

**DEVELOPMENT OF VALORIZED PRODUCTS FROM  
FLORAL WASTE USING INTEGRATED APPROACH  
AND THEIR POTENTIAL APPLICATIONS**

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**INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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**by**

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Submitted

in fulfillment of the requirements of degree of Doctor of Philosophy

to the



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*Dedicated to*  
*“The Mother”*

## CERTIFICATE

This is to certify that the thesis entitled “**Development of Valorized Products from Floral Waste using Integrated Approach and their Potential Applications**”, being submitted by Mrs. Sushree Titikshya 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 this thesis. To the best of our knowledge, the results contained in this 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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## ABSTRACT

Wasted flowers from religious places, as well as cultural celebrations, end up in landfills and waterways, where they pose a threat to wildlife and human health. Therefore, alternative processes have been devised to turn these wastes into valuable resources and build a circular economy in close loop system for environmental management and construction of zero waste approach. This research focuses on recovering and reusing valuable components from two flower waste: marigold (*Tagetes erecta*) and rhododendron (*Rhododendron arboreum*). The primary goal of this study was to record several methods of drying food to increase its shelf life and various methods of extracting bioactive compounds for use in food. In addition to being used in a closed-loop system for waste water treatment, the wastes were also put to use in the manufacture of biochar by slow pyrolysis. The physicochemical properties of the floral wastes were evaluated before any applications were carried out. This included testing for moisture, ash, volatiles, CHNS, crude fibre, and mineral content. Both biomasses were found to have a volatile concentration of more than 85 wt.%, making them ideal candidates for biochar applications targeting the elimination of poisonous dyes and heavy metals. The phosphorus content in marigolds (30386.5  $\mu\text{g/g}$ ) and rhododendrons (24752.65  $\mu\text{g/g}$ ) were found to be much greater than those of other minerals. Dried flower scraps were analyzed for their ability to retain TPC (Total phenolic content), TFC (Total flavonoid content), and antioxidant characteristics. Infrared drying at 250W preserved the most phytochemical and antioxidant qualities out of three drying methods (infrared drying, microwave drying, and hot air drying). Microwave drying at high temperatures of 600W for shorter durations reduces phytochemical concentrations and antioxidant qualities in floral biomass, in contrast to prolonged drying times at lower temperatures. The following results were found for the TPC, TFC, DPPH, and FRAP of infrared-dried marigold flowers at 250 W: GAE/g = 57.51mg, QE/g = 68.96mg, IC<sub>50</sub> = 35.52mg/ml, and Fe<sup>2+</sup>/g = 165.51mg/g. Once infrared dried at 250 W, rhododendron showed highest values of 67.14 mg GAE/g, 240.24 mg QE/g, 25.95 IC<sub>50</sub> g/ml, and 159.53 mg Fe<sup>2+</sup>/g for TPC, TFC, DPPH, and FRAP.

The infrared dried floral petals were utilized for extraction of bioactives using microwave and ultrasound aid. Bioactive compounds and antioxidants are found in abundance in flower biomass.

Here are the conditions for microwave-assisted extraction that yielded the best results from spent marigold flowers: TPC: 119.51 mg/100g, TFC: 145.18 mg GAE/g, DPPH: 34.39 IC<sub>50</sub> g/ml, and FRAP: 569.15 mg Fe<sup>2+</sup>/g; microwave power: 310W; yield recovery: 74.22%; TCC: 119.51 mg/100g. The expected results for UAE under ideal conditions (amplitude: 30%; sonication time: 12 min; solvent volume: 150 ml) were 42.61 % for yield recovery, TPC: 139.83 mg GAE/g, TFC: 374.45 mg QE/g, DPPH: 45.51 IC<sub>50</sub> g/ml, FRAP: 474.97 mg Fe<sup>2+</sup>/g, and TCC: 100.21 mg/100g. Similarly, for rhododendron floral wastes extracted with ideal microwave aided conditions (microwave power: 300W; time: 7 min; solvent volume: 211.10 ml), the expected values for yield recovery, TPC, TFC, DPPH, FRAP, and TAC were as follows: Results: 86.45%, 208.84 mg GAE/g, 826.11 mg QE/g, 29.38 IC<sub>50</sub> g/ml, 586.94 mg Fe<sup>2+</sup>/g, and 230.73 mg/g. The expected values for the yield recovery, TPC, TFC, DPPH, FRAP, and TAC of UAE under ideal conditions (sonication time: 15 minutes; solvent volume: 150 ml; amplitude: 29.6%) are: 70.84%, 193.00 mg GAE/g, 523.86 mg QE/g, 35.17 IC<sub>50</sub> g/ml, 504.61 mg Fe<sup>2+</sup>/g, and 139.41 mg/100g. When processing rhododendron and marigold, MAE drastically shortened treatment time compared to UAE, cutting it from 5 minutes to 8 minutes while still maintaining bioactive component preservation and improving extraction efficiency.

