

Volume 7, Issue 2, Year 2024 ISSN: 2645-9000

Owner

IZMIR DEMOCRACY UNIVERSITY

Chief Editor

Prof. Dr. Günay ÖZTÜRK

Vice Editors

Assoc. Prof. Tuğba KESKİN GÜNDOĞDU Assoc. Prof. Sema ÇARIKÇI

Journal Contact: Izmir Democracy University Natural and Applied Sciences Journal

E-mail: idunas@idu.edu.tr

Web: https://dergipark.org.tr/en/pub/idunas

Publisher: Izmir Democracy University

EDITORIAL BOARD

Head of Editorial Board: Prof. Dr. Günay OZTURK Vice Editor: Assoc. Prof. Tuğba KESKİN GÜNDOĞDU Vice Editor: Assoc. Prof. Sema ÇARIKÇI Departmental Editor: Assist. Prof. Gizem KALELİ CAN Publishing Editor: Assist. Prof. Gizem KALELİ CAN English Language Editor: Dr. Kudret ÖKTEM, Kadriye Tilbe ESER Layout Editor: Bengü GÜNGÖR

International Editorial Board Member: Prof. Dr. Elza Maria Morais Fonseca/ Polytechnic Ins of Porto International Editorial Board Member: Prof. Dr. Ercan Yılmaz/ Abant İzzet Baysal University International Editorial Board Member: Prof. Dr. Fatih İnci/ Standford University International Editorial Board Member: Prof. Dr. Hariharan Muthusamy/Nat Ins of Tech.-Uttarakhand International Editorial Board Member: Prof. Dr. Kemal Polat/ Abant İzzet Baysal University International Editorial Board Member: Prof. Dr. Murat Şimşek/İnönü University International Editorial Board Member: Prof. Dr. Sinan Akgöl/ Ege University International Editorial Board Member: Prof. Dr. Soner Çakmak/ Hacettepe University International Editorial Board Member: Assoc. Dr. Elif Güngör /Balıkesir University International Editorial Board Member: Asst. Selma Çelen Yücetürk/Balıkesir University International Editorial Board Member: Prof. Dr. Cihan Özgür/ Izmir Democracy University International Editorial Board Member: Prof. Dr. Timur Canel / Kocaeli University International Editorial Board Member: Prof. Dr. Irem Bağlan/ Kocaeli University International Editorial Board Member: Prof. Dr. Bengü Bayram/Balıkesir University International Editorial Board Member: Assoc.. Dr. Zeynel Abidin Çil/ Izmir Democracy University International Editorial Board Member: Assoc.. Dr. Damla Kızılay / Izmir Democracy University International Editorial Board Member: Assoc. Dr. Mine Güngörmüşler/İzmir Economy University International Editorial Board Member: Dr. Gülizar Çalişkan/ Izmir Democracy University International Editorial Board Member: Assoc.. Dr. Müge Işleten Hoşoğlu/ Gebze Tech. Uni. International Editorial Board Member: Assoc.. Dr. Gözde Duman Taç/ Ege University International Editorial Board Member: Dr. Sıdıka Tugçe Dağlıoğlu/ Ege University International Editorial Board Member: Asst. Prof. Hilal Betül Kaya Akkale/Manisa Celal Bayar Uni.

INDEXES AND DATABASES

IDUNAS is indexed by 4 national and international databases.

INDEXED DATABASES						
ACARINDEX	https://www.acarindex.com/search?type=article&q=N					
	atural+and+Applied+Sciences+Journal					
Academic Resource Index ResearchBib	https://journalseeker.researchbib.com/view/issn/264					
	5-9000					
Google Scholar	https://scholar.google.com.tr/scholar?q=izmir+democ					
	racy+university+natural+and+applied+sciences+journa l&hl=tr&as sdt=0&as vis=1&oi=scholart					
Ideal Online	https://www.idealonline.com.tr/IdealOnline/					
APP	LIED DATABASES					
Directory of Research Journals Indexing	http://olddrji.lbp.world/					
Directory of Academic and Scientific	https://ojs.europubpublications.com/ojs/index.php					
Journals						
OJOP	https://www.ojop.org/					

ABOUT

Natural & Applied Sciences Journal is an international open access, peer-reviewed, free of cost academic journal that includes articles and reviews on natural and applied sciences. It is published twice a year in June and December. Our journal accepts only English content from the second issue.

AIM & SCOPE

It aims to contribute to the knowledge of the field of natural and applied sciences by publishing qualified scientific studies in the field.

Natural & Applied Sciences Journal is an international open access, peer-reviewed, free of cost academic journal that includes articles and reviews on natural and applied sciences published by İzmir Democracy University. It is published twice a year in June and December. Our journal accepts only English content from the second issue.

PERIODS

June and December

ETHICAL PRINCIPLES AND PUBLICATION POLICY

All articles submitted for publication in the journal must conform to the ethical rules of scientific research. The authors of each article are required to sign the Copyright Form confirming that they have granted permission for their work to be published in Natural and Applied Science Journal. Publication will not take place, even if the manuscript is accepted without this form. Authors are solely responsible for the content of their articles and any responsibilities they may incur regarding copyrights. The work submitted to the journal should not have been published in any language, in any journal, or in the process of being evaluated in any other publication.

Articles should be prepared in accordance with the general ethical rules specified by DOI and DOAJ. Plagiarism Control All articles submitted to Natural and Applied Science Journal are checked using the iThenticate plagiarism detection software. Based on the similarity report generated by the software, the editorial board determines whether the article should be submitted to peer review or rejected.

PRICE POLICY

No fee is charged from the author or institution under any name.

INTERNATIONAL STANDARDS FOR AUTHORS

RESPONSIBLE RESEARCH PUBLICATION

A position statement developed at the 2nd World Conference on Research Integrity, Singapore, July 22-24, 2010.

Elizabeth Wager & Sabine Kleinert

Contact details: liz@sideview.demon.co.uk sabine.kleinert@lancet.com

SUMMARY

• The research being reported should have been conducted in an ethical and responsible manner and should comply with all relevant legislation.

• Researchers should present their results clearly, honestly, and without fabrication, falsification, or inappropriate data manipulation.

• Researchers should strive to describe their methods clearly and unambiguously so that their findings can be confirmed by others.

• Researchers should adhere to publication requirements that submitted work is original, is not plagiarized, and has not been published elsewhere.

• Authors should take collective responsibility for submitted and published work.

• The authorship of research publications should accurately reflect individuals' contributions to the work and its reporting.

• Funding sources and relevant conflicts of interest should be disclosed.

Cite this as: Wager E & Kleinert S (2011) Responsible research publication: international standards for authors. A position statement developed at the 2nd World Conference on Research Integrity, Singapore, July 22-24, 2010. Chapter 50 in: Mayer T & Steneck N (eds) Promoting Research Integrity in a Global Environment. Imperial College Press / World Scientific Publishing, Singapore (pp 309-16). (ISBN 978-981-4340-97-7)

INTRODUCTION

Publication is the final stage of research and therefore a responsibility for all researchers. Scholarly publications are expected to provide a detailed and permanent record of research. Because publications form the basis for both new research and the application of findings, they can affect not only the research community but also, indirectly, society at large. Researchers therefore have a responsibility to ensure that their publications are honest, clear, accurate, complete and balanced, and should avoid misleading, selective or ambiguous reporting. Journal editors also have responsibilities for ensuring the integrity of the research literature and these are set out in companion guidelines.

This document aims to establish international standards for authors of scholarly research publications and to describe responsible research reporting practice. We hope these standards will be endorsed by research institutions, funders, and professional societies; promoted by editors and publishers; and will aid in research integrity training.

Responsible research publication

1 Soundness and reliability

1.1 The research being reported should have been conducted in an ethical and responsible manner and follow all relevant legislation. [See also the Singapore Statement on Research Integrity, www.singaporestatement.org]

1.2 The research being reported should be sound and carefully executed.

1.3 Researchers should use appropriate methods of data analysis and display (and, if needed, seek, and follow specialist advice on this).

1.4 Authors should take collective responsibility for their work and for the content of their publications. Researchers should check their publications carefully at all stages to ensure methods and findings are reported accurately. Authors should carefully check calculations, data presentations, typescripts/submissions, and proofs.

2 Honesty

2.1 Researchers should present their results honestly and without fabrication, falsification, or inappropriate data manipulation. Research images (e.g. micrographs, X-rays, pictures of electrophoresis gels) should not be modified in a misleading way.

2.2 Researchers should strive to describe their methods and to present their findings clearly and unambiguously. Researchers should follow applicable reporting guidelines. Publications should provide sufficient detail to permit experiments to be repeated by other researchers.

2.3 Reports of research should be complete. They should not omit inconvenient, inconsistent, or inexplicable findings or results that do not support the authors' or sponsors' hypothesis or interpretation.

2.4 Research funders and sponsors should not be able to veto publication of findings that do not favor their product or position. Researchers should not enter agreements that permit the research sponsor to veto or control the publication of the findings (unless there are exceptional circumstances, such as research classified by governments because of security implications).

2.5 Authors should alert the editor promptly if they discover an error in any submitted, accepted or published work. Authors should cooperate with editors in issuing corrections or retractions when required.

2.6 Authors should represent the work of others accurately in citations and quotations.

2.7 Authors should not copy references from other publications if they have not read the cited work.

3 Originality

3.1 Authors should adhere to publication requirements that submitted work is original and has not been published elsewhere in any language. Work should not be submitted concurrently to more than one publication unless the editors have agreed to co-publication. If articles are co-published this fact should be made clear to readers.

3.2 Applicable copyright laws and conventions should be followed. Copyright material (e.g. tables, figures, or extensive quotations) should be reproduced only with appropriate permission and acknowledgement.

3.3 Relevant previous work and publications, both by other researchers and the authors' own, should be properly acknowledged, and referenced. The primary literature should be cited where possible.

3.4 Data, text, figures, or ideas originated by other researchers should be properly acknowledged and should not be presented as if they were the authors' own. Original wording taken directly from publications by other researchers should appear in quotation marks with the appropriate citations.

3.5 Authors should inform editors if findings have been published previously or if multiple reports or multiple analyses of a single data set are under consideration for publication elsewhere. Authors should provide copies of related publications or work submitted to other journals.

3.6 Multiple publications arising from a single research project should be clearly identified as such and the primary publication should be referenced. Translations and adaptations for different audiences should be clearly identified as such, should acknowledge the original source, and

should respect relevant copyright conventions and permission requirements. If in doubt, authors should seek permission from the original publisher before republishing any work.

4 Appropriate authorship and acknowledgement

4.1 The research literature serves as a record not only of what has been discovered but also of who made the discovery. The authorship of research publications should therefore accurately reflect individuals' contributions to the work and its reporting.

4.2 In cases where major contributors are listed as authors while those who made less substantial, or purely technical, contributions to the research or to the publication are listed in an acknowledgement section, the criteria for authorship and acknowledgement should be agreed at the start of the project. Ideally, authorship criteria within a particular field should be agreed, published and consistently applied by research institutions, professional and academic societies, and funders. While journal editors should publish and promote accepted authorship criteria appropriate to their field, they cannot be expected to adjudicate in authorship disputes. Responsibility for the correct attribution of authorship lies with authors themselves working under the guidance of their institution. Research institutions should promote and uphold fair and accepted standards of authorship and acknowledgement. When required, institutions should adjudicate in authorship disputes and should ensure that due process is followed.

4.3 Researchers should ensure that only those individuals who meet authorship criteria (i.e. made a substantial contribution to the work) are rewarded with authorship and that deserving authors are not omitted. Institutions and journal editors should encourage practices that prevent guest, gift, and ghost authorship.

Note:

• Guest authors are those who do not

• Gift authors are those who do meet accepted authorship criteria but are listed because of their seniority, reputation or supposed influence not

• Ghost authors are those who meet authorship criteria but are not listed meet accepted authorship criteria but are listed as a personal favor or in return for payment

4.4 All authors should agree to be listed and should approve the submitted and accepted versions of the publication. Any change to the author list should be approved by all authors including any who have been removed from the list. The corresponding author should act as a point of contact between the editor and the other authors and should keep co-authors informed and involve them in major decisions about the publication (e.g. responding to reviewers' comments).

