares em caule estreito (Figura 7). Usualmente destinadas ao paisagismo e produção de artesanato a partir de suas folhas, as palmeiras de Aricuriroba apresentam informações escassas quanto ao seu potencial biotecnológico.

Figura 6: Distribuição geográfica. A- Gênero *Syagrus* no Brasil e B-Éspecie *Syagrus schizophylla*



Fonte: (PRESS; ZEALAND, 2017)

Figura 7: *Syagrus schizophylla*. A- Palmeira de *S.schizophylla*, B- Pseudopetioles espinhoso, C - Intumecência com pedúnculo longo, D - Fruto maduro, E-Endocarpo com 1,5 cm



Fonte: Elaborada pelo autor

### 3 OBJETIVOS

#### 3.2 GERAL

Propor alternativas para a valorização dos frutos de *Syagrus schizophylla* (Mart.) Glassman para aplicação nos setores da indústria energética, farmacológica e alimentícia.

#### 3.2 ESPECÍFICOS

##### **Artigo I**

- Remover a umidade do resíduo sólido após a extração do óleo vegetal;
- Avaliar seu conteúdo centesimal e quantificar elementos minerais;
- Avaliar as propriedades tecnológicas da biomassa residual;
- Extrair e quantificar proteínas da biomassa residual;
- Realizar a extração de compostos químicos por solventes orgânicos de acordo com a escala eulotrópica;
- Avaliar os extratos quanto ao potencial antimicrobiano;
- Avaliar o potencial antioxidante dos extratos obtidos.

##### **Artigo II:**

- Extrair o óleo vegetal de *Syagrus schizophylla* (Mart.) Glassman por prensagem a frio;
- Caracterizar o óleo vegetal quanto as condições físico-químicas;
- Caracterizar constituição química do óleo vegetal por transesterificação e identificação por cromatografia;
- Realizar a transesterificação ácida do óleo vegetal para produção de biodiesel B100
- Caracterizar o biodiesel B100 quanto às condições físico-químicas;
- Caracterizar a composição química e estrutural do biodiesel B100;
- Caracterizar o tempo de oxidação do biodiesel B100;
- Caracterizar o biodiesel B100 quanto ao seu tempo de degradação térmica.

##### **Artigo III: Patente de Invenção**

- Elaborar um fotoprotetor solar a partir do óleo vegetal;
- Avaliar o tempo de estabilidade térmica;
- Avaliar o índice de espalhabilidade.

## 4 RESULTADOS

### **Artigo I - Residual cake of *Syagrus schizophylla* (Mart.) Glassman, a Brazilian palm tree: Source of protein, fiber, and antioxidants compounds**

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### **Abstract**

*Syagrus schizophylla* is a moderate palm tree that occurs endemically in Brazil's Northeast and Southeast regions. It is prevalent in the Atlantic Rain Forest's phytogeographic domain, occupying coastal areas plains. The extraction of oil from the seeds generates a residual cake usually despised without added economic value. In this work, the residual cake from *S. schizophylla* (RCS) oilseed is evaluated for its nutritional

content, technological properties, and potential resource of phenolic compounds and antioxidant activity. The RCS showed high content of protein ( $20.89 \pm 0.45$  g/100g), fibers ( $25.59 \pm 0.07$  g/100g), mineral elements, and phenolic compounds (46.71 to 48.98 g GAE/g) in eight different extracts that have antioxidant activity in the total antioxidant capacity (13.84 to 65.41 mg/mL) and reducing power assay (2.96 to 4.43 mg/mL). The concentration of phenolic varied among the different solutions applied in the extraction. FTIR and X-ray diffraction analysis allowed inferring the amylose-lipid complex that influences phenolic compounds' bioavailability for better absorption and activity. To the best of our knowledge, these findings are the first for this species of *Syagrus* and contribute to the conscious reuse of residual cake by the local community once *S. schizophylla* is a potential source of nutrients and bioactive compounds.

**Key-words:** Bioactivity, Sustainability, Aricuriroba, Oilseed, Nutraceutical

## 1. Introduction

With six biomes in Brazil's different regions, the Atlantic Rain Forest and Caatinga phytogeographic domain together account for 23% of the entire national territory. The interaction between these two different phytogeographic domains can be observed through the vegetation adapted for various conditions. As seen in the northeast of the country, mainly in coastal regions with the presence of Atlantic Rain Forest with the transition to the semi-arid climate of the Caatinga in areas further from the states of Maranhão, Piauí, Ceará, Rio Grande do Norte, Paraíba, Pernambuco, Alagoas, Sergipe e Bahia (BRAZIL, [s. d.]). The combination of the specific soil and microclimatic variation between these biomes in Brazil's Northeast results in the biodiversity used by the local communities for food supply, livestock feeding, ornaments, and medicines (RICARDO *et al.*, 2018). These activities contribute to local socio-economic development once Brazil's northeast region presents one of its worst local development indexes.

The genus *Syagrus* is the most representative palms trees in South America. Only in Brazil have recognized 39 species distributed throughout the territory with the majority of growth in the dry forest or transitional forests, such as Caatinga and Atlantic Rain Forest (BRUNO *et al.*, 2019; PRESS; ZEALAND, 2017). As can be seen with *S. coronata* (Licuri) and *S. cearenses* (coco-babão) that are used to production of variant foods, and *S. schizophylla* (Aricuriroba) that have the leaves for craft and the palm for ornamental.

The main product from *Syagrus* species is a large amount of vegetable oil used in the culinary. The other fraction of the fruit is despised generated non-reusable solid waste (residual biomass). According to the Food and Agriculture Organization of the United Nations (FAO), 1.3 billion of food per year is lost or wasted globally, representing economic and environmental problems (ANDRADE, Mariana A. *et al.*, 2019; TSANG *et al.*, 2019). Some studies indicate that some residual biomass from processed vegetable oilseeds can be an essential resource of bioactive compounds, with equivalent biological activities compared with natural consumption (ANDRADE, Mariana A. *et al.*, 2019). In residual olive cake (residual biomass), 98% of the fruit's phenolic compound is retained in solid waste after oil extraction (E.C., 2008; Suárez, Romero, Ramo, Macià, & Motilva, 2009). Studies with Licuri showed phenolic compounds and secondary metabolites from the seeds (BELVISO *et al.*, 2013). Like other native plants in Brazil, the *S. schizophylla* do not have enough scientific data about the plant's fruits and different fractions. This species can be an essential source for local economic development in addition to ornamental and culinary use, mainly in regions with a low human development index of the country, such as the North and Northeast of Brazil.

In this context, the present paper's proposal aims at studying *S. schizophylla* residual cake after extraction of the fixed oil. To the best of our knowledge, there is no current literature about the *S. schizophylla* nutritional quality, antioxidants and mineral compound contents, and technological properties. This study contributes to expanding scientific knowledge proposing a sustainable solution for the residual biomass after extraction of fixed oil with potential economic prospects for the local communities.

## **2. Materials and Methods**

### 2.1 Sample preparation of residual biomass

The residual biomass of *S. schizophylla* (Mart.) Glassman was obtained after fixed oil extraction in a hydraulic press. The seed was harvested in Coruripe Power Plant, Industry Ordinance, Alagoas, Brazil. The specimen was identified and recorded in Herbarium of the Biology Department of Universidade Federal Rural de Pernambuco (UFRPE) with voucher Nº 55.148 BR-AL-CORURIPE, B.O. VERAS. The residual biomass was processed in raw analytical mill IKA® A11 and dried in the chamber at 60°C for 48h. The processed residual cake of *Syagrus schizophylla* (RCS) was restored in selected bags and kept refrigerated at 4° C until respective analyzes.

## 2. Nutritional composition

### 2.2.1 Proximate composition of the residual cake

The centesimal composition was determined according to the Association of Official Analytical Chemists (AOAC). Moisture (Method AOAC 934-01), ash (Method AOAC 923-03), lipids (Method AOAC 920-39), fibers (AOAC 925-09), and proteins (Method AOAC 984-13), for convert nitrogen to protein value a factor 6.25 was used. Carbohydrate value was determined according to the sum of all elements to pass above at least 100%.

In addition to identifying the group protein in RCS extraction, the fraction of proteins was carried out according to Know et al. (KWON; PARK; RHEE, 1996). The RCS was defatted with hexane (1:10 w/v) and dried. Different solutions were previously prepared, including deionized water (Albumins), NaCl (Globulins), 70% 2-propanol (IPA) (Prolamins), 50% glacial acetic acid (Glutein-1), and 0.1 M NaOH (Glutein-2) and used in sequence to extract proteins in RCS. The extraction in each solution occurred with the following procedures: temperature of 4°C, overnight stirring. After extraction, the sample was centrifuged at 20.000 x g for 30 minutes, and the supernatant was collected and dialyzed in deionized water at 4°C for 48-56h. The resulting fraction was lyophilized and stored at -20°C, and protein concentration was determined by the Bradford method (BRADFORD, 1976).

### 2.2.2 Identification of fatty acid profile

The fatty acid profile of vegetable oil obtained by hydraulic pressing was identified by gas chromatography (GC). For the procedure, an Agilent Technologies Gas Chromatograph (Palo Alto, CA, USA) 5975C series, with triple quadrupole detection system equipped with DB-5MS column Agilent Technologies (30 m × 0.25 mm x 0.25 µm) was used. The oven temperature was set to rise to 150°C at 2°C/min, increased to 280°C, and held for 6 min. with an injection temperature of 230°C and an interface temperature of 260°C. 1 µL of hexane extract was injected in the gas chromatography coupled to CG-MS mass spectrometry. The FAME (Mix C4-C22) mix C22 fatty acid standard was used and compared by the similarity of mass spectra in available libraries (NIST08 and Wiley Registry™ 9th Edition) and retention Index to authentic standards open in the Adams (2011). For quantification, a Thermo Trace GC Ultra system (Thermo Scientific, Milan, Italy) equipped with a flame ionization detector (GC-FID) and column

VB-5 (30mm x 0.25 mm) was applied. The injection of samples occurred under the same conditions as GC-MS.

### 2.2.2 Mineral analysis

The mineral elements contents in the RCS sample were analyzed by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) with previous HNO<sub>3</sub> digestion carried out in a MARS microwave digester at elevated temperature and pressure. After mineral extraction, the volume was adjusted to 15ml with deionized water and then analyzed. The elements analyzed and quantified were Ca, Cu, Fe, P, K, Na, Zn, and Mg. The values were compared with Recommended Dietary Allowances (RDA) in accordance with Food and Nutrition Board, National Academies.

### 2.3 Infrared spectroscopy

Infrared spectroscopy analysis was carried out using Vertex-70 spectrometer (Bruker, USA) with a diffuse reflectance accessory at 25°C. The data was recorded in the range of 500 to 4000cm<sup>-1</sup> wavenumbers with a spectral resolution of 4 cm<sup>-1</sup>. The reflectance spectra were analyzed in GraphPad® Prism 6.