An antioxidant-fortified product was developed by combining microwave-assisted marigold (ME) and rhododendron extract (RE) with three unique oils: sesame, peanut, and mustard. The peroxide value (PV), acid value (AV), and iodine value (IV) were measured to determine the efficacy of the flower extract in comparison to the synthetic antioxidant (BHT 200 ppm) when added to groundnut, sesame, and mustard oil at concentrations of 200, 400, 600, 800, and 1000 ppm. Primary and secondary oxidation products were both significantly reduced at the maximum ME dose compared to the control. Extracts with concentrations of 600 ppm or above suppress lipid oxidation more effectively than BHT at 200 ppm. The shelf life of all three oils was increased by about 50-60 days as a result of this improvement, and at some concentrations, the oils could be stored for over 100 days. In light of these results, it seems reasonable to consider ME and RE as potential organic replacements for synthetic antioxidants. In order to get a bird's-eye view of the sample variations and spot behavioural patterns, we used a modest number of principle components (PC) to decrease the initial variables. PC1 and PC2 components accounted for 96.84 % and 1.87 %, respectively, of the total variance in groundnut oil with ME. 69.53 and

32.89 percent of the variance in groundnut oil, 98.21 and 1.07 % in sesame oil, and 98.2 and 1.52 % of the variance in mustard oil could be accounted for by ME.

Slow pyrolysis at 400 °C, 500 °C, and 600 °C for 60 min was used to convert the depigmented biochar residue into a green bioadsorbent. After completion of the pyrolysis processes, the physicochemical analysis of the rhododendron residue derived biochar (RRB), marigold residue derived biochar (MRB) and marigold husk derived biochar (MHB) was investigated by proximate, ultimate and different instrumental analysis like XRD, SEM, FTIR and BET studies. Low volatile matter (from 13.54 to 20.5 %) and high fixed carbon (from 81.85 to 85.16 %) were observed in RRB, MRB, and MHB samples at a range of pyrolytic temperatures (from 400 to 600 °C), demonstrating compliance with the requirements for biochar production. Biochar obtained at 600 °C had higher quality for further uses, as determined by studying the biochar yields and physicochemical evaluations. In addition, it was shown that the specific surface area of RRB is the largest ( $412.00 \text{ m}^2 \text{ g}^{-1}$ ) compared to MRB ( $6.21 \text{ m}^2 \text{ g}^{-1}$ ) and MHB ( $17.09 \text{ m}^2 \text{ g}^{-1}$ ). All of the biochar samples were found to be significantly porous, with the creation of pores, cracks, and ridges on the structure, as determined by both the structural analysis and the morphological evaluation by SEM analysis. This study's findings lend credence to the idea that biochar, thanks to its high surface area (particularly RRB), porosity, and surface functional groups, can be used in wastewater treatment processes such, for example, the degradation of dyes and the accumulation of heavy metals. When compared to biochar made from marigolds, which was tested for its ability to remove Congo red (CR) and methylene blue (MB) dyes from aqueous solutions at a fixed initial concentration of 50 mg/L, rhododendron-derived biochar showed significantly higher removal percentages (91.60 % for CR and 88.30 % for MB). Modelling their adsorption behaviour and surface homogeneity with the Langmuir isotherm provided more confirmation. The primary idea is "waste to wealth and circular economy," which lays emphasis on closing the loop between the efficient avoidance, recycling, and utilization of floral waste for its potential use in the food business and the environmental remediation sector in India and around the world.

