

**DEVELOPMENT OF AN ADVANCED TREATMENT SYSTEM FOR
THE EFFECTIVE REMOVAL OF EMERGING CONTAMINANTS
FROM WASTEWATER**

DEEPCHANDRA JOSHI



**DEPARTMENT OF BIOCHEMICAL ENGINEERING AND BIOTECHNOLOGY
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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by

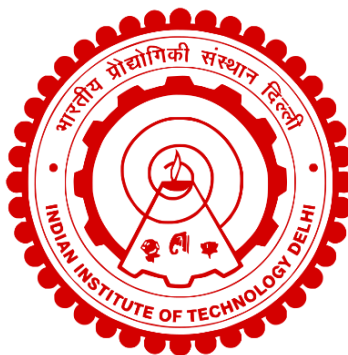
DEEPCHANDRA JOSHI

DEPARTMENT OF BIOCHEMICAL ENGINEERING AND BIOTECHNOLOGY

Submitted

in fulfilment of the requirement of the degree of Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

August 2024

Dedicated to my Nation

राष्ट्र देवो भवः

CERTIFICATE

This is to certify that the thesis titled '**Development of an advanced treatment system for the effective removal of emerging contaminants from wastewater,**' being submitted by **Mr. Deepchandra Joshi**, to the Indian Institute of Technology Delhi, for the award of the degree of **Doctor of Philosophy**, is a record of bonafide research work carried out by him. He has worked under my guidance and supervision and fulfilled the requirements for thesis submission, which has attained the standard required for a Ph.D. degree at this institute. The results contained in this thesis have not been submitted, in part or in full, to any other university or institute for the award of any degree or diploma.

Prof. T. R. Sreekrishnan

Department of Biochemical Engineering and Biotechnology

Indian Institute of Technology Delhi

Hauz Khas, New Delhi- 110016 INDIA

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ABSTRACT

Emerging contaminants (ECs) are the latest class of environmental contaminants which are highly recalcitrant, and toxic and currently lack specific regulatory guidelines. In the absence of suitable treatment methods against ECs, their concentration is increasing and has reached a level impacting human health and ecology. Therefore, to mitigate the effect of ECs, suitable treatment methods are required, and recently, plasma technology has shown potential for ECs degradation in a sustainable manner. Therefore, this work was conducted to study the suitability of Dielectric Barrier Discharge (DBD) plasma against ECs treatment from real wastewater (WW) using three different plasma reactors. Initially, the DBD plasma feasibility study was performed in a single jet plasma system, and degradation of dyes Rhodamine B (Rd. B) and Methylene Blue (Met. Blue) was performed. The DBD plasma jet provided a complete degradation and a 70% mineralization of both dyes in 30 min. The plasma jet efficiency was then evaluated for pharmaceuticals degradation in real WW, and 90% degradation efficiency was obtained, showing plasma's effectiveness in real environmental conditions. Then, to evaluate the DBD plasma jet's suitability with the conventional WW treatment system, a hybrid system consisting of the sequencing batch reactor (SBR) and DBD plasma jet was developed. The hybrid system with complete removal of the pharmaceuticals also provided a 50% mineralization, showing the plasma system's feasibility for real environmental applications. Continuing this, an attempt was made to scale up the plasma jet system, and a multiarray plasma jet system was developed, but the inter-jet interactions and high feed gas flow rate (>30 ml/min) requirement affected the uniform plasma formation. Therefore, improving the plasma systems capacity in the form of MultiJet was considered unsuitable, especially for its application in WW treatment. The plasma jet study showed that the limited plasma area is a major limitation for their utilization for practical purposes, and to overcome this issue, a novel S-shaped neon DBD plasma reactor

was designed in which curved electrodes were used in a linear configuration, providing a flat filamented plasma spreading entirely over the electrode length and the liquid surface. A low-cost commercial neon power supply (12 kV and 30 mA) was used, and the plasma generation capacity was compared with air, argon, and oxygen as the feed gases. The larger plasma area provided by the neon plasma system enhanced the plasma-liquid interface, thereby improving the diffusion of reactive species into the bulk liquid. This increased diffusion of reactive species was anticipated to increase the degradation of contaminants present in the bulk liquid. However, the removal of surface-active compounds exceeded that of the bulk contaminants due to the preferential interactions between the surface-active compounds and the reactive species at the plasma-liquid interface. To better understand the role of surface activity on contaminants degradation, a comparative study for ECs degradation in neon plasma and bubble column plasma reactor (BCPR) was performed. BCPR utilizes the gas bubbles for plasma generation, and the continuous sparging of the gas ensures a high homogenization in the system. The contaminant's degradation in BCPR provided a linear correlation between the contaminant's degradation rate (k_{obs}) and their surface activity (Log K_{ow}) with a preferential degradation for more surface-active contaminants. The BCPR was more efficient for contaminants degradation, but when it was operated in real environment conditions using samples with high initial conductivity and the presence of additional contaminants (surfactants and organic pollutants), its degradation efficiency reduced, which showed the limitation of BCPR for real WW treatment. No such issue was present with neon plasma, and it was also compatible with the conventional wastewater treatment system. Therefore, the neon plasma system was found to be more suitable for practical application. Finally, to further validate the neon plasma reactor as an effective advanced WW treatment system, a risk assessment of plasma-treated samples and an economic assessment of the plasma system study were performed, and its results also favored neon plasma.