4.5 Authors should not use acknowledgements misleadingly to imply a contribution or endorsement by individuals who have not, in fact, been involved with the work or given an endorsement.

5 Accountability and responsibility

5.1 All authors should have read and be familiar with the reported work and should ensure that publications follow the principles set out in these guidelines. In most cases, authors will be expected to take joint responsibility for the integrity of the research and its reporting. However, if authors take responsibility only for certain aspects of the research and its reporting, this should be specified in the publication.

5.2 Authors should work with the editor or publisher to correct their work promptly if errors or omissions are discovered after publication.

5.3 Authors should abide by relevant conventions, requirements, and regulations to make materials, reagents, software, or datasets available to other researchers who request them. Researchers, institutions, and funders should have clear policies for handling such requests. Authors must also follow relevant journal standards. While proper acknowledgement is expected, researchers should not demand authorship as a condition for sharing materials.

5.4 Authors should respond appropriately to post-publication comments and published correspondence. They should attempt to answer correspondents' questions and supply clarification, or additional details where needed.

6 Adherence to peer review and publication conventions

6.1 Authors should follow publishers' requirements that work is not submitted to more than one publication for consideration at the same time.

6.2 Authors should inform the editor if they withdraw their work from review or choose not to respond to reviewer comments after receiving a conditional acceptance.

6.3 Authors should respond to reviewers' comments in a professional and timely manner.

6.4 Authors should respect publishers' requests for press embargos and should not generally allow their findings to be reported in the press if they have been accepted for publication (but not yet published) in a scholarly publication. Authors and their institutions should liaise and cooperate with publishers to coordinate media activity (e.g. press releases and press conferences) around publication. Press releases should accurately reflect the work and should not include statements that go further than the research findings.

7 Responsible reporting of research involving humans or animals

7.1 Appropriate approval, licensing or registration should be obtained before the research begins and details should be provided in the report (e.g. Institutional Review Board, Research Ethics Committee approval, national licensing authorities for the use of animals).

7.2 If requested by editors, authors should supply evidence that reported research received the appropriate approval and was carried out ethically (e.g. copies of approvals, licenses, participant consent forms).

7.3 Researchers should not generally publish or share identifiable individual data collected in the course of research without specific consent from the individual (or their representative). Researchers should remember that many scholarly journals are now freely available on the internet and should therefore be mindful of the risk of causing danger or upset to unintended readers (e.g. research participants or their families who recognize themselves from case studies, descriptions, images, or pedigrees).

7.4 The appropriate statistical analyses should be determined at the start of the study and a data analysis plan for the prespecified outcomes should be prepared and followed. Secondary or post hoc analyses should be distinguished from primary analyses and those set out in the data analysis plan.

7.5 Researchers should publish all meaningful research results that might contribute to understanding. In particular, there is an ethical responsibility to publish the findings of all clinical trials. The publication of unsuccessful studies or experiments that reject a hypothesis may help prevent others from wasting time and resources on similar projects. If findings from small studies and those that fail to reach statistically significant results can be combined to produce more useful information (e.g. by meta-analysis) then such findings should be published.

7.6 Authors should supply research protocols to journal editors if requested (e.g. for clinical trials) so that reviewers and editors can compare the research report to the protocol to check that it was carried out as planned and that no relevant details have been omitted. Researchers should follow relevant requirements for clinical trial registration and should include the trial registration number in all publications arising from the trial.

INTERNATIONAL STANDARDS FOR EDITORS

RESPONSIBLE RESEARCH PUBLICATION

A position statement developed at the 2nd World Conference on Research Integrity, Singapore, July 22-24, 2010.

Sabine Kleinert & Elizabeth Wager

Contact details: sabine.kleinert@lancet.com

liz@sideview.demon.co.uk

Cite this as: Kleinert S & Wager E (2011) Responsible research publication: international standards for editors. A position statement developed at the 2nd World Conference on Research Integrity, Singapore, July 22-24, 2010. Chapter 51 in: Mayer T & Steneck N (eds) Promoting Research Integrity in a Global Environment. Imperial College Press / World Scientific Publishing, Singapore (pp 317-28). (ISBN 978-981-4340-97-7)

Summary

• Editors are accountable and should take responsibility for everything they publish

• Editors should make fair and unbiased decisions independent from commercial consideration and ensure a fair and appropriate peer review process

• Editors should adopt editorial policies that encourage maximum transparency and complete, honest reporting

• Editors should guard the integrity of the published record by issuing corrections and retractions when needed and pursuing suspected or alleged research and publication misconduct

• Editors should pursue reviewer and editorial misconduct

- Editors should critically assess the ethical conduct of studies in humans and animals
- Peer reviewers and authors should be told what is expected of them
- Editors should have appropriate policies in place for handling editorial conflicts of interest

Introduction

As guardians and stewards of the research record, editors should encourage authors to strive for, and adhere themselves to, the highest standards of publication ethics. Furthermore, editors are in a unique position to indirectly foster responsible conduct of research through their policies and processes. To achieve the maximum effect within the research community, ideally all editors should adhere to universal standards and good practices. While there are important differences between different fields and not all areas covered are relevant to each research community, there are important common editorial policies, processes, and principles that editors should follow to ensure the integrity of the research record.

These guidelines are a starting point and are aimed at journal editors in particular. While books and monographs are important and relevant research records in many fields, guidelines for book editors are beyond the scope of these recommendations. It is hoped that in due course such guidelines can be added to this document.

Editors should regard themselves as part of the wider professional editorial community, keep themselves abreast of relevant policies and developments, and ensure their editorial staff is trained and kept informed of relevant issues.

To be a good editor requires many more principles than are covered here. These suggested principles, policies, and processes are particularly aimed at fostering research and publication integrity.

Editorial Principles

1. Accountability and responsibility for journal content

Editors have to take responsibility for everything they publish and should have procedures and policies in place to ensure the quality of the material they publish and maintain the integrity of the published record (see paragraphs 4-8).

2. Editorial independence and integrity

An important part of the responsibility to make fair and unbiased decisions is the upholding of the principle of editorial independence and integrity.

2.1 Separating decision-making from commercial considerations

Editors should make decisions on academic merit alone and take full responsibility for their decisions. Processes must be in place to separate commercial activities within a journal from editorial processes and decisions. Editors should take an active interest in the publisher's pricing policies and strive for wide and affordable accessibility of the material they publish.

Sponsored supplements must undergo the same rigorous quality control and peer review as any other content for the journal. Decisions on such material must be made in the same way as any other journal content. The sponsorship and role of the sponsor must be clearly declared to readers.

Advertisements need to be checked so that they follow journal guidelines, should be clearly distinguishable from other content, and should not in any way be linked to scholarly content.

2.2 Editors' relationship to the journal publisher or owner

Editors should ideally have a written contract setting out the terms and conditions of their appointment with the journal publisher or owner. The principle of editorial independence should be clearly stated in this contract. Journal publishers and owners should not have any role in decisions on content for commercial or political reasons. Publishers should not dismiss an editor because of any journal content unless there was gross editorial misconduct, or an independent investigation has concluded that the editor's decision to publish was against the journal's scholarly mission.

2.3 Journal metrics and decision-making

Editors should not attempt to inappropriately influence their journal's ranking by artificially increasing any journal metric. For example, it is inappropriate to demand that references to that journal's articles are included except for genuine scholarly reasons. In general, editors should ensure that papers are reviewed on purely scholarly grounds and that authors are not pressured to cite specific publications for non- scholarly reasons.

3. Editorial confidentiality

3.1 Authors' material

If a journal operates a system where peer reviewers are chosen by editors (rather than posting papers for all to comment as a pre-print version), editors must protect the confidentiality of authors' material and remind reviewers to do so as well. In general, editors should not share submitted papers with editors of other journals, unless with the authors' agreement or in cases of alleged misconduct (see below). Editors are generally under no obligation to provide material to lawyers for court cases. Editors should not give any indication of a paper's status with the journal to anyone other than the authors. Web-based submission systems must be run in a way that prevents unauthorized access.

In the case of a misconduct investigation, it may be necessary to disclose material to third parties (e.g., an institutional investigation committee or other editors).

3.2 Reviewers

Editors should protect reviewers' identities unless operating an open peer review system. However, if reviewers wish to disclose their names, this should be permitted.

If there is alleged or suspected reviewer misconduct it may be necessary to disclose a reviewer's name to a third party.

General editorial policies

4. Encourage maximum transparency and complete and honest reporting

To advance knowledge in scholarly fields, it is important to understand why particular work was done, how it was planned and conducted and by whom, and what it adds to current knowledge. To achieve this understanding, maximum transparency and complete and honest reporting are crucial.

4.1 Authorship and responsibility

Journals should have a clear policy on authorship that follows the standards within the relevant field. They should give guidance in their information for authors on what is expected of an author and, if there are different authorship conventions within a field, they should state which they adhere to.

For multidisciplinary and collaborative research, it should be apparent to readers who has done what and who takes responsibility for the conduct and validity of which aspect of the research. Each part of the work should have at least one author who takes responsibility for its validity. For example, individual contributions and responsibilities could be stated in a contributor section. All authors are expected to have contributed significantly to the paper and to be familiar with its entire content and ideally, this should be declared in an authorship statement submitted to the journal.

When there are undisputed changes in authorship for appropriate reasons, editors should require that all authors (including any whose names are being removed from an author list) agree

with these in writing. Authorship disputes (i.e., disagreements on who should or should not be an author before or after publication) cannot be adjudicated by editors and should be resolved at institutional level or through other appropriate independent bodies for both published and unpublished papers. Editors should then act on the findings, for example by correcting authorship in published papers.

Journals should have a publicly declared policy on how papers submitted by editors or editorial board members are handled (see paragraph on editorial conflicts of interest: 8.2).

4.2 Conflicts of interest and role of the funding source

Editors should have policies that require all authors to declare any relevant financial and non-financial conflicts of interest and publish at least those that might influence a reader's perception of a paper, alongside the paper. The funding source of the research should be declared and published, and the role of the funding source in the conception, conduct, analysis, and reporting of the research should be stated and published.

Editors should make it clear in their information for authors if in certain sections of the journal (e.g., commissioned commentaries or review articles) certain conflicts of interest preclude authorship.

4.3 Full and honest reporting and adherence to reporting guidelines

Among the most important responsibilities of editors is to maintain a high standard in scholarly literature. Although the standards differ among journals, editors should work to ensure that all published papers make a substantial new contribution to their field. Editors should discourage so-called 'salami publications' (i.e., publication of the minimum publishable unit of research), avoid duplicate or redundant publication unless it is fully declared and acceptable to all (e.g., publication in a different language with cross-referencing), and encourage authors to place their work in the context of previous work (i.e., to state why this work was necessary/done, what this work adds or why a replication of previous work was required, and what readers should take away from it).

Journals should adopt policies that encourage full and honest reporting, for example, by requiring authors in fields where it is standard to submit protocols or study plans, and, where they exist, to provide evidence of adherence to relevant reporting guidelines. Although devised to improve reporting, adherence to reporting guidelines also makes it easier for editors, reviewers, and readers to judge the actual conduct of the research.

Digital image files, figures, and tables should adhere to the appropriate standards in the field. Images should not be inappropriately altered from the original or present findings in a misleading way.

Editors might also consider screening for plagiarism, duplicate or redundant publication by using anti-plagiarism software, or for image manipulation. If plagiarism or fraudulent image manipulation is detected, this should be pursued with the authors and relevant institutions (see paragraph on how to handle misconduct: 5.2)

5. Responding to criticisms and concerns

Reaction and response to published research by other researchers is an important part of scholarly debate in most fields and should generally be encouraged. In some fields, journals can facilitate this debate by publishing readers' responses. Criticism may be part of a general scholarly debate but can also highlight transgressions of research or publication integrity.

