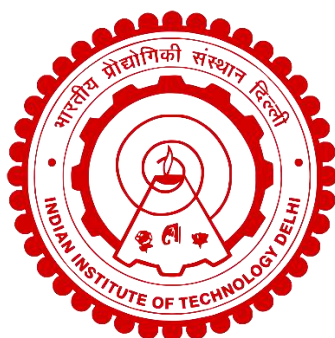


Strategies for Trans Fatty Acid Reduction and Rapid Detection in Fried/ Baked Foods

Tanya



Centre for Rural Development and Technology

Indian Institute of Technology Delhi

August 2024

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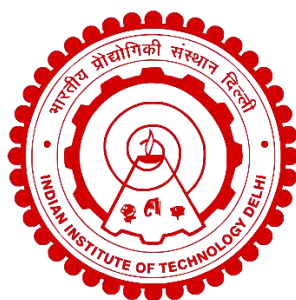
Tanya

Submitted

In fulfillment of the requirements of the degree of

Doctor of Philosophy

to the



Indian Institute of Technology Delhi

August 2024

CERTIFICATE

This is to certify that the thesis entitled, “**Strategies for Trans Fatty Acid Reduction and Rapid Detection in Fried/ Baked Foods**” being submitted by **Ms. Tanya** to the **Indian Institute of Technology Delhi** for the award of “**Doctor of Philosophy**” is a record of bonafide research work carried out by her. She has worked under our guidance and supervision and has fulfilled the requirements for the submission of the thesis. To the best of our knowledge, the results contained in the thesis have not been submitted, in part or full, to any other university or institute for the award of any degree or diploma.

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ACKNOWLEDGMENT

My PhD journey, despite its challenges, has been remarkably rewarding and enriching. I would like to express my appreciation and gratitude to all those who supported me throughout these years.

Firstly, I express my gratitude to Prof Jatindra K Sahu, Prof Sunil Kumar Khare, and Prof Soumik Siddhanta, who have been my mentors during my PhD program. My research journey has seen thick and thin moments. They have always advised me to keep moving and do good research and entrusted me to work under their supervision. Their guidance, advice, and motivation were priceless resources.

I am incredibly thankful to my SRC members, Prof. Shashank Deep (SRC Chairperson), Prof. Tanmay Dutta (External Expert), and Prof. Hariprasad P (Internal Expert) for helping me with valuable suggestions during my PhD work.

I express my sincere gratitude and appreciation to the Erasmus+ International Credit Mobility ICM and Commonwealth Split-Site Fellowship for providing the scholarship for my visiting doctoral studies at Universidad de Santiago de Compostella (USC), Spain, and Sheffield Hallam University (SHU), United Kingdom respectively.

Special thanks to Prof. Massimo Lazzari, USC, Spain and Prof. Bipros Dubey, and Prof Martin Howarth, SHU, UK, who have been my host supervisors during my fellowship tenure at USC, Spain, and SHU, UK respectively.

I am grateful to my Lab Post-Docs, Dr. Shivani Chaturvedi, Dr. Nitya Sharma, Dr. Priyanka Prasad, Dr. Anita, Dr. Amrik Bhattacharya, and Dr. Tripti Ahuja for supporting and providing valuable insights into the experiments and discussions.

I take this opportunity to acknowledge my lab mates, Asutosh, Nikita, Sukirti, Satheeshkanth, Bareen, Pooja, Sushree, Preeti, Divyang, Prasanna, Dhruv, Achala, Vikas, Ruchi, Aashish, Aarti, Farzana, Arunima and Ruchi. I also express my sincere thanks to my friends from Sheffield Hallam University and Universidad de Santiago de Compostela, Abraham, Agathe, Tasmiyah, Emmanuel, Mariam, Judith, Yolanda, and Hari. Your unwavering dedication and willingness to go above and beyond have made a world of difference to me, and I cannot thank you enough.

I owe many thanks to my friends Bhavna, Nidhi, and Deepali. Their friendships are invaluable to me.