### 2.4 X-ray diffractograms (XDR)

The X-ray diffractions of RCS were performed using a D8-Advance X-ray diffractometer (Bruker, USA) at 25 °C, employing Cu K $\alpha$  radiation ( $\lambda=1.5406\text{ \AA}$ ) from 4° to 40° (2 $\theta$ ), at the flow rate of 2°/min, and a step size of 0.060°. The reflectance spectra were analyzed in Origin8. The relative crystallinity (RC) was calculated as described by Rabek (1980), RC (%) = [(I<sub>c</sub> - I<sub>a</sub>) / I<sub>c</sub>] x 100, where (I<sub>c</sub>) is crystallized area and (I<sub>a</sub>) is the amorphous area.

### 2.5 Technological properties

#### 2.5.1 Water and oil holding capacity and water solubility

The water holding capacity (WHC) and oil holding capacity (OHC) were determined, according to Texeira et al. (TEIXEIRA *et al.*, 2018a). The water solubility (WS) was analyzed according to Farooq et al. (FAROOQ *et al.*, 2018) with modification. To WHC and OHC, 1.0 g of SSF sample (m<sub>1</sub>) was weighed into a 50 mL centrifuge tube of known weight (m<sub>2</sub>), then 20 mL of water (for WHC) and soybean oil (for OHC) were added and vortex for 2 minutes. The sample was centrifuged at 3000 x g for 15 minutes,

and the supernatant was separated and dispersed in a glass dish to determine the WS index. The sludge was drained and weighting the tube ( $m_3$ ). To WS, the dishes were dried at 105° C to constant weight ( $m_4$ ). The index of absorption and solubility capacity was estimated with the following equations WHC/OHC (g/g of flour) =  $(m_3 - m_2 - m_3) / m_1$  and WS =  $(m_4 / m_1) \times 100$ .

## 2.6 Antioxidant analysis

### 2.6.1 Sample extraction

The dried sample of RCS was submitted to extraction with eight solvents according to the eluotropic series (hexane > ethyl ether > chloroform > dichloromethane > acetone > ethyl acetate > methanol) and completed in sterile distilled water. The same dry material was used in each solvent in the proportion of 1:10 (w/v), remaining under agitation overnight. After extraction, the material was filtered and the solvent recovered by evaporation. For each antioxidant activity the 5 mg of each extract was diluted in 1mL of dimethylsulfoxide (DMSO) and distilled water 1: 1 (v/v) with subsequent serial dilution, varying the concentration from 5 mg/mL to 0.15 mg/mL.

### 2.6.2 Total phenolic and flavonoids compounds

The total phenolic content (TPC) of each extract was determined according to Singleton and Rossi (SINGLETON; ROSSI, 1985) with modifications. Briefly, 20 $\mu$ L of each sample was taken in a microplate, and Folin-Ciocalteu reagent 100 $\mu$ L was added. Sodium carbonate (80 $\mu$ L) was added to each well for a neutralization reaction. The microplate was allowed to stand for 30 min at room temperature in the dark followed. The resultant's absorbance was read in a microplate reader at 765 nm (Bioteck FLx800 Fluorescence Microplate Reader, Agilent Technologies, Palo Alto, CA, USA). The TPC in each extract was determined and expressed as an equivalent gram of quercetin per gram of extract (g GAE/g).

The total flavonoid content (TFC) was determined by Woisky and Salantino (WOISKY; SALATINO, 1998) colorimetric method with some modifications for microplate use. In the microplate, 100 $\mu$ L of each extract solution was mixed with 100 $\mu$ L of aluminum chloride solution ( $AlCl_3$ ) previously diluted in 2% ethanol. The microplates were kept in the dark at room temperature for 60 min, and absorbance was read at 420 nm. The TFC was calculated from a standard curve for quercetin and results expressed as an equivalent gram of quercetin per gram of extract (g QUE/g).

### 2.6.3 DPPH free radical scavenging assay

Antioxidant activity was assessed by the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging assay described by Blois et al. (GROUP; GROUP, 1958) with modifications for 96-well microplate. For each previously diluted extract, 40 µL was added to a microplate with 250 µL of DDPH (60 µM; MM = 394.32 g.mol<sup>-1</sup>). Absorbance was measured at 517 nm after 30 min. DPPH solution in the absence of antioxidant was used to control (no consumption of DPPH expected). The percent inhibition (I %) was calculated using the following equation: I% = [(Abc - Abs)/ Abc] x 100, where Ac is the absorbance of the control and As is the absorbance of the sample. IC<sub>50</sub> of DPPH activity was calculated based on the linear regression of the remaining DPPH against the sample concentration. Experiments were performed in triplicate, and the results were expressed as IC<sub>50</sub> (mg/mL).

### 2.6.4 Reducing power assay

The reducing power was determined according to Rahman et al. (RAHMAN; COSTA DE CAMARGO; SHAHIDI, 2018) with modifications. In a centrifuge tube, 200 µL of sample extract was mixed with 200 µL of a phosphate buffer solution (200 mM, pH 6.6) and subsequently to which 100 µL of potassium ferricyanide (1%, w/v) were added. The mixture was incubated at 50 °C for 20 min, and then 180 µL of 10% trichloroacetic acid was added. Solution of FeCl<sub>3</sub> 20 µL of 0.1% (w/v) was added in each tube with subsequent addition of 500 µL phosphate buffer solution (200 mM, pH 6.6). The absorbance of the reaction mixture was read at 700 nm using a spectrophotometer. The standard curve was prepared using Trolox. The percent of reducing power (RP) was calculated using the following equation, RP (%) = [(Ab<sub>c</sub> - Ab<sub>s</sub>) / Ab<sub>s</sub>] x 100, where Ab<sub>s</sub> is the absorbance of the sample and Ab<sub>c</sub> is the absorbance of control. The results were expressed as the same for IC<sub>50</sub> (mg/mL) based on the percentage's linear regression.

### 2.6.5 Total antioxidant capacity: Formation of a Phosphomolybdenum Complex

The total antioxidant capacity (TAC) was carried out according to Prieto et al. (PRIETO; PINEDA; AGUILAR, 1999) with modifications. Of each extract 100µL was added in a centrifuge tube with 1 mL of phosphomolybdenum solution (sodium phosphate 0.498 + ammonium molybdate 0.494 + 60 mL of sulfuric acid) previously prepared. The mixture was incubated at 95 °C for 90 min. After the incubation period, 200 µL aliquot

of each tube was transferred to a 96-well plate, and absorbance read at 700nm. Ascorbic acid diluted in methanol (1mg/mL) was used as a positive control, following the test samples' same procedures. The total antioxidant capacity (TAC) of the extracts was calculated using the following equation,  $TAC = [(Ab_s - Ab_b) / (Ab_{ac} - Ab_b)] \times 100$ , where  $Ab_s$  is the absorbance of the sample,  $Ab_b$  is the absorbance of blank and  $Ab_{ac}$  is the absorbance of ascorbic acid. The results were expressed as the same for  $IC_{50}$  (mg/mL) based on the percentage's linear regression.

## 2.7 Statistical analysis

The assays were performed in triplicate, and the values were considered significantly different at  $p < 0.05$ . The data were analyzed using the Graph Pad Prism® version 5.0 and expressed in mean  $\pm$  S.D. Statistically significant differences were calculated by one-way analysis of variance (ANOVA) followed by Turkey posthoc test.

## 3. Results and Discussion

### 3.1 Proximate composition and mineral analysis

The residual cake from *Syagrus schizophylla* (RCS) showed significant levels of macronutrients (Table 1 and 2). The RCS consists of 47.44% saturated fatty acids (SFAs) (Table 2), with 12.9% of lauric acid C12:0 (LA). Although studies point to the pro-inflammatory nature of SFAs, mostly long-chain saturated fatty acids, Santos et al. (2019) point out that LA's intake contributes positively to the increases ApoA-1 and *High-density lipoprotein* (HDL) (SANTOS, Heitor O. *et al.*, 2019). That may occur through two metabolic pathways, as a substrate for the formation of Apo-A, and reducing HDL catabolism with the increased cholesterol reverse transport Apo-A production (Cardoso, Moreira, De Oliveira, Luiz, & Rosa, 2015; Hayek *et al.*, 1993). Through clinical trials, it was observed that the substitution of 1% diet with an increase in L.A. did not show a significant rise in ApoB 5.6 mg / L associated with *low-density lipoprotein* (LDL) while a significant increase in ApoA-1 was observed in 13.8 mg / L (MENSINK *et al.*, 2003). Another important factor regarding the consumption of medium-chain fatty acids, such as L.A., is their low storage rate via adipocytes. Studies point out that the consumption of diets with concentrations of medium-chain fatty acids does not influence weight gain correlated with metabolic disorders (MCCARTY; DINICOLANTONIO, 2016).

Fibers (25.59%) and protein (20.89%) showed to be similar to other Brazilian oilseeds (JOSHI; LIU; SATHE, 2015). Fiber intake has been linked to improvements in

risk factors associated with metabolic disorders such as hypercholesterolemia, hypertriglyceridemia, hyperglycemia, body weight, appetite, and the relationship between dietary fiber and decreased arterial blood pressure values (ALEIXANDRE; MIGUEL, 2016). In addition to the benefits cited, fiber-rich foods or supplements are generally recommended for various gastrointestinal functional disorders, such as irritable bowel syndrome, constipation, and diverticular disease. Because of this evidence, daily consumption of fiber is recommended of 25 g/day and 30 g/day for males and females between the ages of 19 and 50 years (KORCZAK *et al.*, 2017).

The main protein groups present in RCS are albumins and globulins,  $22.9 \pm 0.04$  and  $11.82 \pm 0.02$ , respectively, corroborated with Kwon *et al.* (KWON; PARK; RHEE, 1996) following by prolamine and glutein totaling approximately 7% (Table 1).

According to elemental analysis of RCS Fe, Zn, K, Na, Ca, Mg, Cu and P are present in higher concentration in RCS, possible the presence of outer kernel layers makes the concentration of minerals increase, when compared to cereals, as wheat flour (RAGAEE; ABDEL-AAL; NOAMAN, 2006). In particular, three mineral elements are most prominent, potassium, calcium, and phosphorus (Table 3). As observed by Prasad *et al.*, the potassium value exceeds calcium concentration in most plants (PRASAD *et al.*, 2000). Potassium is an essential element in maintaining the body's extracellular fluid, acting on muscle nerve impulse and muscle contraction, and maintaining blood pressure (MIR-MARQUÉS; CERVERA; DE LA GUARDIA, 2012). In contrast, phosphorus is a major component of phytic acids, an anti-nutritional element present in most unrefined plant organic elements, and it is part of dietary fiber. However, in general, food processing can degrade phytate by releasing inorganic phosphate, which increases the digestible phosphorus (DP) content in the product (ITKONEN *et al.*, 2012). Metal such as copper and zinc may enter food from soil mineralization by crops, and low concentration levels imply contamination-free planting (ADELAKUN *et al.*, 2012). The Cu is one of the essential trace elements associated with many enzymes. Zn has a critical role in cell division, protein synthesis, and growth, which is very important for infants and children (OBINNA-ECHEM; BEAL; KURI, 2015).