सारांश

उभरते हुए प्रदूषक (ECs) पर्यावरणीय प्रदूषकों की एक नवीनतम श्रेणी हैं जो अत्यधिक अवक्षरण-प्रतिरोधी और विषैले होते हैं, और वर्तमान में उनके खिलाफ कोई विशिष्ट नियामक दिशा-निर्देश नहीं हैं। उपयुक्त उपचार विधियों की अनुपस्थिति में, ECs की सांद्रता बढ़ रही है और यह मानव स्वास्थ्य और पारिस्थितिकी पर प्रभाव डाल रही है। इसलिए, ECs के प्रभाव को कम करने के लिए उपयुक्त उपचार विधियों की आवश्यकता है, और हाल ही में, प्लाज़्मा तकनीक ने ECs के अपघटन के लिए संभावित समाधान दिखाया है। इसलिए, इस कार्य का उद्देश्य वास्तविक अपशिष्ट जल (WW) से ECs के उपचार के लिए तीन अलग-अलग प्लाज़्मा रिएक्टरों का उपयोग करके डार्डलेक्ट्रिक बैरियर डिस्चार्ज (DBD) प्लाज़्मा की उपयुक्तता का अध्ययन करना था। प्रारंभ में, DBD प्लाज़्मा का व्यवहार्यता अध्ययन एकल जेट प्लाज़्मा प्रणाली में किया गया, और रोडामाइन बी (Rd. B) और मेटिलीन ब्लू (Met. Blue) रंगों का अपघटन किया गया। DBD प्लाज़्मा जेट ने 30 मिनट में दोनों रंगों का पूरा अपघटन और 70% खनिजकरण प्रदान किया। फिर, वास्तविक WW में फार्मास्यूटिकल्स के अपघटन के लिए प्लाज़्मा जेट की दक्षता का मूल्यांकन किया गया, और 90% अपघटन दक्षता प्राप्त की गई, जो वास्तविक पर्यावरणीय परिस्थितियों में प्लाज़्मा की प्रभावशीलता को दर्शाता है। फिर, DBD प्लाज़्मा जेट की उपयुक्तता का मूल्यांकन करने के लिए, एक हाइब्रिड प्रणाली जिसमें अनुक्रमिक बैच रिएक्टर (SBR) और DBD प्लाज़्मा जेट शामिल थे, विकसित की गई। हाइब्रिड प्रणाली ने फार्मास्यूटिकल्स को पूरी तरह से हटाते हुए 50% खनिजकरण भी प्रदान किया, जो वास्तविक पर्यावरणीय अनुप्रयोगों के लिए प्लाज़्मा प्रणाली की व्यवहार्यता को दर्शाता है। इसके साथ ही, प्लाज़्मा जेट प्रणाली को स्केल-अप करने का प्रयास किया गया, और एक मल्टीएरे प्लाज़्मा जेट प्रणाली विकसित की गई, लेकिन इंटर-जेट इंटरैक्शन और उच्च फीड गैस प्रवाह दर (>30 मि.ली./मिनट) की आवश्यकता ने समान प्लाज़्मा निर्माण को प्रभावित किया। इसलिए, मल्टीजेट के रूप में प्लाज़्मा सिस्टम की क्षमता में सुधार करना, विशेष रूप से WW उपचार में इसके अनुप्रयोग के लिए अनुपयुक्त माना गया। प्लाज़्मा जेट अध्ययन ने दिखाया कि सीमित प्लाज़्मा क्षेत्र उनके व्यावहारिक उद्देश्यों के लिए उपयोगिता में एक प्रमुख सीमा है, और इस समस्या को दूर करने के लिए, एक नवीन S-आकार की नीयन DBD प्लाज़्मा रिएक्टर डिजाइन की गई जिसमें