I express heartfelt gratitude to my parents, brother and Pratik, my unwavering support system and motivation, who stood as my rock, fostering determination throughout my academic journey. This thesis is dedicated to them for always being there for me and supporting my choices even when filled with apprehension.

(Tanya)

ABSTRACT

Trans fats, also known as trans-unsaturated fatty acids (TFA), are a type of dietary fat that has gathered attention for their negative impact on human health. Trans fats are advantageous for the food industry due to their stability and extended shelf life. They play a crucial role in enhancing the texture and mouthfeel of many processed foods, providing crispness to baked goods and smooth consistency to products like margarine. However, high intake of trans fat increases the risk of cardiovascular diseases, diabetes, obesity, fetal loss, and breast and colon cancer. Due to these health concerns, there has been a global shift away from trans fats, with many countries implementing regulations to limit the presence of trans fats in food products. The requirement for the replacement of TFA in the food supply globally has challenged the food industry to find a novel substitute to trans fats without compromising the functionality and organoleptic properties of the product.

The thesis encompasses a detailed study on the detection and reduction of trans fat in baked/fried foods. The study focused on trans fats and explored diverse mitigation approaches, including thermostable antioxidants and oleogelation technology. Natural antioxidants were utilized to reduce trans fats during deep frying, while subsequent research concentrated on structuring oil through oleogelation technology for trans fat replacement in the bakery. The work also features the development of the rapid detection of trans fat using surface-enhanced Raman spectroscopy (SERS).

To address the challenges of trans-fat formation during frying, antioxidant compounds present in *Psidium guajava* (guava) leaves extract were employed with frying oils to reduce the formation of trans fat. Guava leaves contained 173.33 ± 1.95 mg GAE/g extract total phenolic content and 20.43 ± 0.25 mg RUE/g extract total flavonoid content. Some of the phytochemicals in the extract were identified and quantified using HPTLC. The leave extract (1 g) contained 0.039 mg Gallic acid, 0.196 mg Rutin, 0.021 mg naringenin, and 0.059 mg ferulic acid. The antioxidant compounds, present in guava extracts, showed affinity towards lipid oxidation products and inhibited the formation of trans fat. These results of natural antioxidants were compared with synthetic antioxidant - butylated hydroxytoluene (BHT).

A rapid method for detection of oil quality during frying and trans-fat quantification was developed using surface-enhanced Raman spectroscopy (SERS). In the study, plasmon-tuned

and biocompatible Ag@Au core-shell nanoparticle-based SERS substrates were used to obtain optimum enhancement despite matrix interference to detect the oil components efficiently. The potent combination of SERS and the Artificial Neural Network (ANN) method could determine the fatty acid profile and peroxide value with 87-99% accuracy. Moreover, the SERS-ANN method could quantify the low level of trans fats, i.e., < 2% with 97% accuracy.

An attempt was made to optimize oleogelation technology for structuring oil with candelilla wax using high-intensity ultrasound (HIUS) treatment. The optimization strategy included investigating the effect of HIUS at different stages of crystallization on structure and stability of oleogels. Further, the effect of HIUS on stability of oleogels fortified with retinyl palmitate was investigated. Sunflower oil was structured with varying amount of candelilla wax (2-6% w/w). The microstructure analysis indicated that HIUS reduced the size and increased the number of oleogel crystals. Oil binding capacity significantly increased under both HIUS conditions. Notably, there was no significant impact of HIUS on hardness, rheology, or melting enthalpy of oleogels, containing less than 4% wax. Differently, HIUS modified the properties of oleogels with 4% wax or more, with a more pronounced effect when HIUS was applied at mid of crystallization. HIUS had no adverse effect on oxidative stability or retinyl palmitate stability in oleogels. HIUS application at mid of crystallization and a wax concentration above 4% yielded oleogels suitable as a fat substitute and was equally effective in forming retinyl palmitate-fortified oleogel.