### 3.2 Residual biomass characterization

The FTIR spectrum of RCS (Figure 1) shows transmittance in ten different regions; such regions characterize chemical groups present in the set of proteins and lipids found in the residual biomass (SIANO *et al.*, 2019). The absorption peak  $3313\text{ cm}^{-1}$

(3500-3000 cm<sup>-1</sup>) in region 1 is attributed to stretching vibration of N-H (amide) and free hydroxyl O-H (SIANO *et al.*, 2019); this region is also attributed to a relative humidity of the sample (TEIXEIRA *et al.*, 2018b). Region 2 and 3 correspond, respectively, to the vibration of asymmetrical stretching of C-H<sub>2</sub> (2924 cm<sup>-1</sup>) and C-H<sub>3</sub> (2854 cm<sup>-1</sup>). Also, region 4 (1745 cm<sup>-1</sup>) shows functional groups of ester (C=O) all related to the lipid fraction present in the biomass (TEIXEIRA *et al.*, 2018a; ZHENG *et al.*, 2017). From regions 5 and 6, we can identify groups referring to the protein content present in the RBSS. The region 5 (1643 cm<sup>-1</sup>) is attributed to stretching C-O of amide I (LEÃO, Daniela P. *et al.*, 2017; ZHENG *et al.*, 2017).

According to Chen et al., peaks between regions 1700-1600 cm<sup>-1</sup> correspond to structural disorganization of the protein, which may reach a fold of the betas leaves (CHEN *et al.*, 2013). Region 6 (1541 cm<sup>-1</sup>) is relative to the amide II by stretching C-N and bend N-H protein (CHEN *et al.*, 2013; TEIXEIRA *et al.*, 2018a). According to Zhang et al., this region is mostly represented by 60% N.H. bend against 40% of C-N (ZHANG *et al.*, 2015). According to the author, this division does not do so for the amide III conformation present in region 8 (1248 cm<sup>-1</sup>), where 30% corresponds to N-H and 40% C-N. In region 7 (1377 cm<sup>-1</sup>), deformation of C-H with C-O stretching could be representing the presence of aromatic polysaccharide ring (cellulose); a similar area was observed in cellulose fiber from peanut oil cake (SUMESH; KANTHAVEL; KAVIMANI, 2020). Region 9 and 10 (1200–800 cm<sup>-1</sup>) have polysaccharide ring vibrations in C–O–C. This region allows us to indicate the index of crystallinity and order of the carbon chains. The absence of peaks 1045, 1022, and 995 cm<sup>-1</sup> (amorphous regions) allows us to infer that the polysaccharide in the RCS samples is found with a higher degree of crystallinity (GUO *et al.*, 2020). This condition can be observed in the XRD profile (Figure 2); the RCS display diffraction pattern peaks at 5.29°, 15.9°, 17.89, 18.58°, and 20° 2θ. The high intensity of the peak 2θ = 20° marked the presence of the amylose-lipid complex, structural condition generally seen in type-B starch granules. The presence of lipid in starch has an effect on the structural, physical and biological properties of starch. The amylose-lipid complex interferes with starch's bioavailability when ingested, decreasing the susceptibility of enzymatic hydrolysis of amylose, decreasing solubility and digestibility with a direct effect on the glycemic index (LI *et al.*, 2016) considering the amorphous region between 2θ = 10 °- 15°. The relative crystallinity (RC) in RCS was 75% (HUNG; VIEN; LAN PHI, 2016; ZHANG *et al.*, 2016).

### 3.2 Functional properties

Results of water absorption index (WAI), water solubility index (WSI), and oil absorption index (OAI). The WAI and WSI can be attributed to the interaction of polymeric chains of starch granules (LI *et al.*, 2016). The RCS exhibited low water absorption  $4.14 \pm 1.92$  with the weakest water holding capacity. These properties were correlated with large-sized B-type starch granules, restricting the granular absorption by complex amylose-lipid (SHANG *et al.*, 2020). This result is supported by Liu et al. and Kusumayanti et al., who reported the low water absorption in flours with high amylose content (KUSUMAYANTI; HANDAYANI; SANTOSA, 2015; LI *et al.*, 2016). According to Kumoro et al., it is not directly correlated with absorption capacity and solubility index. However, factors such as inter-associative forces within the amorphous and crystalline domains, and mineral elements like phosphorous may influence solubility degree (KUMORO *et al.*, 2012). The RCS showed a WSI of  $1.59 \pm 0.78$  g/100g; this value is in agreement with other oilseed such as almond (WHC 1.49), pecan (WHC 1.45), and macadamia nuts ( WHC 0.66) (BARAHENG; KARRILA, 2019; JOSHI; LIU; SATHE, 2015). The relative low index of OAI  $4.18 \pm 0.78$  can be the complexation of the lipid structures within the amylose helix, meaning that the oil's hydrophobic designs are not entirely free to guarantee more excellent oil absorption (TEIXEIRA *et al.*, 2018a).

### 3.2 Antioxidant content

Antioxidant metabolites' consumption is associated with the prevention and regulation of severe clinical conditions, such as cardiovascular disease, cancer, type-2-diabetes, and other chronic diseases (KASKA; ÇİÇEK; MAMMADOV, 2019). Phenolic groups are the main molecules involved in the antioxidant potential in natural products. The phenolic compounds act through redox mechanisms by transferring electrons or hydrogen atoms and chelation of metal ions, and inhibiting oxidase activities (RAHMAN; COSTA DE CAMARGO; SHAHIDI, 2018). In this study, the eight extracts produced were evaluated for the content of phenolic compounds (TPC), flavonoids (TFC), and potential antioxidants in DPPH tests, reduce power (RP), and total antioxidant capacity by phosphomolybdenum (TAC), as shown in table 4.

As expected, the extracts showed variations in the concentrations of phenolic and flavonoids according to the solvents' polarity reflected in the antioxidant activity. The ethyl ether, ethyl acetate, chloroform, and dichloromethane extracts present a higher concentration of phenolic and flavonoids and a lower IC50 in tests for chelation of metal

ions (RP and TAC). Except for dichloromethane, the other extracts showed no activity in reducing the DPPH agent. These results indicate that the extracts produced from the residual biomass of *S. schizophylla*, in the tested concentration (5 mg), do not act as a reducing agent through the donation of H<sup>+</sup> for the radical 2,2-diphenyl-1-picryl-hydrazyl (DPPH<sup>•</sup>) but being able to reduce Fe<sup>+3</sup> ions (RP) with variation in the IC<sub>50</sub> of 2.69 to 4.43 mg/ml. This dependence according to the polarity of the solvent was also observed by Santos et al. (SANTOS, Perla M. *et al.*, 2018). Although the hexane extract possesses the lowest degree of polarity within the group of solvents, Suárez et al. concluded that only 2% of the phenolic compounds are removed in this fraction, where they mostly extract lipid content, as previously observed in this work. According to the authors, 98% remains trapped in solid waste (SUÁREZ *et al.*, 2009).

One of the primary factors in retaining phenolic groups in solid waste is the concentration of fibers; phenolic compounds can bind to food fibers and alter their accessibility (JAKOBÉK; MATIĆ, 2019). In general rules, dietary fibers and phenolic compounds present significant OH- groups' bonding, allowing the bonding by hydrogen bonds and the formation of aggregates through hydrophobic bonds between the fibers and some phenolic compounds (LIU, J. *et al.*, 2018; SAURA-CALIXTO, 2011). According to Liu et al., the release of these fractions of phenolic compounds and dietary fibers occurs under gastric pH 2 and intestinal pH 7 (LIU, Chengzhen *et al.*, 2016). Another critical point is the interaction of amylose in starch with phenolic compounds; according to Chai et al., this interaction contributed to reducing the solubility of this compound in water (CHAI; WANG; ZHANG, 2013). The possible interaction of these phenolic compounds with the structure of fibers and amylose has benefits for the lower region of the intestine (colon), where they are released from the matrix of dietary fibers (JAKOBÉK; MATIĆ, 2019).

#### **4. Conclusion**

According to the data obtained in this study, the solid residue after extraction of the vegetable oil of *Syagrus schizophylla* showed important macronutrient contents (proteins, carbohydrates, and fibers) and significant values of minerals, presenting a considerable range of potassium and phosphorus. Regarding the antioxidant evaluation, it was possible to observe the presence of phenolic compounds of hydrophobic character, being possible their interaction in the fiber structure of the *Syagrus schizophylla* residue, allowing intestinal transit with direct action in the lower

intestine. The presence of the amylose-lipid complex guarantees a slower digestibility favoring glycemic control.

## 5. Acknowledgements

The authors thank the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES-Brazil) for financial support and the Coruripe Power Plant for providing the fruits of Aricuriroba.

<b>Peak</b>	<b>Compound</b>	<b>RI Cal</b>	<b>%</b>
1	Octanoic acid (C:8)	4.55	10.15± 0.06
2	Decanoic acid (C:10)	6.29	6.39± 0.08
<b>3</b>	<b>Dodecanoate acid (C:12)</b>	<b>7.84</b>	<b>43.56 ± 0.23</b>
4	Tetradecanoic acid (C:14)	14.53	15.08 ± 0.18
5	Hexadecanoic acid (C:16)	19.43	7.35 ± 0.05
<b>6</b>	<b>Cis-9-Octadecenoic acid (C18:1)</b>	<b>23.61</b>	<b>10.35 ± 0.27</b>
7	cis-9,12-Octadecadienoic acid (C18:2)	23.72	3.42 ± 0.23
8	Octadecenoic acid (C:18)	24.16	3.70 ± 0.02
<b>Total (%)</b>			100%

**Table 1:** Fatty acid profiles of the hexane extract of residual cake from *Syagrus schizophylla*. In Highlighting in bold the major components. RI Cal = Experimental retention indices relative

	<b>Moisture</b>	<b>Ash</b>	<b>Proteins</b>	<b>Lipids</b>	<b>Fiber</b>	<b>Carbohydrate</b>	<b>Energetic value</b>
<b>RCS</b>	5.05 ± 0.14	6.27 ± 0.01	20.89 ± 0.45	30.10 ± 0.64	25.59 ± 0.07	12.02 ± 0.55	402.54 ± 1.36

<b>Total protein fraction extract (%)</b>				
<b>Albumins</b>	<b>Globulins</b>	<b>Prolamins</b>	<b>Gluteins-1</b>	<b>Gluteins-2</b>
22.9 ± 0.03	11.85 ± 0.02	6.30 ± 0.01	0.04 ± 0.005	1.32 ± 0.001

**Table 2:** Proximate composition analyzed and concentrations of groups proteins present in the residual cake from *Syagrus schizophylla*. For proximate composition, results are expressed in g/100g, and the Energetic value is expressed in kcal/100g. Results expressed as mean ± standard deviation (n=3), p < 0.005.