5.1 Ensuring integrity of the published record - corrections

When genuine errors in published work are pointed out by readers, authors, or editors, which do not render the work invalid, a correction (or erratum) should be published as soon as possible. The online version of the paper may be corrected with a date of correction and a link to the printed erratum. If the error renders the work or substantial parts of it invalid, the paper should be retracted with an explanation as to the reason for retraction (i.e., honest error).

5.2 Ensuring the integrity of the published record – suspected research or publication misconduct

If serious concerns are raised by readers, reviewers, or others, about the conduct, validity, or reporting of academic work, editors should initially contact the authors (ideally all authors) and allow them to respond to the concerns. If that response is unsatisfactory, editors should take this to the institutional level (see below). In rare cases, mostly in the biomedical field, when concerns are very serious and the published work is likely to influence clinical practice or public health, editors should consider informing readers about these concerns, for example by issuing an 'expression of concern', while the investigation is ongoing. Once an investigation is concluded, the appropriate action needs to be taken by editors with an accompanying comment that explains the findings of the investigation. Editors should also respond to findings from national research integrity organizations that indicate misconduct relating to a paper published in their journal. Editors can themselves decide to retract a paper if they are convinced that serious misconduct has happened even if an investigation by an institution or national body does not recommend it.

Editors should respond to all allegations or suspicions of research or publication misconduct raised by readers, reviewers, or other editors. Editors are often the first recipients of information about such concerns and should act, even in the case of a paper that has not been accepted or has already been rejected. Beyond the specific responsibility for their journal's publications, editors have a collective responsibility for the research record and should act whenever they become aware of potential misconduct if possible. Cases of possible plagiarism or duplicate/redundant publication can be assessed by editors themselves. However, in most other cases, editors should request an investigation by the institution or other appropriate bodies (after seeking an explanation from the authors first and if that explanation is unsatisfactory).

Retracted papers should be retained online, and they should be prominently marked as a retraction in all online versions, including the PDF, for the benefit of future readers.

For further guidance on specific allegations and suggested actions, such as retractions, see the COPE flowcharts and retraction guidelines (http://publicationethics.org/flowcharts; http://publicationethics.org/files/u661/Retractions COPE gline final 3 Sept 09 2 .pdf).

5.3 Encourage scholarly debate

All journals should consider the best mechanism by which readers can discuss papers, voice criticisms, and add to the debate (in many fields this is done via a print or on-line correspondence section). Authors may contribute to the debate by being allowed to respond to comments and criticisms where relevant. Such a scholarly debate about published work should happen in a timely manner. Editors should clearly distinguish between criticisms of the limitations of a study and criticisms that raise the possibility of research misconduct. Any criticisms that raise the possibility of misconduct should not just be published but should be further investigated even if they are received a long time after publication. Editorial policies relevant only to journals that publish research in humans or animals.

6. Critically assess and require a high standard of ethical conduct of research

Especially in biomedical research but also in social sciences and humanities, ethical conduct of research is paramount in the protection of humans and animals. Ethical oversight, appropriate consent procedures, and adherence to relevant laws are required by authors. Editors need to be vigilant to concerns in this area.

6.1 Ethics approval and ethical conduct

Editors generally require approval of a study by an ethics committee (or institutional review board) and the assurance that it was conducted according to the Declaration of Helsinki for medical research in humans but, in addition, should be alert to areas of concern in the ethical conduct of research. This may mean that a paper is sent to peer reviewers with particular expertise in this area, to the journal's ethics committee if there is one, or that editors require further reassurances or evidence from authors or their institutions.

Papers may be rejected on ethical grounds even if the research had ethics committee approval.

6.2 Consent (to take part in research)

If research is done in humans, editors should ensure that a statement on the consent procedure is included in the paper. In most cases, written informed consent is the required norm. If there is any concern about the consent procedure, if the research is done in vulnerable groups, or if there are doubts about ethical conduct, editors should ask to see the consent form and enquire further from authors, exactly how consent was obtained.

6.3 Consent (for publication)

For all case reports, small case series, and images of people, editors should require the authors to have obtained explicit consent for publication (which is different from consent to take part in research). This consent should inform participants which journal the work will be published in, make it clear that, although all efforts will be made to remove unnecessary identifiers, complete anonymity is not possible, and ideally state that the person described has seen and agreed with the submitted paper.

The signed consent form should be kept with the patient file rather than sent to the journal (to maximize data protection and confidentiality, see paragraph 6.4). There may be exceptions where it is not possible to obtain consent, for example when the person has died. In such cases, careful consideration about possible harm is needed and out of courtesy attempts should be made to obtain assent from relatives. In very rare cases, an important public health message may justify publication without consent if it is not possible despite all efforts to obtain consent and the benefit of publication outweighs the possible harm.

6.4 Data protection and confidentiality

Editors should critically assess any potential breaches of data protection and patient confidentiality. This includes requiring properly informed consent for the actual research presented consent for publication where applicable (see paragraph 6.3) and having editorial policies that comply with guidelines on patient confidentiality.

6.5 Adherence to relevant laws and best practice guidelines for ethical conduct

Editors should require authors to adhere to relevant national and international laws and best practice guidelines where applicable, for example when undertaking animal research. Editors should encourage registration of clinical trials.

Editorial Processes

7. Ensuring a fair and appropriate peer review process

One of the most important responsibilities of editors is organizing and using peer review fairly and wisely. Editors should explain their peer review processes in the information for authors and also indicate which parts of the journal are peer reviewed.

7.1 Decision whether to review

Editors may reject a paper without peer review when it is deemed unsuitable for the journal's readers or is of poor quality. This decision should be made in a fair and unbiased way. The criteria used to make this decision should be made explicit. The decision not to send a paper for peer review should only be based on the academic content of the paper and should not be influenced by the nature of the authors or the host institution.

7.2 Interaction with peer reviewers

Editors should use appropriate peer reviewers for papers that are considered for publication by selecting people with sufficient expertise and avoiding those with conflicts of interest. Editors should ensure that reviews are received in a timely manner.

Peer reviewers should be told what is expected of them and should be informed about any changes in editorial policies. In particular, peer reviewers should be asked to assess research and publication ethics issues (i.e., whether they think the research was done and reported ethically, or if they have any suspicions of plagiarism, fabrication, falsification, or redundant publication). Editors should have a policy to request a formal conflict of interest declaration from peer reviewers and should ask peer reviewers to inform them about any such conflict of interest at the earliest

opportunity so that they can make a decision on whether an unbiased review is possible. Certain conflicts of interest may disqualify a peer reviewer. Editors should stress confidentiality of the material to peer reviewers and should require peer reviewers to inform them when they ask a colleague for help with a review or if they mentor a more junior colleague in conducting peer review. Editors should ideally have a mechanism to monitor the quality and timeliness of peer review and to provide feedback to reviewers.

7.3 Reviewer misconduct

Editors must take reviewer misconduct seriously and pursue any allegation of breach of confidentiality, non-declaration of conflicts of interest (financial or non-financial), inappropriate use of confidential material, or delay of peer review for competitive advantage. Allegations of serious reviewer misconduct, such as plagiarism, should be taken to the institutional level (for further guidance see: http://publicationethics.org/files/u2/07_Reviewer_misconduct.pdf).

7.4 Interaction with authors

Editors should make it clear to authors what the role of the peer reviewer is because this may vary from journal to journal. Some editors regard peer reviewers as advisors and may not necessarily follow (or even ask for) reviewers' recommendations on acceptance or rejection. Correspondence from editors is usually with the corresponding author, who should guarantee to involve co-authors at all stages. Communicating with all authors at first submission and at final acceptance stage can be helpful to ensure all authors are aware of the submission and have approved the publication. Normally, editors should pass on all peer reviewers' comments in their entirety. However, in exceptional cases, it may be necessary to exclude parts of a review, if it, for example, contains libelous or offensive remarks. It is important, however, that such editorial discretion is not inappropriately used to suppress inconvenient comments.

There should always be good reasons, which are clearly communicated to authors, if additional reviewers are sought at a late stage in the process.

The final editorial decision and reasons for this should be clearly communicated to authors and reviewers. If a paper is rejected, editors should ideally have an appeals process. Editors, however, are not obliged to overturn their decision.

8. Editorial decision-making

Editors are in a powerful position by making decisions on publications, which makes it very important that this process is as fair and unbiased as possible, and is in accordance with the academic vision of the particular journal.

8.1 Editorial and journal processes

All editorial processes should be made clear in the information for authors. In particular, it should be stated what is expected of authors, which types of papers are published, and how papers are handled by the journal. All editors should be fully familiar with the journal policies, vision, and scope. The final responsibility for all decisions rests with the editor-in-chief.

8.2 Editorial conflicts of interest

Editors should not be involved in decisions about papers in which they have a conflict of interest, for example if they work or have worked in the same institution and collaborated with the authors, if they own stock in a particular company, or if they have a personal relationship with the authors. Journals should have a defined process for handling such papers. Journals should also have a process in place to handle papers submitted by editors or editorial board members to ensure unbiased and independent handling of such papers. This process should be stated in the information for authors. Editorial conflicts of interest should be declared, ideally publicly.

TABLE OF CONTENTS

RESEARCH ARTICLES	PAGES
Akbala Yernazar, İrem Bağlan	
Fourier Analysis of Inverse Coefficient Nonlinear Hyperbolic Equations under Periodic Boundary Conditions	1 - 7
David N. Enyiukwu, Anderson C. Amadioha, Inemesit N. Bassey	
Ethnobotany and Bioactive Constituents of <i>Tabernaemontana pachysiphon</i> Stapf (Apocynaceae) Used in Indigenous Medicine Practices in Abia State, Nigeria	8 - 28
Majekodunmi Adedayo, Taofeeq Abdulkareem	20 20
Bacteriocinogenic Activity of <i>Lysinibacillus fusiformis</i> NR_042072.1 Isolated from Cow Milk	29 - 38
Ahmad Hosseinpour Sheikhrajab, Ahmet Erdal Osmanlıoğlu	20 (0
The Effect of Tunçbilek Thermal Power Plant Waste Fly Ash on Mechanical Properties of Portland Cement	39 - 60
İrem Bağlan	
The Heat Problem with Non-Local Boundary Conditions	61 - 68
Satılmış ÜRGÜN, Mustafa Özgür BORA, Sinan FİDAN, Şeref Tosunoğlu, Timur CANEL	
Determination of the Optimum Laser Processing Parameters Required to Obtain a Stable Hydrophobic Surface on the Surface of AA1050 Aluminum Alloy	69 - 85

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

Fourier Analysis of Inverse Coefficient Nonlinear Hyperbolic Equations under Periodic Boundary Conditions

Research Article

Akbala Yernazar 🔟, İrem Bağlan 🔟

Kocaeli University, Faculty of Arts and Sciences, Department of Mathematics, Kocaeli, Türkiye

Author E-mails: kz.akbala.95@gmail.com isakinc@kocaeli.edu.tr A. Yernazar ORCID ID: 0000-0003-4900-6027 İ. Bağlan ORCID ID: 0000-0002-1877-9791

*Correspondence to: İrem Bağlan, Kocaeli University, Faculty of Arts and Sciences, Department of Mathematics, Kocaeli, Türkiye DOI: 10.38061/idunas.1590039

Received: 22.11.2024; Accepted: 09.12.2024

Abstract

This study presents an analytical investigation of a one-dimensional inverse coefficient nonlinear hyperbolic equation with periodic boundary conditions. The analytical solution is derived from applying the Fourier method. An iterative approach is used to establish convergence and to assess the existence, uniqueness, and stability of the solution to the nonlinear problem.

Keywords: The hyperbolic problem, Fourier iterative method, Nonlocal conditions.

1. INTRODUCTION

The study of inverse coefficient problems in hyperbolic equations has gained significant attention recently due to its wide-ranging applications in physics, engineering, and medical imaging. These problems are centered around the challenge of identifying unknown coefficients or parameters from observed data, a task that can be quite difficult given their inherently ill-posed nature. In this context, Tekin [1] highlights the conditions necessary for the unique solvability of an inverse problem related to second-order hyperbolic equations, stressing the critical role that additional constraints can play in ensuring a well-defined solution.