## सारांश

धार्मिक स्थलों और सांस्कृतिक उत्सवों से आने वाले बर्बाद फूल, जो कि भूमि में और जलमार्गों में जाकर वन्यजीव और मानव स्वास्थ्य के लिए खतरा पैदा करते हैं, इन सामग्रियों को मूल्यवान संसाधनों में परिणामी बनाने और पर्यावरण प्रबंधन और शून्य कचरा दृष्टिकोण के निर्माण के लिए एक सीधे लूप प्रणाली में बुनने के लिए वैकल्पिक प्रक्रियाएं बना दी गई हैं। इस अनुसंधान का मुख्य उद्देश्य दो फूल अपशिष्ट: मेरीगोल्ड (*Tagetes erecta*) और रोडोडेंड्रन (*Rhododendron arboreum*) से मूल्यवान घटकों को पुनः प्राप्त और पुनः उपयोग करना है। इस अध्ययन का प्रमुख लक्ष्य था कि खाद्य की आवधि बढ़ाने के लिए उसे सुखाने के कई तरीके और खाद्य में उपयोग के लिए जैव-सक्रिय यौगिकों को निकालने के कई तरीके दर्ज किए जाएं। इन अपशिष्टों का उपयोग केवल एक सीधे लूप प्रणाली के लिए केवल एक छाया तथा अलगत्रात्मक प्रक्रिया में किया गया था। किसी भी आवेदन को आगे बढ़ाने से पहले फूलीय अपशिष्टों की भौतिक-रासायनिक गुणधर्मों का मूल्यांकन किया गया। इसमें नमी, खाद्य, वायलेटाइल्स, सीएचएनएस, कूड फाइबर, और खनिज सामग्री की जाँच शामिल थी। दोनों बायोमासेस की वॉलेटाइल एकाग्रता 85 wt.% से अधिक पाई गई, जिससे इन्हें विषाक्त रंगों और भारी धातुओं की निरस्तीकरण को लक्षित करने के लिए बायोचार के उपयोग के लिए आदर्श उम्मीदार बनाता है। मेरीगोल्ड्स (30386.5 µg/g) और रोडोडेंड्रन्स (24752.65 µg/g) में फास्फोरस की धाराएं अन्य खनिजों की धाराओं से कहीं अधिक पाई गईं। सुखे हुए फूलों के टुकड़ों का अध्ययन TPC, TFC, और एंटीऑक्सीडेंट विशेषताओं को बनाए रखने की क्षमता के लिए किया गया। 250W पर इन्फ्रारेड सुखाने से तीन सुखाने के तरीकों (इन्फ्रारेड सुखाना, माइक्रोवेव सुखाना, और हॉट एयर सुखाना) में सबसे अधिक फाइटोकैमिकल और एंटीऑक्सीडेंट गुणरस बने रहते हैं। 600W के उच्च तापमानों पर माइक्रोवेव सुखाने से फूल बायोमास में फाइटोकैमिकल कंसंट्रेशन और एंटीऑक्सीडेंट गुणरस कम हो जाते हैं, विपरीत तापमानों पर लंबे समय तक सुखाने के कारण। इन्फ्रारेड-सुखे हुए मेरीगोल्ड फूलों के लिए 250 W पर निम्नलिखित परिणाम मिले: TPC= 57.51 mg GAE/g, TFC = 68.96 mg QE/g, DPPH= 35.52 IC<sub>50</sub> µg /ml, और FRAP= 165.51mg Fe<sup>2+</sup>/g। एक बार 250 W पर इन्फ्रारेड सुखाए जाने पर, रोडोडेंड्रन ने TPC, TFC, DPPH, और FRAP के लिए 67.14 mg GAE/g, 240.24 mg QE/g, 25.95 IC<sub>50</sub> µg /ml, और 159.53 mg Fe<sup>2+</sup>/g के लिए सर्वोच्च मूल्य प्रदर्शित किए।