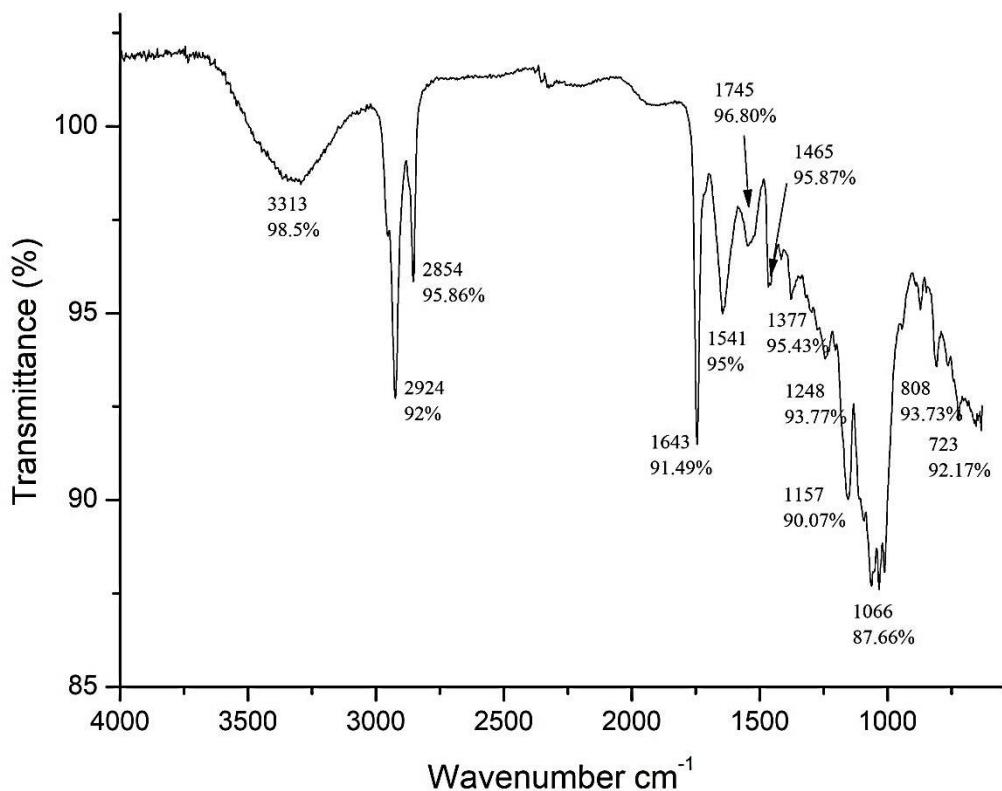
	<b>Fe</b>	<b>Zn</b>	<b>K</b>	<b>Na</b>	<b>Ca</b>	<b>Mg</b>	<b>Cu</b>	<b>P</b>
<b>RDA</b>	8-18	8-11	2,600	1,500	1,000	310-400	900	1,250
<b>RCS</b>	97.61 ± 1.06	40.05 ± 1.03	8643 ± 1.00	137 ± 1.09	507 ± 1.55	35.18 ± 1.04	24.14 ± 0.98	5293 ± 1.04

**Table 3:** Mineral profile of residual cake from *Syagrus schizophylla* (mg/kg of mineral);

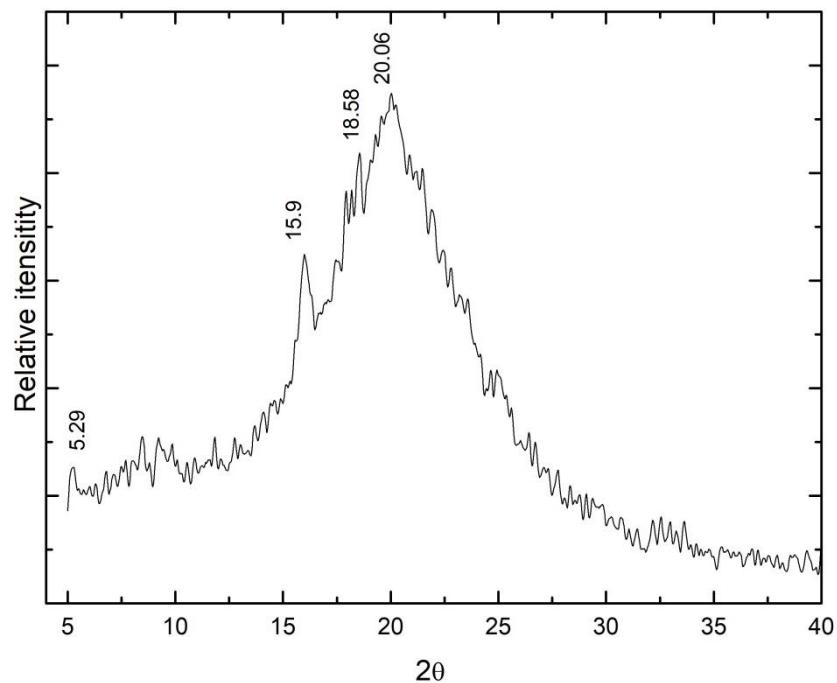
RDA: recommended Dietary Allowances for adults 19-30 years of age (mg/d). Values expressed in mg/kg and the results are expressed as mean ± standard deviation (n=3), p < 0.005.

<b>Samples</b>	<b>TPC</b>	<b>TFC</b>	<b>DPPH</b>	<b>RP</b>	<b>TAC</b>
Hexane	15.30 ± 3.40	7.43 ± 0.03	ND	8.71 ± 0.9	116.36 ± 0.18
Ethyl ether	<b>42.98 ± 0.00</b>	<b>5.67 ± 0.33</b>	ND	<b>4.25 ± 0.00</b>	<b>65.91 ± 0.25</b>
Ethyl Acetate	<b>41.01 ± 0.50</b>	<b>6.72 ± 0.15</b>	ND	<b>4.13 ± 0.00</b>	<b>65.61 ± 0.05</b>
Chloroform	<b>41.45 ± 1.90</b>	<b>6.72 ± 0.15</b>	ND	<b>2.69 ± 0.13</b>	<b>65.30 ± 0.00</b>
Dichloromethane	<b>46.71 ± 0.95</b>	<b>7.56 ± 0.07</b>	4.18 ± 0.05	<b>4.43 ± 0.00</b>	<b>13.84 ± 0.08</b>
Acetone	36.89 ± 0.61	6.42 ± 0.07	5.80 ± 0.01	9.25 ± 0.28	24.65 ± 0.02
Methanol	ND	ND	ND	ND	ND
Distilled water	ND	ND	ND	ND	ND
<b>Standard antioxidants</b>					
Trolox®	NT	NT	0.05 ± 0.01	NT	NT
Ascorbic Acid	NT	NT	NT	0.08 ± 0.00	0.52 ± 0.00

**Table 4:** TPC: Total phenolic compound in equivalent gram of galic acid per gram of extract (g GAE/g); TFC: Total flavonoids compounds in equivalent gram of querectin per gram of extract (g QUE/g); DPPH: Radical 2,2-Diphenyl-1-picrylhydrazyl in IC<sub>50</sub> (mg/mL); RP: Reduce Power in IC<sub>50</sub> (mg/mL), TAC: Total antioxidant capacity in IC<sub>50</sub> (mg/mL); ND: could not be determined; NT: Not tested



**Figure 1:** Infrared spectrum of residual cake from *Syagrus schizophylla* in the region of 4000-500  $\text{cm}^{-1}$



**Figure 2:** Diffractogram showing the X-ray profile from the residual cake of *Syagrus schizophylla*.

## **Artigo II - Vegetable oil from *Syagrus schizophylla* (Mart.) Glasman: Brazilian alternative for biofuel production**

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The demand for transport fuel is growing worldwide, which promotes impact with CO<sub>2</sub> emission on the environment. In this perspective, researchers for new resources for use as an alternative fuel are intensified during the years. The use of biomass for biofuel production in the substitution of oil has been considered an essential element for achieving the goals established in Paris Agreement – Agenda 2030. Biodiesel is one of the most promising substitutes of diesel fuel with production from vegetable oil. In this way, this study aims to evaluate the potential of *Syagrus schizophylla* oil on the production of crude biodiesel (B100). The biodiesel produced in this study demonstrated parameters according to the international standard (ASTM and EN) even with 53% of rate conversion. Using the Rancimat method, biodiesel from *S. schizophylla* oil should important oxidative stability reaching 40h for the induction period. As an important conclusion in this paper, the fixed oil from *S. schizophylla* oil could be included as an alternative to complement the biofuel sector in Brazil. It can be a way to increase the income of local communities that present this palm, such as the Brazilian northeast.

**Key-words:** Generation biofuel. Sustainability development. Bioeconomy. Biomass fuel.

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## 1. Introduction

The combustion of crude oil and oil fuels is the most responsible for pollutants present in air pollution and greenhouse gas (GHG) (DECHAMBRE; THIEN; BARDOU, 2017; MOAZENI; CHEN; ZHANG, 2019; REHAN *et al.*, 2018; TORRES-GARCÍA *et al.*, 2019). Air pollution is a complex of particles (PM<sub>10</sub>, PM<sub>2.5</sub>, PM<sub>1</sub>, and Ultrafine particle – UFP) and gaseous pollutants (NO<sub>2</sub>, SO<sub>2</sub>, O<sub>3</sub>, and CO) (KUMAR *et al.*, 2019), causing several health problems like cardiovascular and respiratory disease and yet studies indicate possible neurological damage (AUGUSTUS DE MELO; DE MARTINO JANNUZZI; DE MELLO SANTANA, 2018; HEALTH EFFECTS INSTITUTE, 2019; PETERS *et al.*, 2019; POWER *et al.*, 2016; WARBURTON *et al.*, 2019).

In addition to the damage to human health, the increase in greenhouse gases has caused a collapse in the ecosystem, climate change, and global temperature (BHATIA *et al.*, 2019). The current scenario governments in 21<sup>th</sup> Conference on Climate Change (COP 21)- Paris Agreement fixed goals and responsibilities for countries to reduce CO<sub>2</sub> emissions, limit the temperature increase to below 2°C above pre-industrial levels, and to endeavor to limit the temperature rise to 1.5 ° C above pre-industrial levels (NATIONS, 2015; UNFCCC, 2015). The use of biomass for biofuel production in the substitution of oil has been considered a key element for achieving the Agenda 2030 (KARAGULIAN *et al.*, 2015). It is estimated that by 2030 the global consumption of fossil fuel will be replaced by biofuel between 4% and 7% (DA SILVA CÉSAR *et al.*, 2019).

In the context of the Paris Agreement, the Brazilian government has committed to increasing the share of sustainable bioenergy in its energy matrix to approximately 45% of renewable energy in the composition of the energy matrix in 2030. In this scenario, the country estimates reducing greenhouse gas emissions by 37% in 2025, with a subsequent reduction of 43% below 2030 (BRAZIL, 2015). Brazil is one of the largest producers and consumers of biodiesel. In 2015 the country produced 3.9 million m<sup>3</sup>, surpassed by the United States, producing 4.8 million m<sup>3</sup> (DA SILVA CÉSAR *et al.*, 2019; DOS SANTOS ALVES; BELARMINO; PADULA, 2017). According to the National Petroleum, Gas and Biofuel Agency (ANP), between 2005 and 2019, biodiesel production in Brazil went from 730 million m<sup>3</sup> to 5.9 billion m<sup>3</sup> in 2019, with vegetable oil from soy (70%) being

the primary raw material used for conversion to biodiesel, followed by 11% animal fats and other fatty materials 8.87%.