The introduction of periodic boundary conditions adds another layer of complexity to these inverse problems, as they impose specific constraints on the solutions over a defined interval. Such periodic boundary conditions are vital in various physical scenarios, such as the vibrations of strings or the propagation of waves in bounded media, where system behavior exhibits periodicity over time [2]. Research into the uniqueness and stability of solutions under periodic conditions has shown that, despite the ill-posedness of these problems, unique solutions can still be obtained under certain circumstances [3-5].

(1)

One particularly effective approach to tackling these challenges is the Fourier method, which has been successfully employed in numerous studies to address inverse coefficient problems [6]. This method has demonstrated its capability to establish the existence, uniqueness, convergence, and stability of solutions for a variety of equations, including the Euler-Bernoulli, heat, Burger, and Klein-Gordon equations with periodic boundary conditions [7-9].

In this study, we consider an inverse coefficient nonlinear hyperbolic equation represented as $v_{tt} - v_{xx} = \theta(t) f(y,t,v), (y,t) \in \Omega$

with the initial condition $v(y,0) = \varphi(y)$ $v_t(y,t) = \psi(y),$ (2) the periodic boundary condition $v(0,t) = v(\pi,t)$ $v_y(0,t) = v_y(\pi,t)$ (3) and overdetermination data $E(t) = \int_0^{\pi} yv(y,t)dy$ (4)

for a nonlinear source term represented by f(y, t, v).

Here $\Omega := \{0 < y < \pi, 0 < t < T\}, \overline{\Omega} := \{0 \le y \le \pi, 0 \le t \le T\}.$ The functions φ, ψ, E and f(y, t, v) are given functions. Here $\phi(x) \in [0, \pi], \psi(x) \in [0, \pi]$ and $f(y, t, v), \overline{\Omega} \times (-\infty, \infty)$, for $(y, t) \in \overline{\Omega}, v(y, t) \in (-\infty, \infty)$.

By using the Fourier method, the solution function, determined by $\{\theta, \nu\}$, can be found. The existence, uniqueness, and stability of the solutions to the inverse problems are proven using an iterative approach.

2. ANALYTICAL SOLUTION OF THE PROBLEM

Definition 1. The problem of finding the values of $\{\theta, \nu\}$ that satisfy (1)-(4) is known as the inverse problem.

Definition 2. If the set $\{v(t)\} = \{v_0(t), v_{sk}(t), v_{ck}(t), k = \overline{1, N}\}$ of continuous functions on [0, T] satisfies the norm condition $\|v(t)\| = \max_{0 \le t \le T} |v_0(t)| + \sum_{k=1}^{\infty} \left(\max_{0 \le t \le T} |v_{ck}(t)| + \max_{0 \le t \le T} |v_{sk}(t)|\right)$, then space B is called a Banach space.

Let us assume the following conditions on the data for the problem (1)-(4) (C1) $E(t) \in C^2[0,T], \theta(t) \in C[0,T].$ (C2) $\varphi(y) \in C^1[0,\pi], \psi(y) \in C[0,\pi].$ (C3) Let f(y,t,v) be a function that is continuous in all arguments $\Omega \times (-\infty,\infty)$ and satisfies the following conditions:

1)
$$\left|\frac{\partial^{(k)}f(y,t,v)}{\partial y^{(k)}} - \frac{\partial^{(k)}f(y,t,\tilde{v})}{\partial y^{(k)}}\right| \le b(y,t)|v-\tilde{v}|, k = \overline{0,2}, \text{ where } b(y,t) \in L_2(\Omega), b(y,t) \ge 0.$$

2) $f(y,t,v) \in C[0,\pi], t \in [0,T], |f(y,t,v)| \le M,$
3) $\int_0^{\pi} f(y,t,v)dy \ne 0, \forall t \in [0,T].$

By applying the Fourier method, the solution to (1)-(3) is obtained as follows:

(6)

$$\nu(y,t) = \frac{1}{2} \left(\varphi_0 + \psi_0 t + \int_0^t (t-\tau) \theta(\tau) f_0(\tau) d\tau \right)$$

+ $\sum_{k=1}^{\infty} \left(\varphi_{ck} \cos 2kt + \frac{\psi_{ck}}{2k} \sin 2kt \right) \cos 2ky + \sum_{k=1}^{\infty} \left(\frac{1}{2k} \int_0^t \theta(\tau) f_{ck}(\tau) \sin 2k(t-\tau) d\tau \right) \cos 2ky$
+ $\sum_{k=1}^{\infty} \left(\varphi_{sk} \cos 2kt + \frac{\psi_{sk}}{2k} \sin 2kt \right) \sin 2ky + \sum_{k=1}^{\infty} \left(\frac{1}{2k} \int_0^t \theta(\tau) f_{sk}(\tau) \sin 2k(t-\tau) d\tau \right) \sin 2ky.$ (5)

Differentiating the overdetermination condition under conditions (C1)–(C3), we obtain: $E''(t) = \int_0^{\pi} y v_{tt} dy.$

Equations (5), (6) yield

$$\theta(t) = \frac{E''(t)}{\int_0^{\pi} yf(y,t,v)dy} - \frac{\pi \sum_{k=1}^{\infty} (2k) \left(\varphi_{sk} \cos 2kt + \frac{\psi_{sk}}{2k} \sin 2kt\right)}{\int_0^{\pi} yf(y,t,v)dy} + \frac{\pi \sum_{k=1}^{\infty} \left(\int_0^t \theta(\tau) f_{sk}(\tau) \sin 2k(t-\tau)d\tau\right)}{\int_0^{\pi} yf(y,t,v)dy}.$$
(7)

3. THE EXISTENCE AND UNIQUENESS OF SOLUTIONS

Theorem 1. If conditions (C1)–(C3) are satisfied, then the problem (1)–(4) has a unique solution.

$$\begin{aligned} & \text{Proof. Let us provide an iteration for (5) and the inverse coefficient as follows:} \\ & v_{0}^{(N+1)}(t) = \varphi_{0} + \psi_{0}t + \frac{2}{\pi} \int_{0}^{t} \int_{0}^{\pi} (t-\tau)\theta^{(N)}(\tau)f(\xi,\tau,v^{(N)})d\xi d\tau, \\ & v_{ck}^{(N+1)}(t) = \varphi_{ck} \cos 2kt + \frac{\psi_{ck}}{2k} \sin 2kt + \frac{1}{\pi k} \int_{0}^{t} \int_{0}^{\pi} \theta^{(N)}(\tau)f(\xi,\tau,v^{(N)}) \cos 2k\xi \sin 2k(t-\tau)d\xi d\tau, \\ & v_{sk}^{(N+1)}(t) = \varphi_{sk} \cos 2kt + \frac{\psi_{sk}}{2k} \sin 2kt + \frac{1}{\pi k} \int_{0}^{t} \int_{0}^{\pi} \theta^{(N)}(\tau)f(\xi,\tau,v^{(N)}) \sin 2k\xi \sin 2k(t-\tau)d\xi d\tau, \\ & \theta^{(N+1)}(t) = \frac{E''(t)}{\int_{0}^{\pi} yf(y,t,v^{(N)})dy} - \frac{\pi \sum_{k=1}^{\infty} (2k) \left(\varphi_{sk} \cos 2kt + \frac{\psi_{sk}}{2k} \sin 2kt\right)}{\int_{0}^{\pi} yf(y,t,v^{(N)})dy} + \frac{\pi \sum_{k=1}^{\infty} \left(\int_{0}^{t} \theta^{(N)}(\tau)f_{sk}(\tau) \sin 2k(t-\tau)d\tau\right)}{\int_{0}^{\pi} yf(y,t,v^{(N)})dy}. \end{aligned}$$
(8)

 $v^{(0)}(t) \in B, t \in [0, T]$ is from the conditions of the theorem.

For N = 0, by adding and subtracting $\int_{0}^{t} \int_{0}^{\pi} f(\xi, \tau, 0) d\xi d\tau$ in (8) and applying Cauchy inequality, we obtain $|v_{0}^{(1)}(t)| = |\varphi_{0} + \psi_{0}t| + \left(\int_{0}^{t} (t-\tau)^{2} d\tau\right)^{\frac{1}{2}} \left(\int_{0}^{t} \left\{ \left| \frac{2}{\pi} \int_{0}^{\pi} \theta^{(0)}(\tau) \left[f(\xi, \tau, v^{(0)}) - f(\xi, \tau, 0) \right] d\xi \right| \right\}^{2} d\tau \right)^{\frac{1}{2}}$ $+ \left(\int_{0}^{t} (t-\tau)^{2} d\tau\right)^{\frac{1}{2}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \left| \int_{0}^{\pi} \theta^{(0)}(\tau) f(\xi, \tau, 0) d\xi \right| \right\}^{2} d\tau \right)^{\frac{1}{2}},$ $|v_{ck}^{(1)}(t)| \le |\varphi_{ck}| + \left| \frac{\psi_{ck}}{2k} \right| + \left(\int_{0}^{t} d\tau\right)^{\frac{1}{2}} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi, \tau, 0) \cos 2k\xi \sin 2k(t-\tau) \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}},$

$$\begin{aligned} \left| v_{sk}^{(1)}(t) \right| &\leq \left| \varphi_{sk} \right| + \left| \frac{|\psi_{sk}|}{2k} \right| + \left(\int_{0}^{t} d\tau \right)^{\frac{1}{2}} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) \left[f(\xi, \tau, \nu^{(0)}) - f(\xi, \tau, 0) \right] \sin 2k\xi \sin 2k(t-\tau) \right] \right\}^{2} d\xi d\tau \right)^{\frac{1}{2}} \\ &+ \left(\int_{0}^{t} d\tau \right)^{\frac{1}{2}} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi, \tau, 0) \sin 2k\xi \sin 2k(t-\tau) \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}}. \end{aligned}$$

After using Lipschitz condition, we have

$$\begin{aligned} \left| v_{0}^{(1)}(t) \right| &\leq \left| \varphi_{0} + \psi_{0} t \right| + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi, \tau) v^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi, \tau, 0) \right| d\xi \right\}^{2} d\tau \right), \\ \left| v_{ck}^{(1)}(t) \right| &\leq \left| \varphi_{ck} \right| + \left| \frac{\psi_{ck}}{2k} \right| + \sqrt{t} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi, \tau) v^{(0)}(\xi, \tau) \cos 2k\xi \sin 2k(t - \tau) \right| \right\}^{2} d\xi d\tau \right)^{\frac{1}{2}} + \sqrt{t} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi, \tau, 0) \cos 2k\xi \sin 2k(t - \tau) \right| \right\}^{2} d\xi d\tau \right)^{\frac{1}{2}}, \\ \left| v_{sk}^{(1)}(t) \right| &\leq \left| \varphi_{sk} \right| + \left| \frac{\psi_{sk}}{2k} \right| + \sqrt{t} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi, \tau) v^{(0)}(\xi, \tau) \sin 2k\xi \sin 2k(t - \tau) \right| \right\}^{2} d\xi d\tau \right)^{\frac{1}{2}} + \sqrt{t} \left(\int_{0}^{t} \left\{ \frac{1}{\pi k} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi, \tau, 0) \sin 2k\xi \sin 2k(t - \tau) \right| \right\}^{2} d\xi d\tau \right)^{\frac{1}{2}}. \end{aligned}$$