इन्फ्रारेड सुखे हुए फूलों के पेटलों का उपयोग माइक्रोवेव और अल्ट्रासाउंड सहारे से बायोएक्टिव्स की निकाल के लिए किया गया। फूल बायोमास में बायोएक्टिव यौगिक और एंटीऑक्सीडेंट्स बहुमूल्य मात्रा में

पाए जाते हैं। यहाँ वे शर्तें हैं जिनसे खर्च मेरीगोल्ड फूलों से सबसे अच्छे परिणाम प्राप्त हुए थे: TPC: 119.51 मिलीग्राम/100 ग्राम, TFC: 145.18 mg GAE/g, DPPH: 34.39 IC<sub>50</sub> µg /ml, और FRAP: 569.15 mg Fe<sup>2+</sup>/g; माइक्रोवेव शक्ति: 310W; यील्ड रिकवरी: 74.22%; TCC: 119.51 mg/100g। आदर्श स्थितियों के तहत यूई के लिए अपेक्षित परिणाम (एम्प्लीट्यूड: 30%; सोनिकेशन समय: 12 min; रोगी आंतर: 150 ml) 42.61% यील्ड रिकवरी, TPC: 139.83 mg GAE/g, TFC: 374.45 mg QE/g, DPPH: 45.51 IC<sub>50</sub> µg /ml, FRAP: 474.97 mg Fe<sup>2+</sup>/g, और TCC: 100.21 mg/100 g थे। उसी रूप में, रोडोडेंड्रन फूल अपशिष्टों को आदर्श माइक्रोवेव सहारित स्थितियों के साथ निकाला गया (माइक्रोवेव शक्ति: 300W; समय: 7 min; रोगी आंतर: 211.10 ml), उदाहरण रूप से यील्ड रिकवरी, TPC, TFC, DPPH, FRAP, और TAC के लिए अपेक्षित मूल्य थे: परिणाम: 86.45%, 208.84 mg GAE/g, 826.11 mg QE/g, 29.38 IC<sub>50</sub> µg /ml, 586.94 mg Fe<sup>2+</sup>/g, और 230.73 mg/g। आदर्श स्थितियों के तहत यूई के लिए अपेक्षित मूल्य (सोनिकेशन समय: 15 min; रोगी आंतर: 150 ml; एम्प्लीट्यूड: 29.6%) की यील्ड रिकवरी, TPC, TFC, DPPH, FRAP, और TAC के लिए अपेक्षित मूल्य हैं: 70.84%, 193.00 mg GAE/g, 523.86 मिलीग्राम QE/g, 35.17 IC<sub>50</sub> µg /ml, 504.61 mg Fe<sup>2+</sup>/g, और 139.41 mg/100 g। रोडोडेंड्रन और मेरीगोल्ड को प्रसंस्कृत करते समय, एमई ने यूई की तुलना में उपचार का समय ड्रैस्टिकली कम कर दिया, जिससे यह 5 min से 8 min कम हो गया, फिर भी बायोएक्टिव कॉम्पोनेंट की संरक्षण और निष्कर्षण क्षमता में सुधार की गई।

एक एंटीऑक्सीडेंट-सशक्तकृत उत्पाद को बनाने के लिए माइक्रोवेव-सहारित मेरीगोल्ड (ME) और रोडोडेंड्रन एक्सट्रैक्ट (RE) को तीन अद्वितीय तेलों - तिल, मूँगफली, और सरसों के साथ मिलाया गया। पेरोक्साइड वैल्यू (PV), एसिड वैल्यू (AV), और आयोडीन वैल्यू (IV) को मापन किया गया था ताकि फूल एक्सट्रैक्ट की प्रभावकारिता का तुलनात्मक मूल्यांकन किया जा सके जब यह 200, 400, 600, 800, और 1000 ppm की अधिकतम वितरण में मूँगफली, तिल, और सरसों के तेल में जोड़ा गया। प्राथमिक और सेकेंडरी ऑक्सीडेशन उत्पाद दोनों ही नियंत्रित तुलना में अधिकतम एमई खासी मात्रा में थे। 600 ppm या इससे अधिक की अधिकतम मात्रा वाले एक्सट्रैक्ट्स ने 200 ppm के BHT की तुलना में वसा ऑक्सीडेशन को अधिक प्रभावी रूप से दबाया। इस सुधार के परिणामस्वरूप, इस वृद्धि के कारण तीन तेलों की शेल्फ लाइफ लगभग 50-60 दिनों तक बढ़ गई, और कुछ मात्राओं में तेलों को 100 दिन से अधिक के लिए स्टोर किया जा सकता था। इन परिणामों के प्रकार, यह संभावनात्मक है कि एमई और आरई को सिंथेटिक एंटीऑक्सीडेंट्स के लिए संभावित जैविक परिवर्तन के रूप में लिया जा सकता है। नमूना विविधताओं और