Although vegetable oils are economically essential and fatty acid compositions have been well documented, the world's agricultural and oil industries use only a few species (PINHO; OLIVEIRA; SILVA, 2009). The choice of which raw materials should be used is mainly based on an economic vision that includes companies producing biodiesel seeking competitiveness in the market, overlapping incentives, and attempts by the government to diversify (DOS SANTOS ALVES; BELARMINO; PADULA, 2017; LEÃO, Raphael Riemke de Campos Cesar; HAMACHER; OLIVEIRA, 2011; PADULA *et al.*, 2012). To encourage more efficient use of the land and raise incomes and the National Program for Production and Use of Biodiesel (Programa Nacional de Produção e Uso de Biodiesel - PNPB) also aims to promote diversification of feedstock sources, social inclusion, and regional development for small rural farmers (BERGMANN *et al.*, 2013; DA SILVA CÉSAR; OTÁVIO BATALHA, 2010; SCHAFFEL *et al.*, 2012; VILLELA *et al.*, 2014).

The Northeast of Brazil has a different phytogeographic domain where it is possible to discover new vegetable species with different valuation and economic value. The Northeast region registers some of the lowest human development levels and economic opportunities in Brazil. The discovery of new renewable sources for energy use can promote the sustainable development of the region. Based on this background, the present study aimed to study and analyze the potential of *Syagrus schizophylla* (Mart.) Glasman oil, a tree palm present in Northeast Brazil, as an economical alternative for biodiesel production.

## **2. Methodology**

### 2.1 Seed sampling and preparation analysis

The nuts of *S. schizophylla* (Mart.) Glasman was manually harvested from Coruripe Power Plant, Industry Ordinance, Alagoas, Brazil. The voucher specimen (Nº 55.148 BR-AL-CORURIPE, B.O. VERAS) was deposited in the Herbarium of the Biology Department of Universidade Federal Rural de Pernambuco (UFRPE). The nuts of *S. schizophylla* were processed in a hydraulic press to remove the most quantity of vegetable fix oil (SSOil).

To identify the fatty acids in SSOil was converted into fatty acid methyl esters (FAME) using BF3 protocols (PINHO; OLIVEIRA; SILVA, 2009). The fixed oil

(0.057g) was firstly suspended in chloroform and added 2 ml of sodium hydroxide in methanol (0.5 mol/L). The reaction was maintained under constant stirring in reflux for 5 minutes at 100°C. The catalyst BF<sub>3</sub> (2 ml) was added to the solution and kept over the same conditions for 30 minutes. At the end reaction, the FAME fraction was recovered by adding 4 ml of *n*-heptane (chromatographic grade) and 15 ml of a saturated NaCl solution then filtered. The excess humidity was removed by the addition of 0.1 g Na<sub>2</sub>SO<sub>4</sub> anhydride. The filtered samples were stored until chromatographic analyses in GC-FID Agilent Technology 7890.

## 2.2 Gas chromatography analysis

The fatty acid profile was identified by gas chromatography (GC). For the procedure, an Agilent Technologies Gas Chromatograph (Palo Alto, CA, USA) 5975C series, with triple quadrupole detection system equipped with DB-5MS column Agilent Technologies (30 m × 0.25 mm x 0.25 µm) was used. The oven temperature was set to rise to 150°C at 2°C/min and rise to 280°C and held for 6 min with an injection temperature of 230°C and an interface temperature of 260°C. Fixed oil was previously eluted in n-hexane, and 1 µL was injected in the gas chromatography coupled to CG-MS mass spectrometry. The quantification of fatty acids was performed in a Thermo Trace GC Ultra system (Thermo Scientific, Milan, Italy) equipped with a flame ionization detector (GC-FID) with column VB-5 (30mm x 0.25 mm). The injection of samples occurred under the same conditions as GC-MS. The FAME (Mix C4-C22) mix fatty acid standard was used and compared by the similarity of mass spectra in available libraries (NIST08 and Wiley Registry™ 9th Edition) and retention Index to those of authentic standards available in the Adams (2011).

## 2.3 Biodiesel production – Acid Transesterification

Biodiesel from *S. schizophylla* (SSBD) was obtained by acid transesterification process according to Hasni et al. with modification (HASNI *et al.*, 2017). Fixed oil (34g) was added in a 250 ml round-bottom flask equipped with a reflux condenser and a heating mantle with a magnetic stirrer. A solution with methanol (CH<sub>3</sub>OH) and sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) was previously prepared and added to fixed oil following the molar ratio of 6:1 (CH<sub>3</sub>OH: TGA). The reaction was kept *overnight* under constant stirring in reflux at 60°C. At the end of transesterification, the solution was transferred to another flask to separate the Fatty Acid Methyl Ester -FAME (biodiesel) to glycerol.

The biodiesel was recovered and mixed with 100 mL of sodium bicarbonate in ethyl acetate to neutralize possible residue of unreacted catalyst. Methanol was removed using a rotary evaporator at 70°C. Biodiesel yield was calculated using the following equation (Eq. 1) (M. F. ELKADY, AHMED ZAATOUT, 2015)

$$(Eq.1) \quad \text{Yield (\%)} = \frac{\text{Weight of biodiesel (g)}}{\text{Weight of oil (g)}}$$

## 2.4 Analytical methods

### 2.4.1 FT-IR spectroscopy

Fourier transform infrared spectroscopy was analyzed with diffuse reflectance mode (DRIFTS) using Bruker IFS66® spectrometer. Data were recorded in the range of 400 to 4000cm<sup>-1</sup> wavenumbers with a spectral resolution of 4 cm<sup>-1</sup>. Data were plotted as transmittance (%) in the wavenumber (cm-1) function and analyzed using GraphPad Prism 6.0.

### 2.4.2 Nuclear magnetic resonance (NMR)

For evaluation in Nuclear magnetic resonance (NMR), the SSBD was previously diluted in deuterated chloroform, and TMS was used as an internal standard. Relaxation time was measured using an inversion-recovery pulse sequence. <sup>1</sup>H NMR spectra were obtained with a pulse duration of 30°, a recycle delay of 1.5s. <sup>13</sup>C NMR was recorded with a pulse duration of 30°, recycle delay of 2s. The data were analyzed using MestreNova software (Version 11.0, Mestrelab Research S. L.). To calculate the rate of conversion of triglycerides in FAME was applied following equation.

$$(Eq. 2) \% C_{ME} = [(6xI_{ME})/9xI_{\alpha-CH_2}] \times 100 \quad (DONNELL \text{ } et \text{ } al., 2013; SAMIOS \text{ } et \text{ } al., 2009)$$

Where % C<sub>ME</sub> is the percentage of methyl ester, I<sub>ME</sub> is the integration value of the methyl ester peak, and I <sub>$\alpha$ -CH<sub>2</sub></sub> is the integration value of the methylene group.

## 2.4. 3 Characterization of physical-chemical fuel properties

The physical and chemical properties of *S. schizophylla* oil and biodiesel were determinate according to specifics parameters according to the American Standard of Biodiesel (ASTM D6751) and European Standard of Biodiesel (EN14214). Density, kinematic viscosity, acid values, and saponification index, was determinate for both products. Biodiesel was also evaluated for Iodine value (IV), Cetene number (CN), and

Higher heating value (HHV) through mathematical models previously described in the literature.

$$(Eq. 3) \quad IV = 0.6683 \times DU + 25.0364 \quad (WANG, Li Bing *et al.*, 2012)$$

$$(Eq. 4) \quad CN = 46.3 + (5458/SV) + 0.225(IV) \quad (KRISNANGKURA, 1986)$$

$$(Eq. 5) \quad HHV = 49.3 - [0.041(SV) + 0.015(IV)] \quad (DEMIRBAŞ, 1998)$$

As expressed in the formulas above,  $A_i$  is the percentage of each component.  $DU$  is the number of double bonds,  $MW_i$  is the molecular mass of each component, and  $SV$  is saponification calculated by ASTM standard method.

#### 2.4.4 Thermal Gravimetric Analysis (TGA)

The thermal analysis of SSBD was carried out using thermal gravimetric analysis (TGA-PERKIN ELMER, STA 6000) with a heating rate of 10°C/min starting from ambient conditions up to 900°C.

#### 2.2.5 Oxidative stability Assay - Rancimat method

The study of oxidative biodiesel stability was performed using the Rancimat method as described in EN14112. Biodiesel sample, 3g was weighed in a glass test tube and analyzed under constant airflow of 10 L/h within a thermostat-controlled block heater at 110°C. During the test, the volatile products formed during the oxidation process were transferred to a flask with distilled water, a change in the recorded electrical conductivity. Induction time was defined as the time taken from the start of the test to the onset of the oxidation period

### 3. Results and discussion

#### 3.1 Transesterification reaction analysis

The transesterification results can be observed through analytical methods, such as FTIR and NMR, to indicate the ideal conditions during this process. Figure 1 shows the oil and biodiesel spectrogram, with similar peaks in some reading regions. A discreet peak in the  $3474 \text{ cm}^{-1}$  could be observed only in the SSOil sample, corresponding to the  $-OH$  stretching vibration of water molecules in pure oil (FOCKE; WESTHUIZEN; OOSTHUYSEN, 2016; KUDRE; BHASKAR; SAKHARE, 2017a). The presences of moisture as well as the concentration of free fatty acid content in the oil are predisposal

factors for the formation of soap, as well as redirecting the reaction to the hydrolysis of the biodiesel when in contact with primary catalysts decreasing the yield and the conversion rate of TGA in FAME (YAAKOB *et al.*, 2013). It is possible to visualize the acid value (2 mgKOH / g) in table 2, representing the concentration of free fatty acid present in the SSO. In this case, acid catalysts are recommended as an alternative to avoid secondary reactions (SAMIOS *et al.*, 2009).

Absorption bands in region 1425-1447 cm<sup>-1</sup> are observed only in SSBD, not vegetable oil. The peak 1437 cm<sup>-1</sup> is associated with asymmetric stretching of O-CH<sub>3</sub> for methyl ester (KUDRE; BHASKAR; SAKHARE, 2017a). The peaks 1160 and 1055 in biodiesel spectrum are due to O-CH<sub>3</sub> extension and indicate that the product results from oil conversion (ABDALLA, 2018; HASNI *et al.*, 2017; KUDRE; BHASKAR; SAKHARE, 2017b). According to some authors, some regions such as 1370-1400 cm<sup>-1</sup> are present in feedstock oil but absent in biodiesel (MAHAMUNI; ADEWUYI, 2009; SIATIS *et al.*, 2006). Although this condition was not observed in our results, both SSBD and SSOil samples show peaks in this region, implying mono-, di- and triglycerides groups in both samples. This region is associated with asymmetric stretching of O-CH<sub>2</sub> (KUDRE; BHASKAR; SAKHARE, 2017b), suggesting that the transesterification reaction was not complete. Samios et al. suggested that transesterification reaction do not occur in ideal form with the formation of three methyl ester molecules and one glycerol. The authors suggested the transesterification results in byproducts that does not considered of stoichiometric parameter and resulting in a complex of tri-, di- and monoglycerides, biodiesel ester, glycerol, alcohol and catalytic (SAMIOS *et al.*, 2009). To minimize these byproducts, the alcohol and TGA molar ratio should be reviewed, included with catalyst and temperature, time, and pressure conditions. Other regions can be observed in both samples corresponding to the C=O bond (1700-1800 cm<sup>-1</sup>) and CH stretching of olefins (2800-3000 cm<sup>-1</sup>). These results obtained by the FTIR are in agreement with that observed by Rosset et al. (ROSSET; PEREZ-LOPEZ, 2019).