By applying Hölder inequality,

$$\begin{aligned} \left| v_{0}^{(1)}(t) \right| &\leq \left| \varphi_{0} + \psi_{0} t \right| + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi,\tau) v^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi,\tau,0) \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} + \sqrt{t} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right)^{\frac{1}{2}} \left(\sum_{k=1}^{\infty} \frac{1}{k^{2}} \right$$

and applying Bessel's inequality, we have

$$\begin{aligned} \left| \nu_{0}^{(1)}(t) \right| &\leq \left| \varphi_{0} + \psi_{0} t \right| + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi,\tau) \nu^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t^{3}}{3}} \left(\int_{0}^{t} \left\{ \frac{2}{\pi} \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi,\tau,0) \right| d\xi \right\}^{2} d\tau \right), \\ \left| \nu_{ck}^{(1)}(t) \right| &\leq \sum_{k=1}^{\infty} \left| \varphi_{ck} \right| + \frac{1}{2} \sqrt{\frac{\pi^{2}}{6}} \left(\sum_{k=1}^{\infty} \left| \psi_{ck} \right|^{2} \right)^{\frac{1}{2}} + \sqrt{\frac{t}{6}} \left(\int_{0}^{t} \left\{ \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi,\tau) \nu^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t}{6}} \left(\int_{0}^{t} \left\{ \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi,\tau) \nu^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t}{6}} \left(\int_{0}^{t} \left\{ \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi,\tau,0) \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}}, \\ \left| \nu_{sk}^{(1)}(t) \right| &\leq \sum_{k=1}^{\infty} \left| \varphi_{sk} \right| + \frac{1}{2} \sqrt{\frac{\pi^{2}}{6}} \left(\sum_{k=1}^{\infty} \left| \psi_{sk} \right|^{2} \right)^{\frac{1}{2}} + \sqrt{\frac{t}{6}} \left(\int_{0}^{t} \left\{ \int_{0}^{\pi} \left| \theta^{(0)}(\tau) b(\xi,\tau) \nu^{(0)} \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}} + \sqrt{\frac{t}{6}} \left(\int_{0}^{t} \left\{ \int_{0}^{\pi} \left| \theta^{(0)}(\tau) f(\xi,\tau,0) \right| d\xi \right\}^{2} d\tau \right)^{\frac{1}{2}}. \end{aligned}$$

Finally, we get

$$\begin{aligned} \left\| v^{(1)}(t) \right\| &= \max_{0 \le t \le T} \frac{\left| v_0^{(1)}(t) \right|}{2} + \sum_{k=1}^{\infty} \left[\max_{0 \le t \le T} \left| v_{ck}^{(1)}(t) \right| + \max_{0 \le t \le T} \left| v_{sk}^{(1)}(t) \right| \right] \le \\ \frac{\left\| \varphi_0 \right\| + \left\| \psi_0 \right\| \left\| T \right\|}{2} + \sum_{k=1}^{\infty} \left\| \varphi_{ck} \right\| + \left\| \varphi_{sk} \right\| + \frac{\pi}{2\sqrt{6}} \sum_{k=1}^{\infty} \left\| \psi_{ck} \right\| + \left\| \psi_{sk} \right\| + A \left\| \theta^{(0)}(t) \right\| \left\| b(y,t) \right\| \left\| v^{(0)}(t) \right\| + A \left\| \theta^{(0)}(t) \right\| M, \end{aligned}$$
where $A = \left(\frac{\sqrt{3}T\sqrt{T}(T + \sqrt{2}\pi)}{3} \right).$
Hence from the conditions of the theorem $v^{(1)}(t) \in B, t \in [0,T].$

If considered
$$2k \int_0^{\pi} \varphi(y) \sin 2ky dy = \int_0^{\pi} \varphi'(y) \cos 2ky dy$$
 for (7), we have

$$\theta^{(1)}(t) = \frac{E''(t) - \pi \sum_{k=1}^{\infty} \varphi'_{ck} \cos 2kt - \pi \sum_{k=1}^{\infty} \psi_{sk} \sin 2kt}{\int_0^{\pi} yf(y,t,v^{(0)}) dy} - \frac{2\sum_{k=1}^{\infty} \int_0^{t} \theta^{(0)}(\tau) f(\tau) \sin 2k\xi \sin 2k(t-\tau) d\xi d\tau}{\int_0^{\pi} yf(y,t,v^{(0)}) dy}$$

applying the Cauchy and Bessel inequalities, along with the Lipschitz condition, we obtain: $\left\|\theta^{(1)}(t)\right\| \leq \frac{2}{\pi^2 M_0} \left(E''(t) + \pi \left\|\varphi_{ck}'\right\| + \pi \left\|\psi_{sk}\right\|\right) + \frac{4T\sqrt{T}}{\pi M_0} \left(\left\|\theta^{(0)}(t)\right\| \left\|b(y,t)\right\| \left\|v^{(0)}(t)\right\| + \left\|\theta^{(0)}(t)\right\| M\right).$

The same estimations apply for step N, we obtain:

$$\begin{aligned} \left\|v^{(N+1)}(t)\right\| &= \max_{0 \le t \le T} \frac{\left|v_{0}^{(N)}(t)\right|}{2} + \sum_{k=1}^{\infty} \left[\max_{0 \le t \le T} \left|v_{ck}^{(N)}(t)\right| + \max_{0 \le t \le T} \left|v_{sk}^{(N)}(t)\right|\right] \right] \\ &\leq \frac{\left\|\varphi_{0}\right\| + \left\|\psi_{0}\right\|\left\|T\right|}{2} + \sum_{k=1}^{\infty} \left\|\varphi_{ck}\right\| + \left\|\varphi_{sk}\right\| + \frac{\pi}{2\sqrt{6}} \sum_{k=1}^{\infty} \left\|\psi_{ck}\right\| + \left\|\psi_{sk}\right\| + A\left\|\theta^{(N)}(t)\right\|\left\|b(y,t)\right\|\left\|v^{(N)}(t)\right\| + A\left\|\theta^{(N)}(t)\right\|M, \\ \left\|\theta^{(N+1)}(t)\right\| &\leq \frac{2}{\pi^{2}M_{0}} \left(E''(t) + \pi\left\|\varphi_{ck}'\right\| + \pi\left\|\psi_{sk}\right\|\right) + \frac{4T\sqrt{T}}{\pi M_{0}} \left(\left\|\theta^{(N)}(t)\right\|\left\|b(y,t)\right\|\left\|v^{(N)}(t)\right\| + \left\|\theta^{(N)}(t)\right\|M\right). \end{aligned}$$

We obtain $v^{(N+1)}(t) \in B, t \in [0,T]$ because of $v^{(N)}(t) \in B, t \in [0,T]$.

By applying same methods for convergence, we get:

$$\left\| v^{(N+1)}(t) - v^{(N)}(t) \right\| \le \left(A + SM \right)^N \times \left\| \theta^{(N)}(t) \right\| \dots \left\| \theta^{(1)}(t) \right\| C \frac{1}{\sqrt{N!}} \left(\int_0^t \int_0^\pi b^2(\xi, \tau) d\xi d\tau \right)^{\frac{N}{2}}.$$

Here $S = \frac{2\sqrt{T}}{M_* - 2\sqrt{T}M}$ and $C = \left\| v^{(0)} - v^{(1)} \right\|.$

As
$$N \to \infty, \nu^{(N+1)} \to \nu^{(N)}$$
 and $\theta^{(N+1)} \to \theta^{(N)}$.

Let us show that $\lim_{N \to \infty} \nu^{(N+1)}(t) = \nu(t), \lim_{N \to \infty} \theta^{(N+1)}(t) = \theta(t).$

Let's take the difference between the exact and approximate inverse coefficients: $\theta(t) - \theta^{(N+1)}(t) = \frac{\pi \sum_{k=1}^{\infty} \int_{0}^{t} \int_{0}^{\pi} \theta^{(N+1)}(\tau) f(\xi, \tau, v^{(N+1)}) \sin 2k\xi \sin 2k(t-\tau) d\xi d\tau}{\int_{0}^{\pi} yf(y, t, v^{(N+1)}) dy} - \frac{\pi \sum_{k=1}^{\infty} \int_{0}^{t} \int_{0}^{\pi} \theta(\tau) f(\xi, \tau, v) \sin 2k\xi \sin 2k(t-\tau) d\xi d\tau}{\int_{0}^{\pi} yf(y, t, v) dy}.$

Then add and subtract $\int_0^t \int_0^{\pi} \theta(\tau) f(\xi, \tau, \nu^{(N+1)}) d\xi d\tau$, and applying consecutively Cauchy, Bessel inequalities and Lipschitz condition, we have

$$\left\|\theta(t) - \theta^{(N+1)}(t)\right\| \le S \|\theta(t)\| \|b(y,t)\| \|v(t) - v^{(N+1)}(t)\|.$$

By applying the same methods for $v(t) - v^{(N+1)}(t)$, we find

$$\|\nu(t) - \nu^{(N+1)}(t)\| \le A (A + SM)^N \|\theta^{(N)}(t)\| \|\theta^{(N)}(t)\| \dots \|\theta^{(1)}(t)\| C \times \frac{1}{\sqrt{N!}} \Big(\int_0^t \int_0^{\pi} b^2(\xi, \tau) d\xi d\tau \Big)^{\frac{N}{2}} \exp(2A + SM) \|\theta(t)\| \|b(y,t)\|.$$

As $N \to \infty$, $\nu^{(N+1)}(t) \to \nu(t)$ and $\theta^{(N+1)}(t) \to \theta(t).$

Let consider we have two solutions (ν, θ) and (ω, ρ) of (1)-(4). We obtain the following, applying the same methods:

$$\|\theta(t) - \rho(t)\| \le S \|\theta(t)\| \|b(y,t)\| \|v(t) - \omega(t)\|.$$
(10)

$$\|v(t) - \omega(t)\| \le A \|\theta(t)\| \|b(y,t)\| \|v(t) - \omega(t)\| + A \|\theta(t) - \rho(t)\| M.$$
(11)

By using (10) in (11), we get $\|v(t) - \omega(t)\| \le (A + SM) \|\theta(t)\| \|b(y, t)\| \|v(t) - \omega(t)\|$.

Finally, applying Gronwall inequality to the last inequality, we have:

$$\|\nu(t) - \omega(t)\| \le 0 \times \exp(A + SM) \left(\int_0^t \int_0^\pi \theta^2(\tau) b^2(\xi, \tau) d\xi d\tau \right)^{\frac{1}{2}}.$$

Then $v(t) = \omega(t)$, therefore $\theta(t) = \rho(t)$.

The proof is completed.

4. STABILITY OF THE SOLUTION (v, θ)

Theorem 2. If the assumptions (C1)–(C3) hold, then the solution pair (ν, θ) of the problem (1)-(4) depends continuously on the input data ϕ, ψ and *E*.

Proof. Let sets $\|\Phi\| = \{\varphi, \psi, E\}$ and $\|\bar{\Phi}\| \le \{\bar{\varphi}, \bar{\psi}, \bar{E}\}$ denote two data groups that conform to the assumptions (C1)–(C3). Assume there are positive constants M_i that ensure the following inequalities are satisfied:

$$\left\|\boldsymbol{\varphi}\right\| \leq \boldsymbol{M}_1, \left\|\boldsymbol{\psi}\right\| \leq \boldsymbol{M}_2, \left\|\boldsymbol{E}\right\| \leq \boldsymbol{M}_3$$

Let us denote

$$\begin{split} \|\Phi\| &\leq \|\varphi\| + \|\psi\| + \|E\|, \quad \|\overline{\Phi}\| \leq \|\overline{\varphi}\| + \|\overline{\psi}\| + \|\overline{E}\|. \\ \text{Let } (\nu, \theta) \text{ and } (\bar{\nu}, \bar{\theta}) \text{ be the solutions of the problem (1)-(4) according to the data } \Phi \text{ and } \bar{\Phi}. \text{ By applying} \\ \|\theta(t) - \overline{\theta}(t)\| &\leq D \Big(\Big(E''(t) - \overline{E}''(t) \Big) + \pi \sum_{k=1}^{\infty} \|\varphi_{ck}' - \overline{\varphi}_{ck}'\| \Big) B \Big(\sum_{k=1}^{\infty} \|\psi_{sk} - \overline{\psi}_{sk}\| + 2\sqrt{T} \|\theta(t)\| \|b(y,t)\| \|\nu - \overline{\nu}\| \Big), \\ \|\nu - \overline{\nu}\|^2 &\leq 2 \|\Phi - \overline{\Phi}\|^2 \times \exp 2L^2 \Big(\int_0^t \int_0^{\pi} b^2(\xi, \tau) d\xi d\tau \Big). \\ \text{Here } D &= \frac{2}{M_0 \pi^2 - 4M_0 M \sqrt{T}}. \end{split}$$

For $\Phi \to \overline{\Phi}$ then $\nu \to \overline{\nu}$. Hence $\theta \to \overline{\theta}$.