Going forward, to replace trans fats in bakery applications bigels were formed by mixing oleogel and hydrogel in different fractions and phases. Higher fractions of oleogel increased the mechanical strength (storage modulus, firmness) and decreased the liquid binding capacity of bigels. Among different bigel formulations, it was found that mixing both the oleogel and hydrogel in their molten states had the highest liquid binding capacity. Specifically, the bigel created by combining molten oleogel and hydrogel in a 75:25 ratio exhibited the maximum mechanical strength and was consequently employed as a substitute for fat in cakes at varying ratios. Remarkably, the replacement of commercial fat with this bigel did not result in any oil migration. The substitution of fat with bigel up to 50% had no significant impact on the texture of the cakes. However, replacing more than 50% of fat with bigel resulted in the formation of harder cakes.

Keywords: Antioxidant; Bigel; Crystallization; Hydrogel; Oleogels; Sunflower oil; Trans fat

सारांश

ट्रांस वसा, जिसे ट्रांस-असंतृप्त फैटी एसिड के रूप में भी जाना जाता है, एक प्रकार का आहार वसा है जिसने मानव स्वास्थ्य पर अपने नकारात्मक प्रभाव के लिए ध्यान आकर्षित किया है। ट्रांस वसा अपनी स्थिरता और विस्तारित शेल्फ जीवन के कारण खाद्य उद्योग के लिए फायदेमंद हैं, जिससे तेल और वसा में ऑक्सीकरण और बासीपन की संवेदनशीलता कम हो जाती है। वे प्रसंस्कृत खाद्य पदार्थों की बनावट और स्वाद बढ़ाने, पके हुए माल को कुरकुरापन प्रदान करने और मार्जरीन जैसे उत्पादों को चिकनी स्थिरता प्रदान करने में महत्वपूर्ण भूमिका निभाते हैं। ट्रांस फैट के अधिक सेवन से हृदय रोग, मधुमेह, मोटापा, भ्रूण हानि, स्तन और पेट के कैंसर का खतरा बढ़ जाता है। इन स्वास्थ्य संबंधी चिंताओं के कारण, वैश्विक स्तर पर ट्रांस वसा से दूरी बढ़ी है, कई देशों ने खाद्य उत्पादों में उनकी उपस्थिति को सीमित करने के लिए नियम लागू किए हैं। विश्व स्तर पर खाद्य आपूर्ति में टीएफए के प्रतिस्थापन की आवश्यकता ने खाद्य उद्योग को उत्पाद की कार्यक्षमता और ऑर्गेनोलेप्टिक गुणों से समझौता किए बिना ट्रांस वसा के लिए एक नया विकल्प खोजने की चुनौती दी है।

थीसिस में पके हुए/तले हुए खाद्य पदार्थों में ट्रांस वसा का पता लगाने और उसे कम करने पर एक विस्तृत अध्ययन शामिल है। अध्ययन ट्रांस वसा पर केंद्रित है और थर्मोस्टेबल एंटीऑक्सिडेंट और ऑलियोजेलेशन तकनीक सहित विभिन्न शमन दृष्टिकोणों की खोज करता है। गहरे तलने के दौरान ट्रांस वसा को कम करने के लिए प्राकृतिक एंटीऑक्सिडेंट का उपयोग किया गया था, जबकि बाद के शोध ने बेकरी में ट्रांस वसा प्रतिस्थापन के लिए ऑलियोजेलेशन तकनीक के माध्यम से तेल की संरचना पर ध्यान केंद्रित किया। कार्य में सतह-संवर्धित रमन स्पेक्ट्रोस्कोपी का उपयोग करके ट्रांस वसा का तेजी से पता लगाने का विकास भी शामिल है।