व्यावहारिक पैटर्न को स्थिति करने के लिए हमने प्रारंभिक चरणों को कम करने के लिए माध्यम संख्या (पीसी) का उपयोग किया। पीसी<sub>1</sub> और पीसी<sub>2</sub> घटकों ने मिलाकर कुल वेरिंसेस का 96.84% और 1.87% हिस्सा किया, क्रमशः, मूँगफली तेल में एमई के साथ। 69.53 और 32.89 प्रतिशत वेरिंसेस मूँगफली तेल में, 98.21 और 1.07% तिल तेल में, और 98.2 और 1.52% वेरिंसेस सरसों तेल में एमई द्वारा विवरित किए जा सकते थे।

60 मिनट के लिए 400 °C, 500 °V, और 600 °C पर धीमी पायरोलिसिस का उपयोग विकसित हरित बायोअब्जॉर्बेंट में डीपिगमेंटेड बायोचार अवशेष को बदलने के लिए किया गया। पायरोलिसिस प्रक्रियाओं के पूर्ण होने के बाद, रोडोडेंड्रन अवशेष से प्राप्त बायोचार (RRB), मेरीगोल्ड अवशेष से प्राप्त बायोचार (MRB), और मेरीगोल्ड हस्क से प्राप्त बायोचार (MHB) की भौतिक-रासायनिक विश्लेषण की गई, जिसमें प्रॉक्सीमेट, अल्टीमेट, और एक्सआरडी, एसईएम, एफटीआईआर, और बीईटी अध्ययन जैसे विभिन्न उपकरणों का अध्ययन शामिल था। न्यून वॉलेटाइल मैटर (13.54 से 20.5 % तक) और उच्च स्थिर कार्बन (81.85 से 85.16 % तक) का अध्ययन प्यारोलिटिक तापमान की श्रृंगार में (400 से 600 °सेल्सियस) RRB, MRB, और MHB सैंपल्स में देखा गया, जो बायोचार उत्पाद के लिए आवश्यकताओं के साथ आत्मसमर्पण को दर्शाता है। 600 °सेल्सियस पर प्राप्त बायोचार ने उच्च गुणवत्ता की दिखाई और बायोचार यील्ड और भौतिक-रासायनिक मूल्यांकन का अध्ययन करके इसे आगे के उपयोगों के लिए बेहतर माना गया। इसके अलावा, यह दिखाया गया कि RRB का विशिष्ट सतह क्षेत्र सबसे बड़ा है (412.00 m<sup>2</sup> g<sup>-1</sup>), MRB (6.21 m<sup>2</sup> g<sup>-1</sup>) और MHB (17.09 m<sup>2</sup> g<sup>-1</sup>) की तुलना में। सभी बायोचार सैंपल्स को साइकॉडक्टिव साबित किया गया कि स्थानिक विश्लेषण और एसईएम विश्लेषण द्वारा संरचना पर गड्ढों, दरारों, और रिज की रचना के साथ सहारा हैं। इस अध्ययन के अनुसार प्राप्त नतीजे यह सिद्ध करते हैं कि बायोचार, उच्च सतह क्षेत्र (विशेषकर RRB), पोरसिटी, और सतह कार्यात्मक समूहों के कारण, वास्तविक प्रक्रियाओं में उपयोग किया जा सकता है, जैसे कि रंगों का विघटन और भारी धातुओं का संचुरण। मेरीगोल्ड से बनाए गए बायोचार की तुलना में, जिसे उसकी क्षमता का परीक्षण किया गया था कि यह स्थापित कर सकता है कि काँगो रेड (सीआर) और मेथिलीन ब्ल्यू (एमबी) रंगों को जलीय समाधानों से हटाने की क्षमता के लिए, रोडोडेंड्रन से प्राप्त बायोचार ने साइनिफिकेंटली अधिक हटाने के प्रतिशत दिखाए (सीआर के लिए 91.60% और एमबी के लिए 88.30%)। उनके अवशोषण व्यवहार और लैंगम्यूयर आइसोथर्म के साथ सतह समृद्धि का मॉडलिंग ने अधिक पुष्टि प्रदान की। मुख्य विचार "कचरे को संपत्ति और चक्रीय अर्थव्यवस्था" है, जिसमें फूल के