5. CONCLUSION

An analytical investigation of a one-dimensional inverse coefficient nonlinear hyperbolic equation with periodic boundary conditions is conducted in this study. The analytical solution is obtained using the generalized Fourier method. Furthermore, an iterative approach is implemented to demonstrate convergence and to examine the existence, uniqueness, and stability of the solution for the nonlinear problem.

6. ACKNOWLEDGMENTS

We would like to thank the reviewer.

REFERENCES

- Tekin, I. (2018). Existence and uniqueness of an inverse problem for a second order hyperbolic equation. Universal Journal of Mathematics and Applications, 1(3), 178-185. https://doi.org/10.32323/ujma.439662
- 2. Hill, G.W. (1886). On the part of the motion of the lunar perigee which is a function of the mean motions of the sun and moon. Acta Mathematica, 8, 1–36.
- Asanova, A., Dzhumabaev, D. (2004). Periodic solutions of systems of hyperbolic equations bounded on a plane. Ukrainian Mathematical Journal, 56(4), 682-694. https://doi.org/10.1007/s11253-005-0103-0
- Huntul, M., Abbas, M., Băleanu, D. (2021). An inverse problem of reconstructing the timedependent coefficient in a one-dimensional hyperbolic equation. Advances in Difference Equations, 2021(1). https://doi.org/10.1186/s13662-021-03608-1
- Denisov, A.M., Shirkova, E.Y. (2013). Inverse problem for a quasilinear hyperbolic equation with a nonlocal boundary condition containing a delay argument. Differ. Equations, 49, 1053– 1061. doi: 10.1134/S0012266113090012
- Mehraliyev, Y., Huntul, M., Ramazanova, A., Tamsir, M., & Emadifar, H. (2022). An inverse boundary value problem for transverse vibrations of a bar. Boundary Value Problems, 2022(1). https://doi.org/10.1186/s13661-022-01679-x
- 7. Kanca, F., Bağlan, İ. (2018). Inverse problem for Euler-Bernoulli equation with periodic boundary condition. Filomat, 32(16).

- 8. Bağlan, İ. (2019). Analysis of two-dimensional non-linear burgers'equations. TWMS Journal of Applied and Engineering Mathematics, 9(1), 38-48.
- 9. Baglan, I. (2015). Determination of a coefficient in a quasilinear parabolic equation with periodic boundary condition. Inverse Prob. Sci. Eng., 23, 884–900. doi: 10.1080/17415977.2014.947479

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

2024 Vol. 7 No. 2 (8-28)

Ethnobotany and Bioactive Constituents of *Tabernaemontana pachysiphon* Stapf (Apocynaceae) Used in Indigenous Medicine Practices in Abia State, Nigeria

Research Article

David N. Enyiukwu^{1*}, Anderson C. Amadioha¹⁰, Inemesit N. Bassey²

¹Department of Plant Health Management, Michael Okpara University of Agriculture, Umudike, KM 10 Umuahia-Ikot Ekpene Road, PMB 7267 Umuahia, Abia State, Nigeria. Tel.: +234(0)09092309790

²Department of Botany and Ecological Studies, University of Uyo, PMB 1017 Uyo, Akwa-Ibom State, Nigeria

Author E-mails: enyidave2003@gmail.com amadioha4u@yahoo.com inemesitnbassey@uniuyo.edu.ng David N. Enyiukwu ORDID ID: 0000-0003-0595-0630 Anderson C. Amadioha ORDID ID: 0009-0003-5050-8697 Inemesit N. Bassey ORDID ID: 0009-0005-2677-2062

*Correspondence to: David N. Enyiukwu, Department of Plant Health Management, Michael Okpara University of Agriculture, Umudike, KM 10 Umuahia-Ikot Ekpene Road, PMB 7267 Umuahia, Abia State,

DOI: 10.38061/idunas.1500241

Received: 12.06.2024; Accepted: 06.11.2024

Abstract

This work surveyed the ethno-botanical relevance of *Tabernaemontana pachysiphon* in the indigenous medicinal practices of the people of Ekebedi ancient Kingdom, Abia State, Nigeria. In rural tropical localities of Africa such as the Kingdom, modern western Medicare is scarcely available, and plants have provided substantial part of the medicines used by the natives over the years to treat both metabolic and pathological diseases as well as fractures amongst many other health conditions. Questionnaires and oral interviews were utilized to capture relevant data about the plant from 102 indigenous herbal doctors, traditional medicine vendors and natives of the Kingdom and its environs. Phytochemical analysis of the test extract was conducted using GC-MS fingerprinting. The results obtained from the survey indicated that preparations from seeds, leaves, stems and root barks of the plant are used by traditional medicine men and natives of the Kingdom to treat fevers, pains, diabetes, hypertension, stomach troubles, ringworms, malaria, gonorrhea, syphilis, venereal diseases, tumors and bouts of poisoning etc.

GC-MS profiling identified 32 bioactive principles in the methanol leaf extract of the plant, with 9, 12octadecadienoic acid (Z, Z)-, methyl ester (16.35%); octadecenoic acid, methyl ester, (E)-, (19.17%); dodecanoic acid 1, 2, 3-propanetrieyl ester (29.69%) being the major ingredients. Amongst many others were hexadecenoic acid, methyl ester (2.91%); cyclopentadecanone, oxime (1.01%); Z,Z-8,10-hexadecadien-1-ol (1.82); 9-octadecenoic acid, 1,2,3-propanetriyl ester, E (5.86%); 9-octadecenoic acid, 1,2,3-propanetriyl ester, (E- (1.72%); d-mannitol, 1-O-(22-hydroxydocosyl)- (6.00); and dodecanoic acid, 1,2,3-propanetriyl ester (6.73%). These principles may underpin the efficacy of *T. pachysiphon* in folk medicine and possibly explain the reasons for the medicinal uses of this plant by the natives of the Kingdom.

Keywords: Ethnobotanical relevance, Folk-medicine, Tabernaemontana pachysiphon, Bioactive constituents

1. INTRODUCTION

Tabernaemontana pachysiphon Stapf (Apocynaceae) (Synonyms: *Conopharyngia pachysiphon, T. angolensis* Stapf, *T. holstii* Schum.) is commonly called *Osi* in Eastern Nigeria (Personal communication, 2020). The genus is native to Africa and is distributed throughout tropical regions of the world [1-2]. *T. pachysiphon* is a small tree that grows up to 15 m in height [2]. The leaves of the plant are leathery, glossy, simple and elliptical in shape. It blooms twice yearly, producing sweet-scented, cup-shaped flowers that are predominantly white or pale yellow in colour [3].

Several members of this genus play roles as shade plants, windbreakers, and ornamentals in gardens and landscapes [1] and are good sources of timber [3]. Plants in this tropical genus serve diverse medicinal purposes for natives in traditional medicine practices across Asia, Americas and Africa [4-6]. Decoctions or infusions of leaves, roots, and stem bark of *T. pachysiphon* and its related species (4g/200 mL; 5 leaves/250 mL) in traditional medicine are effectively used to treat insanity, scabies, hypertension, pains, headache, stomach-ache, constipation, syphilis, ulcers and tumors [1,5,7]. Furthermore, preparations from the plants are used in diverse cultures of the tropics to treat dementia, fevers, and as styptic to control bleeding and facilitate wound healing [6,8]. They have also been used as anti-nociceptive agents to relieve breast inflammation in local settings of Nigeria [9].

Scientifically, the genus *Tabernaemontana* has demonstrated activities such as vasorelaxation, analgesic, anthelminthic, antitumor, antimicrobial and antiviral effects [5,10]. Methanol and hexane leaf and stem bark extracts of *Tabernaeontana* sp. effectively inhibited methicillin–resistant *Staphylococcus aureus, Escherichia coli, Pseudomonas aeruginosa, Bacillus subtilis and Candida* sp. [6]. Retardation of growth and development of *Entamoeba histolytica, Cryptococcus neoformans* and *Penicilium notatum* by extracts of *T. alternifolia* L., *T. stapfiana* Britten and *T. pachysiphon* have also been documented [1,7,9]. Pallant [13] reported that fractions and isolates from *T. pachysiphon* demonstrated strong antibacterial activities against stubborn clinical strains of *S. aureus* and *Mycobacterium tuberculosis,* causative agent of tuberculosis in humans.

Phytochemical analysis of extracts from different parts of *Tabernaemontana* species has revealed the presence of bioactive alkaloids, including ibogine, voacristine, cononaridine, and conolidine [1,7,9–12]. Additional alkaloids such as tubotaiwine, ovoacangine, ibogaline, 3R-hydroxyconopharyngine, 3S-hydroxyconopharyngine, and 11-demethylconoduramine have been isolated from the stem bark and leaves of *T. pachysiphon* in Kenya and Nigeria [13–14]. Flavonoids, polyphenols, and tannins have also been identified in *T. pachysiphon* and related species [10,15–18]. Fatty acids, including n-hexadecenoic acid, octadecanoic acid, tetracosanol-1, n-nonadecanol-1, α -linolenic acid, and pentadecanoic acid, have been detected in organic root and stem bark extracts of *Tabernaemontana* species [6,19–22].

Statistics show that 80% of the rural populace worldwide still depend on using medicinal plants to assuage disease conditions [23-24]. Owing to the challenge posed by the growing number of antimicrobial resistant strains of medically important pathogens which cause about 5 million deaths per annum around the world [25], scientists are turning their focus to secondary metabolites from higher plants with a view to finding

alternative or complementary carbon skeleton leads for drug candidates [26]. These bioactive phytoingredients act in synergy and could bind on multiple target sites or elements of complex cellular pathways of disease-causing agents, and thus are less likely for pathogens to develop resistance to them than modern allopathic medicines [6,26]. All these give impetuses to elicit, and to document so as to preserve indigenous knowledge of medicinal plants [26].

Although *T. pachysiphon* is widely used as a medicinal plant by the natives of Ikwuano Local Government Area in Abia State, its ethnobotanical relevance has not yet been systematically documented in this region. Furthermore, while numerous alkaloids have been isolated from the plant, the bioactive fatty acid constituents in its leaves are, to the best of our knowledge, scantily documented or entirely uncharacterized.

Therefore, the ethnobotany and bioactive chemical constituents of *T. pachysiphon* used in the indigenous medicine practices of Ekebedi ancient Kingdom of Ikwuano Local Government Area, Abia State, Nigeria are presented in this work.

2. MATERIALS AND METHODS

2.1 Survey location

Ekebedi Oboro ancient Kingdom is located in Ikwuano Local Government Area (LGA) of Abia State, in the rainforest belt of Southeast Nigeria. The Kingdom lies on latitude 5° 24' 22"N, longitude 7° 34' 5"E with an elevation of 112 meters above sea level (Fig. 1). Estimates show that about 200 households make up the ancient Kingdom.

A modern community health center is conspicuously absent in the Kingdom, though there are pockets of patent medicine stores dotting its landscape. The majority of the people in the Kingdom practice mixed farming characterized by land rotation, fishing, traditional medicinal practices, lumbering, trading, hunting as well as bone-setting.

The warm and humid rainforest zone of the tropics is endemic to malaria and several other kinds of parasitological diseases as well as metabolic dysfunctions including diabetes. A large number of tropical natives usually administer herbal recipes against attacks of these diseases [6,23,27]. This work seeks hence to validate the use of *Tabernaemontana pachysiphon* by people of Ekebedi Oboro Ancient Kingdom in Abia State, Nigeria, in the treatment of some pathological and metabolic diseases.