In order to evaluate the fat conversion rates in FAME, the <sup>1</sup>H NMR and <sup>13</sup>C NMR techniques were applied in the SSBD sample. Both spectrum are demonstrated in Figure 2. In <sup>1</sup>H NMR, the appearance of a singlet peak at 3.69 ppm and a triplet of 2.35 ppm mark the presence of methoxy proton group (-O-CH<sub>3</sub>) and  $\alpha$ -carbonyl methylene proton ( $\alpha$ -CH<sub>2</sub>). These two peaks are the distinct peaks for confirming triglycerides fractions in FAME (TARIQ *et al.*, 2011). Melo et al. showed that through NMR that the main difference in <sup>1</sup>H NMR spectrum during the conversion process is the appearance of a new

peak at 3.67 ppm that is when methyl fatty acid is produced, the other peaks remain in the same position and same area when compared with fixed oil (MELLO *et al.*, 2008). The olefinic protons (-CH=CH-), also observed in FTIR, imply that unsaturated fatty acid appears and is visible in the 5.37 ppm region. The integrated areas of methoxy group protons in the methyl ester and the  $\alpha$ -carbonyl methylene groups in  $^1\text{H}$  NMR were used according to Eq.2 (Topic 2.4.2) to quantify the rate conversion of methyl ester by acid transesterification. The percentage conversion of triglycerides in FAME was found to be 53.13%. This conversion rate emphasizes the FTIR previously observed and is below that required by international standards. Factors of reaction time, methanol to oil molar ratio, catalyst concentration contribute directly to conversion oil in methyl ester (M. F. ELKADY, AHMED ZAATOUT, 2015), in face that its requiring adjustments in the conditions of transesterification.

The  $^{13}\text{C}$  NMR chemical shifts of lipids are grouped in regions of carbonyl and carboxyl (172-178 ppm), unsaturated carbons (124-134 ppm), glycerol carbons (60-72 ppm), and aliphatic carbons (10-35 ppm) (ALEXANDRI *et al.*, 2017). The signals at 22-35 ppm are related to methylene carbons of long carbon chain in fatty acid methyl ester (SÁNCHEZ-ARREOLA; BACH; HERNÁNDEZ, 2019). Peaks of 174.40 ppm and 52.30 ppm are attributed to ester carbonyl (-COO-) and (C-O), respectively (TARIQ *et al.*, 2011).

### 3.2 Biodiesel parameters

The biodiesel performance in diesel engines is related to the intrinsic conditions of the vegetable oil to which it was derived. An ideal constitution is considered by balancing the number of carbon atoms, fatty acid type, chain length, position, and double-bonded isomers (LANJEKAR; DESHMUKH, 2016; WAN GHAZALI *et al.*, 2015). The profile of SSOil and SSBD are shown in Table 1. As observed, both samples have a higher index of saturated elements, with 86.22% in SSOil and 99.53% in SSBD. The presence of saturated elements in biodiesel has a substantial impact on the density, viscosity, calorific value, and CN of biodiesel (HOEKMAN *et al.*, 2012; TORRES-GARCÍA *et al.*, 2019). In diesel engines, density plays an essential role in the optimal combustion of a fuel. Adequate atomization is achieved according to an ideal ratio between air and the volume (mass) injected into the combustion chamber to guarantee an efficient and complete process (AHMAD *et al.*, 2019; ATABANI *et al.*, 2013; KAYA *et al.*, 2009). The SSBD biodiesel, with a predominance of saturated elements, showed a

density of  $856 \text{ kg/m}^3 \pm 0.001$  within the requirements established by international standards (ASTM and EN). Biodiesel from vegetable oils with a high degree of unsaturation tends to show a relatively higher density index than that achieved in this work (SAJJADI; RAMAN; ARANDIYAN, 2016). The density index above the recommended can be observed in biodiesel from castor oil and groundnut oil. In these conditions, the biodiesel presents as the fundamental characteristic higher diameter of the fuel droplets, greater inertia for the fuel injection (time and speed) increasing the amount of fuel injected (mass) (SAJJADI; RAMAN; ARANDIYAN, 2016; WAN GHAZALI *et al.*, 2015). However, the biodiesel fuel density is also influenced by the transesterification process (SINGH, Digambar *et al.*, 2019).

Influenced directly by density, the kinematic viscosity ( $\text{mm}^2/\text{s}$ ) is correlated with ideal conditions for spraying, mixing (fuel: air), and combustion process (KNOTHE, 2005). The application of fuels with high viscosity in diesel engines, such as vegetable fixed oil  $30\text{-}50 \text{ mm}^2/\text{s}$ , implies an increase in combustion time, low thermodynamics, increase carbon emission, incomplete combustion with a waste deposit (ABDALLA, 2018; AHMAD *et al.*, 2019; PARK; SUH; LEE, 2010). In general, vegetable oil is 10 to 17 times more viscous than biodiesel (SAJJADI; RAMAN; ARANDIYAN, 2016). Our study could observe this condition; SSOil showed a viscosity of  $26.25 \text{ mm}^2 / \text{s}$  while SSBD was  $2.73 \text{ mm}^2 / \text{s}$ . According to Golimowiski *et al.*, the viscosity has a positive correlation with the presence of saturated fatty acids (GOLIMOWSKI *et al.*, 2017). However, the viscosity correlates more robustly by the degree of unsaturation and the double bound arrangement, and trans configuration elevated the viscosity than cis configuration (SINGH, Digambar *et al.*, 2019). The problems observed in fuel with high viscosity can be noticed during low surrounding temperature. During the winter season, fuel's high viscosity leads to inefficient combustion and increased exhaust emission due to fuel mixes with air slowly (MUJTABA *et al.*, 2020).

Cetene number (CN), high heating value (HHV), and iodine value (IV) were determined by the empirical method as mentioned before. Most biodiesels are recognized to show high CN; the SSBD shows CN equals 74 above the minimum limit laid down by regulation ASTM 6751-02 and EN 14214. The CN is correlated with ignition delay time within the combustion chamber (ATABANI *et al.*, 2013). Using the same mathematic model to determined CN in this, Ahmad *et al.* study biodiesel of feedstock flaxseed oil and showed CN resulted of 47 (AHMAD *et al.*, 2019). The SSBD showed high value of CN than other methyl esters, such as palm oil (59.5), sesame seed (56.35), soybean

(53.8), and linseed (48) (MUJTABA *et al.*, 2020). The CN results can be associated with the chemical characteristic of fatty acid present in feedstock oil. According to Singh *et al.*, the CN level is directly affected by the degree of unsaturation present in feedstock oil and biodiesel (SINGH, Digambar *et al.*, 2019). CN values relate to the quality of biodiesel combustion in diesel engines.

The HHV or calorific value measures the heat produced by burning a specific quantity of fuel to produce CO<sub>2</sub> and H<sub>2</sub>O at its initial temperature. The SSBD showed an HHV of 41.46 MJ/kg, which is closely in other studies (AHMAD *et al.*, 2019; HARIRAM *et al.*, 2018). The HHV is influenced by high unsaturation than the carbon quantity in the chain length. In biodiesel, the HHV decreases 0.21 Mj/kg by each percentage of unsaturation in methyl ester. The presence of water in biodiesel is another factor for the decline in the calorific value (MUJTABA *et al.*, 2020; SINGH, Digambar *et al.*, 2019) not evidenced by previously infrared analysis.

The iodine value (IV) is related to an index of double bonds in biodiesel and oil feedstock, is a measure of total unsaturation of relative proportion of mono-, di, and tri-unsaturated sample. The IV of SSBD was 36.84 mg I<sub>2</sub>/g than the maximum established by EN 14214. The iodine value shows the tendency of biodiesel to oxidize, polymerize, and form deposits on diesel engines directly correlated with the increase in the level of unsaturation in oil and biodiesel. The explanation for this relation is based on the susceptibility oxidative attack in bis-allylic position within polyunsaturated ester molecules (SAJJADI; RAMAN; ARANDIYAN, 2016). The equation model applied in this study considered the unsaturation degree in correlation to the amount of monounsaturated and polyunsaturated fatty acids (%), as previously predicted by Wang *et al.* (WANG, Li Bing *et al.*, 2012). A similar IV value was obtained by Vedaraman *et al.* in the production of biodiesel from Sal seed oil (VEDARAMAN *et al.*, 2012).

For carbon residue (CR) is used to indicate the range of deposits formed by decomposition and pyrolysis of the fuel components that can clog the engine fuel injectors (ATABANI *et al.*, 2013). In this study, the biodiesel SSBD shows CR equal to 0.02 % w/w, a value under the maximum established by ASTM 6751-02 and EN 14214 standard. This result agrees with the observed values referring to density and viscosity, being parameters linked to the atomization quality and waste deposits in incomplete combustion.

### 3.4 Thermal gravimetric analysis (TGA)

Thermogravimetric analysis (TGA) showing the weight loss during variation of temperature and the derivative of weight loss in relation to the temperature (DTG) (ATABANI *et al.*, 2019) and can be observed in Figure 3. The thermal oxidative degradation is dividing into three steps; the SSBD starts degradation at around 160°C until almost complete vaporization at around 481°C. In this temperature range, the mass loss occurs at levels of 13%, 56%, and 97.82%. According to Ratton et al., the residual weight indicated that the biodiesel does not have many non-volatile materials (RATTON COPPOS; KAHN; BORGES, 2018). In general, the most biodiesel starts to thermally decompose at an average temperature of 150°C until complete oxidative degradation (M. F. ELKADY, AHMED ZAATOUT, 2015). Zhou et al. showed that the increase of residual mass is consistent with more severe oxidative degradation of feedstock that results in the presence of insoluble oligomers that have more excellent resistance to decomposition. The higher levels of insoluble oligomers in biodiesel could leave severe damage in fuel injector fouling and combustion chamber deposits (ZHOU; XIONG; LIU, 2017).

### 3.5 Rancimat induction

Oxidative stability is an important quality indicator parameter for biodiesel (ATABANI *et al.*, 2019). The Rancimat method determines the oxidative stability by detecting volatile oxidative products formed by the sample in contact with atmospheric conditions (BUOSI *et al.*, 2016; DE SOUSA *et al.*, 2019; RODRIGUES *et al.*, 2017). Figure 4 shows the induction period by SSBD, and the result showed an induction period (oxidation onset) of 47 hours. The low degree of oil unsaturation minimizes the sensitivity to oxidation. The number of double bound and the structure of fatty acid methyl ester is associated with the deterioration of the biodiesel, such as the high prevalence of linoleic and linolenic acids decreased oxidative stability (KARAVALAKIS; STOURNAS; KARONIS, 2010). Another essential factor that we can consider here is the contribution of natural antioxidant agents in the vegetable oil of *S. schizophylla*. The vegetable oil is recognized as an important source of biofunctional compounds as specific vitamins C, E, and β-carotenes such as phenolic compounds. The phenolic groups are one of the most significant types of antioxidants (RAMÍREZ-ANAYA *et al.*, 2015). In parallel, a study to evaluates the potential of antioxidants in fixed oil through β-carotene / linoleic acid system was developed by Marco et al.(1968) and adapted by Miller et al. (1971). The result indicated that the fixed oil from *S. schizophylla* has an antioxidant capacity of

60.86% in this system compared to the Trolox standard. This factor associated with the low content of oil unsaturation may contribute to the high stability shown in the Rancimat test.