अपशिष्ट का कुशल टालना, पुनः प्रयोग, और उसे खाद्य व्यापार और भारत और दुनिया भर में जैविक प्रदूषण सेतु में उपयोग के लिए बंद करने पर जोर दिया जाता है।

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## LIST OF SYMBOLS

CV: co-efficient of variance

m/z: mass to charge ratio

MHz: mega hertz

N: normal

p-value: probability value

R<sup>2</sup>: co-efficient of determination or regression

meq: milli-equivalent

ppm: Parts per million

R<sub>RMS</sub>: root-mean-square roughness

T: Temperature

v/v: volume by volume

w/v: weight by volume

wt.%: Weight percentage

## LIST OF ABBREVIATION

ANOVA: Analysis of variance	GM: Groundnut oil in marigold extract
ASTM: American Society for Testing and Materials	GR: Groundnut oil in rhododendron extract
AV: Acid value	HAD: Hot air drying
BBD: Box Behnken design	HAE: Heat assisted extraction
BET: Brunauer Emmett Teller	HPLC: High-Performance Liquid Chromatography
BHA: Butylated hydroxyanisole	IC <sub>50</sub> : Half-maximal inhibitory concentration
BHT: Butylated hydroxy toluene	ICP-MS: Inductively coupled plasma-mass spectrometer
CHNS: Carbon, Nitrogen, Oxygen, Sulphur	IV: Iodine value
CR: Congo red	LCA: Life cycle assessment
DPPH: 2,2-Diphenyl-1-picrylhydrazyl	M: Mustard oil
DTG: Differential thermogravimetric analysis	MAE: microwave assisted extraction
EAE: Enzyme assisted extraction	MB: Methylene blue
FE: Floral extract	ME: marigold extract
FFA: Free fatty acids	MEAATPE: Microwave-assisted aqueous two-phase extraction
FRAP: Ferric Reducing Antioxidant Power	MHB: Marigold husk biochar
FTIR: Fourier-Transform Infrared Spectroscopy	MLS: Method of least square
FW: Floral waste	MRB: Marigold residue biochar
G: Groundnut oil	MSW: Municipal Solid Waste
GAE: Gallic acid equivalent	MM: Mustard oil with marigold extract
GC-MS: Gas chromatography- Mass spectrophotometry	MR: Mustard oil with rhododendron extract

NIR: Near Infrared

PCA: Principal component analysis

PEF: Pulsed-electric field

PLE: Pressurized liquid extraction

PV: Peroxide value

QE: Quercetin equivalent

RE: Rhododendron extract

RRB: Rhododendron residue biochar

S: Sesame oil

SbCW: Subcritical water

SCCO<sub>2</sub>: Supercritical CO<sub>2</sub>

SEM: Scanning electron microscope

SM: Sesame oil with marigold extract

SR: Sesame oil with rhododendron extract

TAC: Total anthocyanin content

TBHQ: Tert-butyl hydroquinone

TCC: Total carotenoid content

TFC: Total flavonoid content

TGA: Thermogravimetric analysis

TPC: Total phenolic content

TS: Total solid

UAE: Ultrasound assisted extraction

XRD: X-ray diffraction