2.2 Research design and collection of ethno-botanical data

A good research follows a systematic design and scientific protocol. In this study, a survey of the ethnobotanical relevance of *T. pachysiphon* by people of the ancient Ekebedi Kingdom was undertaken. A total population 102 indigenous people made up of 14 herbal practitioners and 26 indigenous herb vendors and 62 native herbal medicine users from the Kingdom and its environs were randomly selected for the study. The survey employed a house-to-house strategy which lasted over a period of 6 weeks (September 01, 2023 – October 07, 2023). Data collection involved interviews and structured questionnaires, as adopted by Mgbeahuruike [23,27]. The survey questions were tailored in such a way as to elicit responses from the target respondents on whether or not they use *T. pachysiphon* in traditional medicine, and if yes, why, when, and how do they use the plant as medicine in the traditional medicine practices in the ancient Kingdom.

2.3 Source of plant material

Tabernaemontana pachysiphon leaves were obtained from a courtyard in the Ancient Kingdom of Ekebedi Oboro. Fresh aerial parts of the plant were collected and authenticated by Prof. M. C. Dike from the

Department of Forestry, College of Natural Resources and Environmental Management (CNREM), Michael Okpara University of Agriculture, Umudike.



Figure 1. Map of the study area. Left: Map of Abia State, Nigeria showing the 17 Local Government Areas (L.G.A.) of the State. Center: Map of Ekebedi Kingdom in Ikwuano L.G.A. of Abia State, Nigeria. Right: Satellite view of the study location terrain

2.4 Methanol extraction of *T. pachysiphon* leaves

The leaves of the plant were washed with tap water, and dried on the laboratory bench for 21 days, enveloped and oven-dried at 40°C for 30 minutes, and then milled into powder using a Thomas Wiley machine (Model: ED-5 USA). Then 500 g of the milled powder was packed separately into a 2-liter Soxhlet apparatus and extracted exhaustively with 1000 ml of analytical-grade methanol for 24 hours. The methanol leaf extract was concentrated using a rotary evaporator at 45°C and left on the laboratory bench for two days to afford residue [28].

2.5 Gas chromatography-mass spectrometry (GC-MS) analysis of the plant residues

This was conducted according to the standard procedure as adopted by Gopinath [29] and Omotosho 30]. The residue *T. pachysiphon* was re-dissolved in methanol. One (1) ml aliquot of the solution was injected into the GC-MS equipment [Model: QP 2010 Plus Schmadzu, Tokyo, Japan) (AOC-20i auto-sampler, VF 5 MS fixed silica capillary column] The carrier gas was helium (99.99%), flow rate (1.58 ml/min), injector and mass transfer line temperature (250 and 200°C respectively), injection volume of 1 μ l was employed at a split ratio of 10:1. The MS ionization energy (70 eV), ion source temperature (200°C), solvent cut time (2.5 min), and the interface temperature was 250°C. The total running time of the machine was set to 1 hour.

The percentage of the extract was expressed as percentage with peak and normalization. The relative percentage amount of each phyto-component was calculated by comparing its average peak area to the total areas. The detection process utilized the National Institute of Standard and Technology (NIST) (Version 2.0, 2005) library. The compound prediction was based on the Phytochemical and Ethno-botanical Databases of USDA [31]. The interpretation of GC-MS was conducted using the NIST database having more than 62,000 structural patterns. The spectra of the unknown phyto-components were compared with those of known components stored in NIST library. The names, molecular weights and chemical structures of the extracts-derived compounds were ascertained from the National Institute of Standard and Technology (NIST) [32].

2.6 Data analysis

Data generated from this study was analyzed by simple descriptive statistical tools such as range, means and percentages using SPSS computer software version 25.

3. RESULTS AND DISCUSSION

3.1 Results

3.1.1 Demographic characteristics of respondents in the survey

The results of the study presented in Fig. 2 indicate that in the ancient Kingdom both genders (male and female) of the natives were involved in traditional medicine practices and usage. However, women had a higher involvement, representing 62.01% of the respondents engaged in preparation, vending, and utilization of traditional medicines, compared to their male counterparts, who accounted for only 39.99% (Fig. 2A).

In terms of age, majority of the practitioners of indigenous traditional medicine in the Kingdom clustered around the 21–40-year-old age bracket (51.19%). This was closely followed by those between the ages of 41-70 years (27.33%); while practitioners above 71 years old represented only 3.44% (Fig. 2B).

Regarding education, a significant portion of the respondents had no Western education (42.56%) or had received limited Western education (39.82%). Conversely, those with post-secondary or tertiary education exposure accounted for just 1.67% of the respondents (Fig. 2C).

In terms of occupation, farmers (49.69%) and traders/artisans (35.00%) topped the chart of respondents using and vending *T. pachysiphon*-based herbal recipes whereas others who are engaged in elitist occupations were the least (Fig. 2D). Similarly, 47.50% of the herbal medicine users and dealers were of Christian faith, 31.25% others practice typical traditional African religion while 3.08% who were mostly of Yoruba extraction were Muslim faithful (Fig. 2E).

The majority of the indigenous dwellers of the Kingdom are Igbo and they accounted for 76.19% of the people involved in the traditional medicine system of the Kingdom. Next to them are practitioners from the neighbouring Ibibio tribe, which constituted 13.63% of the herbal medicine practitioners and users. The migrants from the Yoruba ethnic group of western Nigeria, however accounted for only 10.18% (Fig. 2F). These plant parts are prepared as decoction or infused as teas or soaked in local gins (macerations/elixirs). In the case of poisons, the leaf juice is mixed with honey and given to the patient bitten by snakes, centipedes or scorpions to induce the patients to nausea, and vomiting the poisonous material (Fig. 3).





Figure 2. Demographic characteristics of respondents on medicinal significance of the test plant (a = gender, b = age, c = education, d = occupation, e = faith, g = ethnicity)



Figure 3. Methods of preparation of *T. pachysiphon* crude drug recipes

3.1.2 Medicinal relevance of *T. pachysiphon* in the traditional medicine system of Ekebedi ancient Kingdom

The ethnobotanical uses of *T. pachysiphon* in the study area are presented in Table 1. The results of the survey showed that the plant has varied ethno-botanical significance in the area. All respondents in the study agreed that the plant is used for treating cases of diabetes, stomach aches, menstrual pains, ringworms and venereal diseases.

About 80% of the respondents use different preparations of leaves of the plant for treating hypertension and gonorrhea, ringworms and poison, 60% of the respondents prescribe or use the plant for diverse purposes, while 70% others employ the root bark of the plant in treating cases of miscarriages, pelvic inflammatory diseases and venereal diseases (Table 1).

Table 1. Survey on medicinal uses and relevance of *T. pachysihon* in ethnobotany of Ekebedi Community and its surrounding villages in Ikwuano LGA, Abia State

Common/ Local Name of The Plant	Number of Medicine Men Interviewed	Respondents Interviewed (%)	Parts Used for Treatment	Traditional Medicinal Uses and Relevance
Giant pin wheel flower	102	80	Leaf	Malaria fever, typhoid fever, diabetes, poison, dysmenorrhea, hypertension, gonorrhea, ringworms, lacerations, bruises, wounds, dementia, sores, tumors
<i>Osi</i> in Igbo	102	50	Stem bark	Fevers, diabetes, stomachache, malaria, ulcers, syphilis, abdominal troubles, gonorrhea, dementia
	102	102 70 R		Malaria fever, miscarriage, pelvic inflammatory disease (PID), waist pains, venereal diseases, ulcers, sores
	102	60	Seed	Diabetes, menstrual pain, venereal diseases, ringworms
<i>Osi</i> in Igbo	102	100	All parts	Diabetes, stomachache, fevers, venereal diseases, hypertension, syphilis, gonorrhea, scabies, dementia, tumors, skin diseases, dysmenorrhea

3.1.3 Mass Spectral data on T. pachysiphon

The mass spectral chromatogram of the GC-MS analysis of the volatile components of 2μ l of the methanol leaf extract of *T. pachysiphon* is presented in Fig. 4. The data indicated the presence of a wide array of bioactive volatile principles in the plant leaf, and a total of 32 compounds were identified as shown by the spectral peaks (Table 2).

The relative abundance of the compounds ranged from as low as 0.01% recorded for 1-Tridecyn-4-ol to 29.69% obtained for dodecanoic acid 1, 2, 3-propanetrieyl ester. The peaks representing major volatile compounds in the chromatogram include peak 7 which had 9, 12-octadecadienoic acid (Z, Z) methyl ester (16.35%); and peak 8 which had octadecenoic acid methyl ester (E) (19.17%) while peak 30 recorded dodecanoic acid 1, 2, 3-propanetrieyl ester (29.69%). Other compounds identified in sizeable quantities were hexadecanoic acid methyl ester (2.91%) at peak 5, cyclopentadecanone oxime (1.01%) at peak 14 and

Z,Z-8,10-hexadecadien-1-ol (1.82%) at peak 20. Also, peaks 21 and 26 had 9-Octadecenoic acid, 1,2,3propanetriyl ester E (5.86%) and 9-octadecenoic acid, 1,2,3-propanetriyl ester (E- (1.72%) respectively while d-mannitol 1-o-(22-hydroxydocosyl) (6.00%) and dodecanoic acid, 1,2,3-propanetriyl ester (6.73%) were recorded at Peaks 31 and 32 respectively. Some of the bioactive constituents which were detected in trace quantities (area %) in the methanol extract of *T. pachysiphon* as shown in Table 2 include: cyclononasiloxane, lauroyl peroxide, dichloroacetic acid, cyclopentadecanone oxime, undecanal, 1octadecyne, hexadecanoic acid 2-methyl ester etc. The structures of some of the bioactive chemical compounds are shown in Fig. 5.



Figure 4. Chromatogram of volatile compounds of T. pachysiphon leaf

Table 2: Bioactive compounds in *T. pachysiphon* methanol leaf extract

Peak Num.	Retention Time	Area	Peak Area (%)	Height	Height %	A/H	Name of Compound (Constituent)
1	26.584	71163	0.09	16422	0.12	4.33	Phthalic acid, ethyl pentyl ester
2	30.595	31132	0.04	11914	0.09	2.61	Propane
3	30.851	37194	0.05	18011	0.13	2.07	Pentanoic acid, 2-methyl-
4	34.253	134521	0.17	47832	0.35	2.81	2- (2',4',4',6',6',8', 8'- Heptamethyltet rasiloxan-2'-y
5	36.754	2314114	2.91	737134	5.38	3.14	Hexadecenoic acid, methyl ester

$ \begin{array}{c c c c c c c c c c c c c c c c c c c $							T	1
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	6	38.592	253355	0.32	74813	0.55	3.39	2- (2',4',4',6',6',8', 8'- Heptamethyltet rasiloxan-2'-y
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	7	41.288	1300072 2	16.35	3676453	26.82	3.54	9,12- Octadecadieno ic acid (Z,Z)-, methyl ester
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	8	41.469	1523591 1	19.17	4232968	30.89	3.60	9- Octadecenoic acid, methyl ester, (E)-
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	9	41.592	406786	0.51	99316	0.72	4.10	6- Octadecenoic acid, methyl ester, (Z)-
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	10	42.130	1000514	1.26	302490	2.21	3.31	Methyl stearate
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	11	42.584	460671	0.58	108772	0.79	4.24	Methoprene
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	12	43.624	67666	0.09	17571	0.13	3.85	1-Octadecyne
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	13	44.150	8084	0.01	3380	0.02	2.39	1-Tridecyn-4-ol
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	14	46.217	799068	1.01	150764	1.10	5.30	Cyclopentadeca none, oxime
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	15	46.428	159768	0.20	33670	0.25	4.75	14- Heptadecenal
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	16	47.054	99756	0.13	31285	0.23	3.19	Methyl 2- hydroxydodecan oate
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	17	49.164	164729	0.21	44614	0.33	3.69	1-Octadecyne
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	18	49.292	207023	0.26	58195	0.42	3.56	1-Octadecyne
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	19	49.572	614100	0.77	169557	1.24	3.62	Cyclononasiloxa ne, octadecamethyl-
$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	20	50.247	1448690	1.82	334895	2.44	4.33	Z,Z-8,10- Hexadecadien- 1-ol
22 51.299 50300 0.06 38106 0.28 1.32 Dichloroacetic acid, 4-tridecyl ester 23 51.611 345252 0.43 64002 0.47 5.39 Lauroyl peroxide	21	50.367	4659702	5.86	569101	4.15	8.11	9-Octadecenoic acid, 1,2,3- propanetriyl ester, (E
23 51.611 345252 0.43 64002 0.47 5.39 Lauroyl peroxide	22	51.299	50300	0.06	38106	0.28	1.32	Dichloroacetic acid, 4-tridecyl ester
	23	51.611	345252	0.43	64002	0.47	5.39	Lauroyl peroxide