#### **4 Conclusions**

The biodiesel production from *Syagrus schizophylla* oil was not completed ideal once the FAME rate was under the indicated for standard ASTM 6751-02 and EN 14214, suggesting future modification in the transesterification process. However, despite having only 53.13% FAME, the biodiesel produced falls within the physical-chemical parameters analyzed and recommended by international agencies. Another essential aspect observed is the high oxidative stability degradation showed by SSBD. In Rancimat, the SSBD presented results superior to those found in the literature data, with 40 hours for the beginning of oxidation; the biodiesel offers numerous storage advantages. With these previous results, it is possible to indicate that the vegetable oil of *Syagrus schizophylla* can be considered another alternative route in the production of biodiesel in Brazil, thus allowing an appreciation and economic development of the Northeast region of the country. Further studies are necessary to determine the ideal conditions used in the production of biodiesel

#### **5 Acknowledgements**

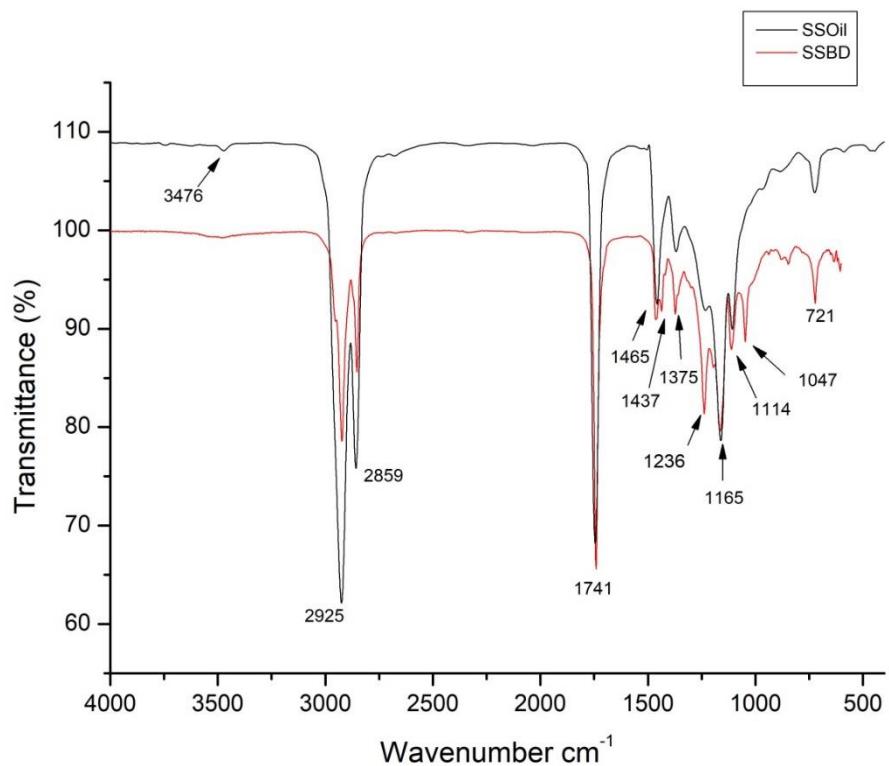
This study was funded by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) (*Coordination for the Advanced of Higher Education Personnel*), Fundação de Amparo a Ciência e Tecnologia do Estado de Pernambuco (FACEPE) (*Foundation for the Support of Science and Technology of the State of Pernambuco*), Coruripe Power Plant, and the Central Analysis of the Federal University of Pernambuco and Federal University of Paraíba.

<b>Compound SSOil</b>	<b>RT</b>	<b>%</b>
Octanoic acid (C:8)	4.55	10.15±0.06
Decanoic acid (C:10)	7.29	6.39±0.08
Dodecanoate acid (C:12)	7.84	<b>43.56±0.23</b>
Tetradecanoic acid (C:14)	14.53	<b>15.08±0.18</b>
Hexadecanoic acid (C:16)	19.43	7.35±0.05
Cis-9-Octadecenoic acid (C18:1)	23.61	3.68±0.02
cis-9,12-Octadecadienoic acid (C18:2)	23.72	<b>10.35±0.27</b>
Octadecenoic acid (C:18)	24.16	3.43±0.23
<b>Compound of SSBD</b>	<b>RT</b>	<b>%</b>
Dodecanoic acid,methyl ester (C:12)	5.17	59.94 ± 4.5
Tetradecanoic acid, methyl ester (C:14)	8.487	17.47 ± 0.05
Hexadecanoic acid, methyl ester (C:16)	12.08	7.36 ± 1.04
Octadecenoic acid, methyl ester (C:18)	15.223	3.36 ± 1.02
Cis-9,12-Octadecenoic acid, methyl ester (C18:2)	15.671	11.2 ± 3.18

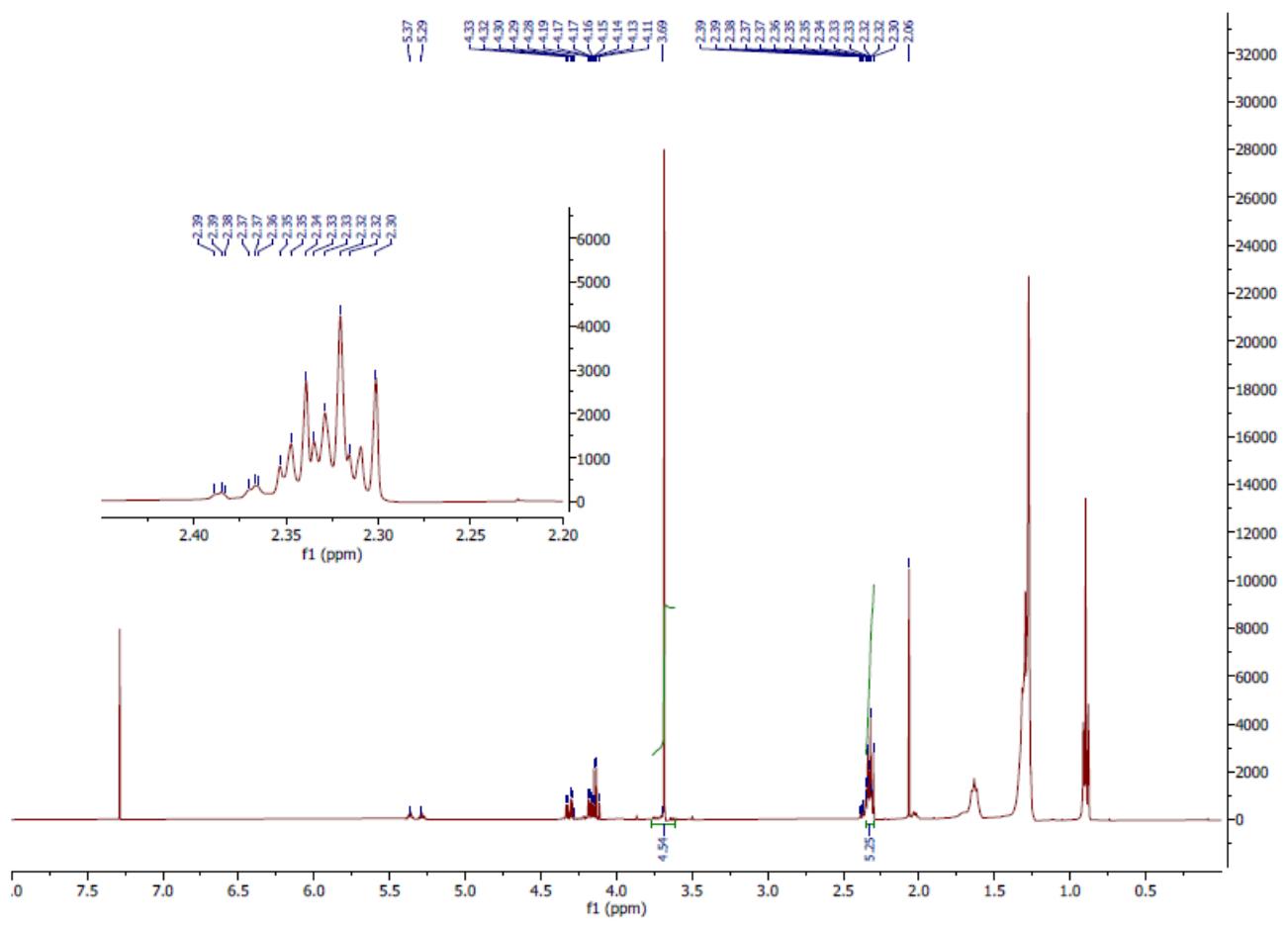
**Table 1:** GC-FID fatty acid profile of *S. schizophylla* oil (SSOil) and biodiesel (SSBD). RT= Retention time

<b>Properties</b>	<b>SSOil</b>	<b>SSBD</b>	<b>ASTM 6751-02</b>	<b>EN 14214</b>
Density at 15°C (kg/m <sup>3</sup> )	907	856	820-900	860-900
Kinematic Viscosity at 40°C (mm <sup>2</sup> /s)	26.251	2.736	1.9-3	3.5-6
Acid Value (mg KOH/g)	2.01	0.622	0,5	0,5
Saponification value (mg KOH/g)	*	194.32	*	*
Carbon residue (% w/w)	*	0.02	0.05	0.3
Cetane number	*	74	47 min	51 min
High heating value (MJ/Kg)	*	41.46	*	*
Iodine value (g Iodine/100g)	*	36.84	*	120
Oxidation stability, min (h, 110°C)	30	48	3	6