तलने के दौरान ट्रांस-फैट निर्माण की चुनौतियों का समाधान करना। अमरूद की पत्तियों के अर्क में मौजूद एंटीऑक्सीडेंट यौगिकों को ट्रांस वसा के गठन को कम करने के लिए तलने वाले तेल में उपयोग किया जाता था। अमरूद की पत्तियों का अर्क तलने के तापमान पर थर्मोस्टेबल था। सिडियम गुजावा की पत्तियों में 173.33 ± 1.95 मिलीग्राम जीई/जी अर्क कुल फेनोलिक सामग्री और 20.43 ± 0.25 मिलीग्राम आरयूई/जी अर्क कुल फ्लेवोनोइड सामग्री होती है। अर्क में कुछ फाइटोकेमिकल्स की पहचान और मात्रा एचपीटीएलसी द्वारा निर्धारित की गई थी। सिडियम गुजावा की पत्तियों का अर्क (1 ग्राम) में 0.039 मिलीग्राम गैलिक एसिड, 0.196 मिलीग्राम रुटिन, 0.021 मिलीग्राम नैरिंगेनिन, 0.059 मिलीग्राम फेरुलिक एसिड होता है। गर्म करने के कारण तेलों के खराब होने से लिपिड ऑक्सीकरण उत्पाद और ट्रांस वसा का निर्माण हुआ। अमरूद के अर्क में मौजूद एंटीऑक्सीडेंट यौगिकों ने लिपिड ऑक्सीकरण उत्पादों के प्रति आकर्षण दिखाया और एक्रिलामाइड के निर्माण को रोक दिया। प्राकृतिक एंटीऑक्सीडेंट के इन परिणामों की तुलना सिंथेटिक एंटीऑक्सीडेंट, बीएचटी (ब्यूटाइलेटेड हाइड्रोक्सीटोल्थूइन) से की गई।

इसके अलावा, तलने के दौरान तेल की गुणवत्ता की निगरानी और ट्रांस-फैट का पता लगाने के लिए सतह-संवर्धित रमन स्पेक्ट्रोस्कोपी का उपयोग करके तीव्र विधि विकसित की गई थी। अध्ययन में, तेल घटकों का कुशलतापूर्वक पता लगाने के लिए मैट्रिक्स हस्तक्षेप के बावजूद इष्टतम वृद्धि प्राप्त करने के लिए प्लास्मोन-ट्यून्ड और बायोकम्पैटिबल एजी@एयू कोर-शेल नैनोकण-आधारित एसईआरएस सबस्ट्रेट्स का

उपयोग किया गया था। एसईआरएस और आर्टिफिशियल न्यूरल नेटवर्क (एएनएन) विधि का शक्तिशाली संयोजन 87 - 99% सटीकता के साथ फैटी एसिड प्रोफाइल और पीवी निर्धारित कर सकता है। इसके अलावा, ^{SERS-ANN} विधि 97% सटीकता के साथ ट्रांस वसा के निम्न स्तर, यानी <2%, को माप सकती है। इसलिए, विकसित एल्गोरिदम-सहायता प्राप्त एसईआरएस प्रणाली ने तेल ऑक्सीकरण की सुचारू और तीव्र निगरानी और ऑन-साइट पता लगाने में सक्षम बनाया।