वक्रित इलेक्ट्रोड का उपयोग किया गया, जो एक रेखीय कॉन्फ़िगरेशन में पूरे इलेक्ट्रोड की लंबाई और तरल सतह पर एक समान प्लाज़्मा फैलाव प्रदान करता है। एक कम लागत वाली वाणिज्यिक नीयन बिजली आपूर्ति (12 kV और 30 mA) का उपयोग किया गया, और प्लाज़्मा उत्पादन क्षमता की तुलना हवा, आर्गन, और ऑक्सीजन के साथ फीड गैस के रूप में की गई। नीयन प्लाज़्मा प्रणाली द्वारा प्रदान किया गया बड़ा प्लाज़्मा क्षेत्र प्लाज़्मा-तरल इंटरफ़ेस को बढ़ाता है, जिससे प्रतिक्रिया प्रजातियों का थोक तरल में प्रवाह सुधारता है। इस प्रकार, प्रतिक्रियाशील प्रजातियों के बढ़े हुए प्रवाह से थोक तरल में उपस्थित प्रदूषकों के अपघटन में वृद्धि होने की संभावना थी। हालांकि, प्लाज़्मा-तरल इंटरफ़ेस पर सतही सक्रिय यौगिकों और प्रतिक्रियाशील प्रजातियों के बीच वरीयता इंटरैक्शन के कारण सतही सक्रिय यौगिकों का अपघटन थोक प्रदूषकों से अधिक हो गया। सतही गतिविधि के प्रभाव को बेहतर ढंग से समझने के लिए, नीयन प्लाज़्मा और बबल कॉलम प्लाज़्मा रिएक्टर (BCPR) में ECs के अपघटन के लिए एक तुलनात्मक अध्ययन किया गया। BCPR गैस बुलबुलों का उपयोग प्लाज़्मा उत्पादन के लिए करता है, और गैस की निरंतर स्पार्जिंग प्रणाली में उच्च समरूपता सुनिश्चित करती है। BCPR में प्रदूषकों के अपघटन ने प्रदूषकों के अपघटन दर (kobs) और उनकी सतही गतिविधि (Log K_{ow}) के बीच एक रेखीय संबंध प्रदान किया, जिसमें अधिक सतही सक्रिय प्रदूषकों के लिए वरीयता अपघटन देखा गया। BCPR प्रदूषकों के अपघटन के लिए अधिक कुशल था, लेकिन जब इसे उच्च प्रारंभिक विद्युत चालकता और अतिरिक्त प्रदूषकों (सर्फैक्टेंट्स और कार्बनिक प्रदूषकों) की उपस्थिति वाले नमूनों का उपयोग करके वास्तविक पर्यावरणीय परिस्थितियों में संचालित किया गया, तो इसकी अपघटन दक्षता कम हो गई, जिसने वास्तविक WW उपचार के लिए BCPR की सीमा को दिखाया। नीयन प्लाज़्मा में ऐसी कोई समस्या नहीं थी, और यह पारंपरिक अपशिष्ट जल उपचार प्रणाली के साथ भी संगत था। इसलिए, व्यावहारिक अनुप्रयोग के लिए नीयन प्लाज़्मा प्रणाली को अधिक उपयुक्त पाया गया। अंत में, नीयन प्लाज़्मा रिएक्टर को एक प्रभावी उन्नत WW उपचार प्रणाली के रूप में आगे मान्य करने के लिए, प्लाज़्मा उपचारित नमूनों की जोखिम मूल्यांकन और प्लाज़्मा प्रणाली अध्ययन की आर्थिक मूल्यांकन की गई, और इसके परिणाम भी नीयन प्लाज़्मा के पक्ष में थे।

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ABBREVIATIONS

AOPs	Advanced oxidation processes
APHA	American Public Health Association
Ar	Argon
ARB	Antibiotic Resistance Bacteria
ARG	Antibiotic Resistance Genes
ASE	Accelerated Solvent Extraction
ASP	Activated sludge process
CFU	Colony Forming Units
DBD	Dielectric Barrier Discharge
ECs	Emerging contaminants
He	Helium
HRAPs	High-rate algal ponds
HQ	Hazard Quotient
LCMS	Liquid chromatography-mass spectrometry
MBR	Membrane Bioreactor
MEC	Measured Environmental Concentration
Met. Blue	Methylene Blue
PFAS	Per- and Polyfluorinated Substances
PEC	Predictive effective concentration
PNEC	Predicted no effective concentration
PPCPs	Pharmaceuticals and personal care products
PPM	Parts per million
PTFE	Polytetrafluoroethylene
Rd. B	Rhodamine B
RONs	Reactive Oxygen and Nitrogen Species
SPE	Solid Phase Extraction
TOC	Total Organic Carbon
UASB	Up-flow anaerobic sludge blanket bioreactor
USEPA	U.S. Environmental Protection Agency
WWTPs	Wastewater treatment plants