**Table 2:** Properties of *S. schizophylla* oil (SSOil) and biodiesel (SSBD). \* Not determined

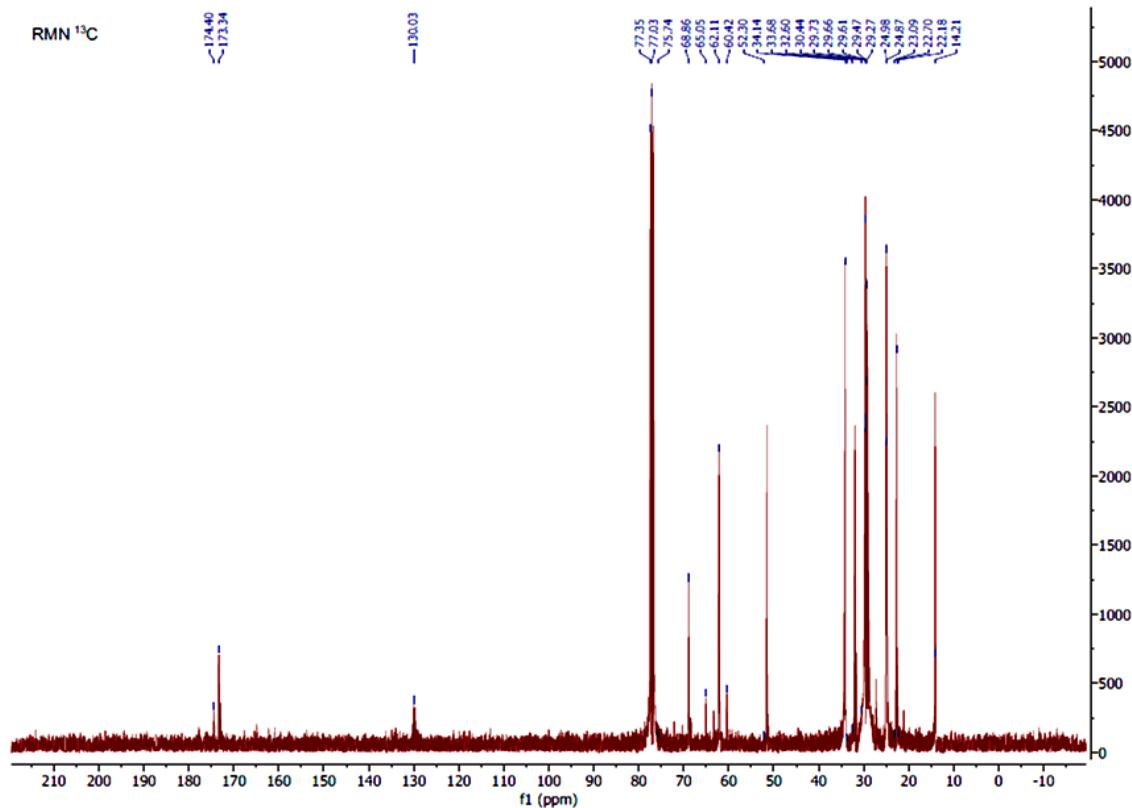


**Figure 1:** Infrared spectra of *S. schizophylla* oil (in black) and biodiesel (in red)

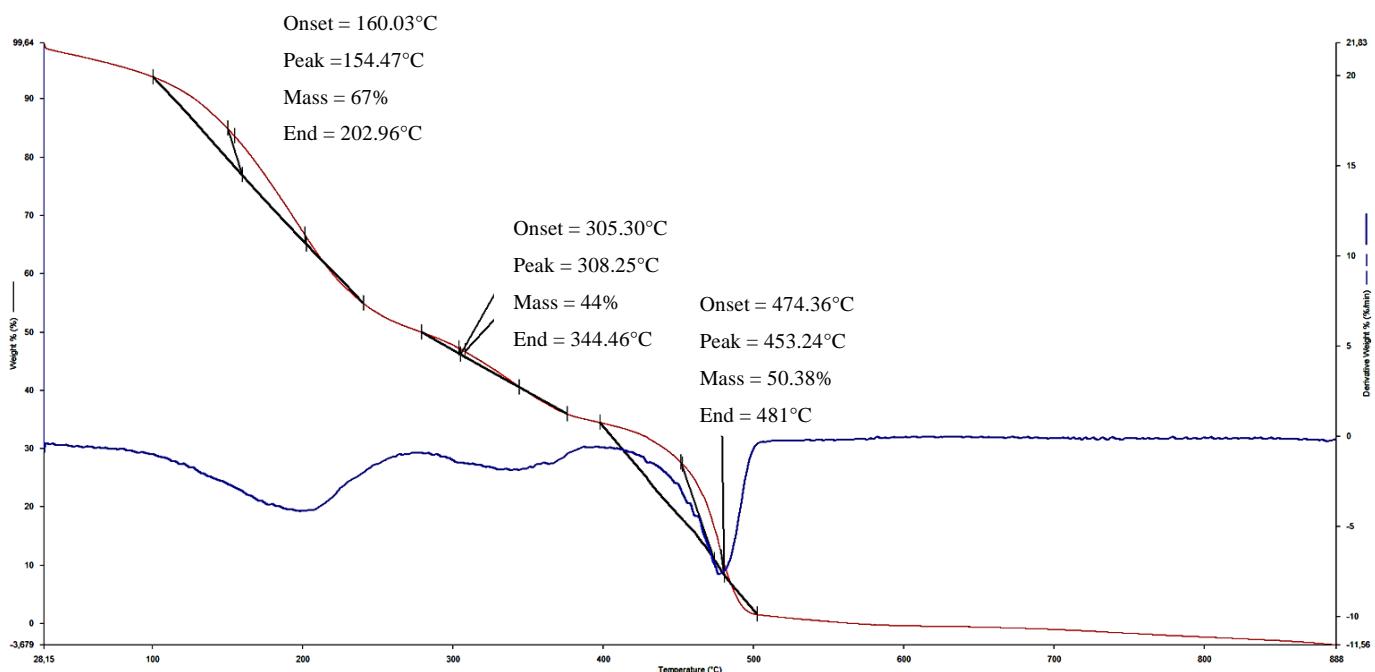


(a)

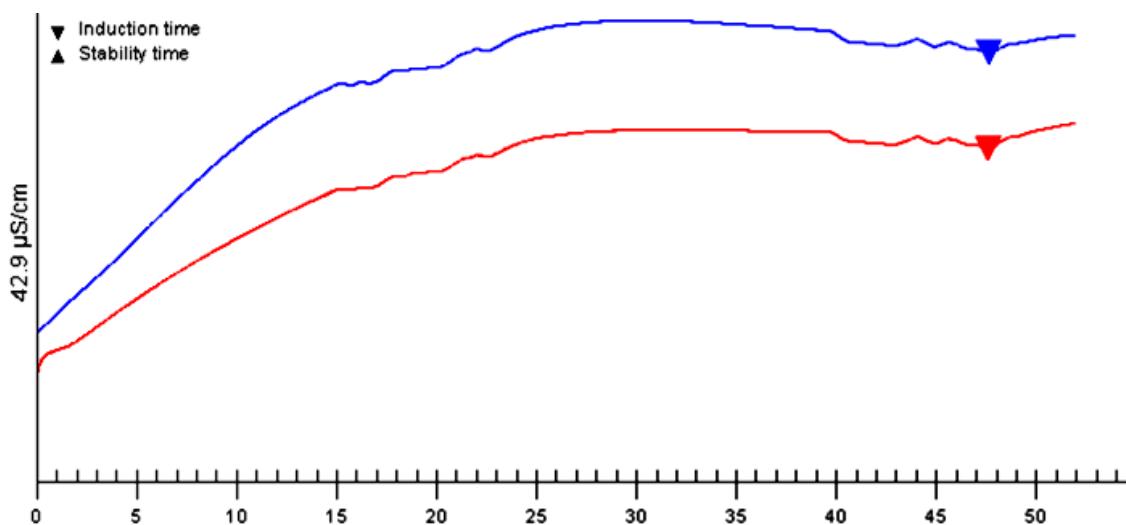
(b)



**Figure 2:** (a)  $^1\text{H}$  NMR and (b)  $^{13}\text{C}$  NMR spectra of SSBD



**Figure 3:** Thermogravimetric analysis (TGA/ DTG) for SSBD



**Figure 4:** Rancimat conductivity curves of SSBD

## 5. CONSIDERAÇÕES FINAIS

Os resultados obtidos neste trabalho demonstram a capacidade de aproveitamento e reutilização das diferentes frações dos recursos naturais proposta pelo novo modelo de produção econômica e impacto direto ambiental com reflexo na saúde populacional. O resíduo sólido após extração do óleo vegetal apresentou elevado teor de proteínas e minerais, sendo um recurso alimentar rico nutricionalmente podendo ser implementado na culinária local. Apresentando compostos bioativos na composição o resíduo sólido da Aricuriroba retoma os conceitos do consumo *in natura* dos elementos naturais podendo ser melhor aproveitados quanto ao seu potencial nutricional e funcional. O óleo vegetal como matéria prima principal das oleaginosas apresentam rotineiramente um forte apelo culinário, muito embora, neste estudo pôde ser demonstrado sua utilização para gerar maior impacto econômico no setor bioenergético. O óleo vegetal de Aricuriroba apresenta uma taxa de deteriorização menor que os óleos vegetais comumente aplicados, sua estrutura química ao apresentar baixas insaturações auxiliam na manutenção do óleo por um período de tempo prolongado, a presença de compostos antioxidantes auxiliam nos efeitos protetivos ao óleo. A concentração elevada de compostos saturados, facilita o uso e manipulação do óleo de Aricuriroba em formulações dermocosméticas, observado a sua biocompatibilidade com o extrato córneo da pele. Como conclusão do presente trabalho os frutos de *Syagrus schizophylla* demonstraram aplicabilidade nas três principais esferas da indústria de transformação: farmacêutica, energética e alimentícia. Espera-se que este modelo circular apresentando com os resultados possam incentivar e abranger novos direcionamentos quanto ao uso dos recursos naturais para fins biotecnológicos demais setores.

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## APÊNDICE A - PATENTE DE INVENÇÃO



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### Pedido nacional de Invenção, Modelo de Utilidade, Certificado de Adição de Invenção e entrada na fase nacional do PCT

Número do Processo: BR 10 2021 012760 0

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#### Dados do Depositante (71)

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##### Depositante 1 de 1

**Nome ou Razão Social:** UNIVERSIDADE FEDERAL DE PERNAMBUCO

**Tipo de Pessoa:** Pessoa Jurídica

**CPF/CNPJ:** 24134488000108

**Nacionalidade:** Brasileira

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#### Dados do Pedido

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**Natureza Patente:** 10 - Patente de Invenção (PI)

**Título da Invenção ou Modelo de Utilidade (54):** FOTOPROTETOR NA FORMA FAMACÊUTICA DE EMULSÃO  
ÓLEO EM ÁGUA CONTENDO ÓLEO FIXO VEGETAL DE *Syagrus schizophylla* (Mart.) Glasmann, COM PROPRIEDADE ANTIOXIDANTE, PARA MANUTENÇÃO DA ESTABILIDADE E/OU AUMENTO DO FATOR DE PROTEÇÃO SOLAR

**Resumo:** A presente invenção trata-se do desenvolvimento de formulações, com finalidade cosmética, contendo o óleo vegetal de Aricuriroba (*Syagrus schizophylla* Mart. Glasmann) como um ativo para fotoproteção e manutenção da estabilidade fotoprotetora, com capacidade antioxidante pelo sistema Beta-caroteno/ácido linoleico. As formulações apresentam fotoproteção na faixa ultravioleta com manutenção estabilidade após ciclo de gelo e desgelo por 12 dias consecutivos. O óleo de Aricuriroba como ativo biológico tem capacidade de aumentar o FPS em formulações com filtros químicos comerciais que tendem a perder seu poder fotoprotetor quando expostos a condições adversas, como variações de temperatura. A presente invenção se enquadra na área de cosméticos por ter aplicação dermatológica.

**Figura a publicar:** 2