उच्च तीव्रता वाले अल्ट्रासाउंड उपचार का उपयोग करके कैंडेलिला मोम के साथ तेल की संरचना के लिए ओलियोजेलेशन तकनीक को अनुकूलित करने का प्रयास किया गया था। अनुकूलन रणनीति में ओलेजेल्स की संरचना और स्थिरता पर क्रिस्टलीकरण के विभिन्न चरणों में उच्च-तीव्रता वाले अल्ट्रासाउंड (एचआईयू) के प्रभाव की जांच करना शामिल है। इसके अलावा, रेटिनिल पामिटेट से मजबूत किए गए ओलेओजेल की स्थिरता पर एचआईयू के प्रभाव की जांच की गई। सूरजमुखी तेल को अलग-अलग मात्रा में कैंडेलिला मोम (2-6% w/w) से संरचित किया गया था। माइक्रोस्ट्रक्चर विश्लेषण से संकेत मिलता है कि ^{HIU} ने आकार कम कर दिया और ओलेगेल क्रिस्टल की संख्या में वृद्धि की। दोनों ^{HIU} स्थितियों के तहत तेल बाइंडिंग क्षमता में उल्लेखनीय वृद्धि हुई। विशेष रूप से, 4% से कम मोम वाले ओलेओजेल की कठोरता, रियोलॉजी, या पिघलने वाली एन्थैल्पी पर एचआईयू का कोई महत्वपूर्ण प्रभाव नहीं पड़ा। अलग ढंग से, ^{HIU} ने 4% या अधिक मोम के साथ ओलेओजेल के गुणों को संशोधित किया, जब ^{HIU} को क्रिस्टलीकरण के मध्य में लागू किया गया तो अधिक स्पष्ट प्रभाव पड़ा। ओलियोजेल में ऑक्सीडेटिव स्थिरता या रेटिनिल पामिटेट स्थिरता पर एचआईयू का कोई प्रतिकूल प्रभाव नहीं पड़ता है। क्रिस्टलीकरण के मध्य में ^{HIU} अनुप्रयोग और 4% से ऊपर मोम सांद्रता से वसा के विकल्प के रूप में उपयुक्त ओलेओजेल प्राप्त हुआ और रेटिनिल पामिटेट-फोर्टिफाइड ओलेओजेल बनाने में भी उतना ही प्रभावी था।

आगे बढ़ते हुए, बेकरी अनुप्रयोग में ट्रांस वसा को प्रतिस्थापित करने के लिए ओलेजेल और हाइड्रोजेल को अलग-अलग अंशों और चरणों में मिलाकर बिगेल्स का गठन किया गया, जिसमें दोनों घटकों की पिघली हुई और जेली अवस्थाएं शामिल थीं। ओलेगेल के उच्च अंशों ने यांत्रिक शक्ति (भंडारण मापांक, दृढ़ता) को बढ़ा दिया और बिगेल्स की तरल बंधन क्षमता को कम कर दिया। विभिन्न बिगेल फॉर्मूलेशन के बीच, यह पाया गया कि ओलेओजेल और हाइड्रोजेल दोनों को उनकी पिघली हुई अवस्था में मिलाने से तरल बंधन क्षमता सबसे अधिक थी। विशेष रूप से, 75:25 के अनुपात में पिघले हुए ओलेओगेल और हाइड्रोजेल को मिलाकर बनाए गए बिगेल ने अधिकतम यांत्रिक शक्ति (कठोरता, भंडारण मापांक) प्रदर्शित की और परिणामस्वरूप इसे अलग-अलग अनुपात में केक में वसा के विकल्प के रूप में नियोजित किया गया। उल्लेखनीय रूप से, इस बिगेल के साथ वाणिज्यिक वसा के प्रतिस्थापन के परिणामस्वरूप कोई तेल प्रवासन नहीं हुआ। 50% तक बिगेल के साथ वसा के प्रतिस्थापन का केक की बनावट पर कोई महत्वपूर्ण प्रभाव नहीं पड़ा। हालाँकि, 50% से अधिक वसा को बिगेल से बदलने के परिणामस्वरूप सख्त केक का निर्माण हुआ।

कीवर्ड: एंटीऑक्सीडेंट, बिगेल, क्रिस्टलीकरण, हाइड्रोजेल, ओलेजेल्स, सूरजमुखी तेल, हाइड्रोजेल, ट्रांस फैट

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LIST OF SYMBOLS AND ABBREVIATIONS

% : Percent

< : Less than

> : Greater than

$\geq$: Greater than or equal to

$^{\circ}\text{C}$: Degree Celsius

Eq. : Equation

g : gram

g.L^{-1} : gram per litre

h : hour

kg : kilogram

μm : micrometer

μL : micro Litre

mm: millimeter

min: minute

meq: mili equivalent

mL: mili Litre

mg: miligram

Pa: Pascal

R^2 : Co-efficient of determination

rad: radian

s: second

SD: Standard deviation

v: volume

w: weight