24	52.724	777478	0.98	217614	1.59	3.57	Cyclononasilo xane, octadecamethy 1-
25	54.885	647612	0.81	160491	1.17	4.04	Z,Z-8,10- Hexadecadien- 1-ol
26	54.972	1422263	1.79	285774	2.09	4.98	9- Octadecenoic acid, 1,2,3- propanetriyl ester, (E
27	55.143	47764	0.06	23573	0.17	2.03	1-Octadecyne
28	55.475	179672	0.23	22197	0.16	8.09	Undecanal
29	55.687	1128589	1.42	266811	1.95	4.23	Cyclononasilo xane, octadecamethy 1-
30	57.389	2359984 5	29.69	1230741	8.98	19.14	Dodecanoic acid, 1,2,3- propanetriyl ester
31	58.462	4766783	6.00	392591	2.86	12.14	d-Mannitol, 1- O-(22- hydroxydocosy l)-
32	61.579	5352187	6.73	264508	1.93	20.23	Dodecanoic acid, 1,2,3- propanetriyl ester
		7949241 4	100.00	13705565	100.00		

 Table 2: Bioactive compounds in T. pachysiphon methanol leaf extract (continue)

*Peak area = % composition of compound in the leaf extract; Num. = Number; A/H = Area/Height

3.2 Discussion

Results presented in Fig. 2 show the demographic characteristics of the population surveyed in this study. The findings indicate that more women than men use *T. pachysiphon*-based herbal medicines. This is in strong agreement with observations of Onyipat et al. [34] in Enugu, southeast Nigeria, but inconsistent with the views of Aina et al. [35] where men dominated the usage of herbal medicine in Ibadan, western Nigeria. Data from this study indicated that middle-aged people were largely involved with preparations and usage of *T. pachysiphon* as medicine in the study area. This is, however, not consistent with the reports of Aina et al. [35] where usage of herbal medicines was common amongst the elderly who are above 70 years old. On the other hand, findings from this study where respondents with religious background in orthodox Christian and traditional faith systems used more of herbal medicine is also in accord with the views expressed in previous studies [35].
This study also showed that the usage of traditional medicine is higher amongst natives with little or no formal education, a view sustained by submissions of earlier findings [35,36]. Ethnicity, education, employment and family size and financial status have been reported as some of the principal predictors of usage of traditional medicines amongst aborigines [37]. Findings in this survey indicated that Yoruba migrants who dwell in the Kingdom were less likely to use *T. pachysiphon* recipes compared to their Igbo and Ibibio counterparts who make up the majority of inhabitants of the Kingdom. This observation is in strong agreement with previous reports [35].

Moreover, findings from this study indicated that respondents with less than secondary education were more likely to use herbal medicines to assuage disease conditions, this view aligns with observations made by previous workers [35-37]. Some workers were of the view that usage of traditional medicine was common amongst traders and artisans [36]. This aligns with findings from the current study, where **farmers** and **traders** were the dominant groups engaged in the preparation and use of *T. pachysiphon*-based recipes for treating medical conditions.

Additionally, earlier studies have reported that **affordability**, **accessibility**, and **efficacy** are the primary drivers behind the widespread use of herbal medicines in local environments. Given the absence of modern private or public healthcare centers and clinics in the Kingdom, these factors likely explain the high utilization of traditional medicines, including *T. pachysiphon* recipes, among the surveyed population.

The results presented in Table 2 indicate that the traditional medicine men of the study area use all parts (leaves, stem and bark and seeds) of *T. pachysiphon* as medicine against wounds, metabolic and pathological diseases. Amongst the many diseases for which the plant finds medicinal use include dementia, tumors, malaria, dysmenorrhea, fevers, diabetes, stomach troubles, syphilis and gonorrhea, and other venereal diseases as well as scabies amongst other skin diseases.





CompName:9,12-Octadecadienoic acid (Z,Z)-, methyl ester \$\$ Linoleic acid, methyl ester \$\$ Methyl cis



Figure 5. Structures of some bioactive chemical compounds from *Tabernaemontana pachysiphon* leaf. a. Phthalic acid, b. 2-(2',4',4',6',6',8',8'-Heptamethyltetrasiloxan-2'-y; c. 2-(2',4',4',6',6',8',8'-Heptamethyltetrasiloxan-2'-y, d. 9, 12-Octadecedienoic acid ME, e. 9-Octadecanoic acid ME, (E)-, f. Cyclononasiloxane, g. 9-Octadecenoic acid, 1,2,3-propyltryl, h. 6-Octadecenoic acid, i. Methaprene, j. Hexadecanoic acid ME, k. Cyclopentadecanone, l. lauryl peroxide, m. Dichloroacetic acid, n. Dodecanoic acid, o. d-mannitol.

In the case of fevers, the plant leaves alone or in conjunction with other parts of the plant are decocted and used in the early hours of the morning to steam the body of the patient, followed by a bath, and a cup (100 ml) of the decoction taken twice daily. Latex from the leaves and stem bark is used as a styptic, and in some cases, a poultice prepared from the leaves, twigs, and stem bark is applied to palliate wounds, lacerations, sores, and to promote the healing of ulcers. The plant is also prepared as tea or hot infusions to alleviate dementia, insanity, and other mental health conditions.

Other respondents agreed to prescribing the plant as a gin-based recipe (elixirs, macerations) of the stem or root bark for matters suspected to be venereal diseases such as herpes, gonorrhea and syphilis, enteric fever,

stomach aches, and dysmenorrhea. This agrees with the reports of Omino and Kowaro [3] and Elia [1] that tropical natives use preparations of members of this genus to treat venereal, abdominal and skin diseases amongst several other ailments.

In conditions relating to diabetes for example, data obtained indicate that the seeds are ground and mixed with coconut (*Cocos nucifera*) water and consumed about 50 ml thrice daily. While leaf juice is applied to itching skin, decoction or infusion of the leaves is drunk to palliate ringworm, scabies and other irritation of the skin. These findings are also consistent with the submissions of Elia [1], who reported the use of *T. pachysiphon* in the treatment of scabies.

For issues suspected to be toxicity from ingesting contaminated substance or poison as a result of snake bites, centipede or scorpion stings on the other hand are treated by mixing leaf juice of the plant with honey and given to the patient to neutralize and induce vomiting of the poisonous substances or their mucilaginous derivatives. This is congruent with the views of Vineetha et al. [33] who noted that in Brazil, Singapore and elsewhere, roots, leaves and flowers *T. alternifolia*, *T. divaricarta* (L.) R.Br. ex Roem. Schult. and *T. catharinensis* (A. DC) are used by native Latin Americans and Asians for the treatment of snake bites such as the Indian cobra, *Echis carinatus* and scorpion stings.

Members of *Apocynaceae* remain one of the primary sources of medicines used in traditional healthcare systems of the tropics to treat numerous diseases including infections, tumors and cancers [24]. Data from this survey showed that all parts of the plant are decocted or macerated in local gin (*kai kai*) and taken to ameliorate hyperglycemia, and to deflate tumors or for arresting development and spread of carcinomas; a view which is in strong accord with the reports of several other previous researchers [4,38-39].

Additionally, alcoholic extracts of leaves and/or root and stem bark of the plant is also used by the native as analgesic against joint aches, and body pains. Edinoff [10] found alkaloidal isolates from *Tabernaemontana* sp. as a chronic pain killing compound. This lends support to the use of *T. pachysiphon* preparations by natives of the study Kingdom as analgesic as seen in this study.

The findings from this study are also consistent with Boligon [40] who reported that *Tabernaemontana* species play important role in complementary medicine of tropical countries, by minimizing oxidative stress-induced diseases, inflammations and tissues damages. Toxicity evaluations revealed that aqueous root bark extract of *T. pachysiphon* (5g/kg) induced neither death nor observable toxicity in mammals [41]. Similarly, fluorescent organoleptic and elemental analysis of the leaves, stem and latex of its close relative, *T. ventricosa* Hochst. ex. A.DC., vindicated the genus of noxious compounds [6].

Data from this survey indicate also that traditional medicine men and natives of the Kingdom use water, local gins and honey to prepare their *T. pachysiphon* medicines. This is consistent in part with the view expressed by some workers about solvents for making traditional medicines as reported by Abduralman and Kolawale [42] and Mgbeahuruike [27]. However, the use of honey as a medium to prepare herbal recipes as noted in this study is divergent from their submissions.

Several bioactive fatty acids and volatile compounds have been identified and characterized from aqueous and organic extracts of many genii of higher plants including *Tabernaemontana* species [43-46]. In this study, different bioactive chemical compounds were identified as shown in the chromatogram of *T. pachysiphon* (Table 1). The predominant compounds were dodecanoic acid 1,2,3-propanetriyl (lauric acid) (29.68%), 9-octadecenoic acid methyl ester (oleic acid) (19.17%), and 9,12-octadecadienoic acid (Z,Z)-methyl ester (linoleic acid) (16.35%). Oleic, α -linoleic and pentadecanoic acids were also found to be major bioactive fatty acids in GC-MS analysis of hexane and methanol leaf, stem and latex extract of *T. ventricosa and T. pachisiphon* [20]. In parallel studies, different solvent extracts of the leaves and seeds oil of *Cnidoscolus aconitifolius*, *Entandrophragma angolense*, Ajuga *relicta*, and bitter apple afforded 9,12-octadecadienoic acid (Z,Z)- methyl (43.20-50.3%), oleic and palmitic acids (23.51-29.50%) amongst others as the most abundant constituents of the plant materials [46-49]. These results are in tandem with findings in this study where these volatile compounds were detected abundantly in *T. pachysiphon* leaf (Table 2).

Data from this study indicated that dodecanoic (lauric) acid was the highest occurring compound in methanol leaf extract of *T. pachysiphon* in this study (Table 2). This is however, not in accord with reports of previous workers where n-hexadecanoic acid an analogue of octadecanoic acid [44] had the highest peak areas ranging between 11.18-27.49% in methanol stem, leaf and flower extracts of *T. pachysiphon* and *T. divaricata* [19-21,43]. Differences in age, genetic constitution, parts of *Tabernaemontana* species, geographic origin, type of extracting solvent, or particle size of the plant powder used in this study may explain the disparities in type and amount of constituents afforded by the test plants.

Strong biological and antimicrobial activities have been attributed to 9, 12-Octadecadienoic acid (Z, Z) - methyl ester, 9-Octadecenoic acid methyl ester, and Dodecanoic acid 1, 2, 3-propanetriyl), the predominant ingredients afforded by *T. pachysiphon* in this study [30,50]. Linolenic acid (9, 12-octadecadienoic acid (Z, Z) - methyl ester, has been associated with hepato-protective, anti-acne, anti-inflammatory, anti-cell proliferation, and anti-cancer activities [19]. Hexadecanoic and n-hexadecanoic (palmitic) acids which are analogues of linolenic acid, exhibits antioxidant, anti-diabetic, anticancer, antimicrobial, anti-enzyme and anti-viral activities [19,44-45]. Hexadecanoic acid present in significant amounts in aqueous root and fruit extracts of *Carica papaya* L., was fingered for recovery of diabetic rat exposed to aqueous root treatment of *C. papaya* in a trial [44-45]. Natives in the present study area use *T. pachysiphon* for the treatment of diabetes, and these esters may be responsible for its medicinal efficacy against the disease.

High concentration (29.68%) of lauric acid (dodecanoic acid 1, 2, 3-propanetriyl) was recorded in methanol extract of *T. pachysiphon* in this study. Kernels of *Cocos nucifera* L. and *Elais guineense* Jacq. kernel oils are rich in dodecanoic acid 1, 2, 3-propanetriyl [51-52]. This ester compound was reported to demonstrate strong antimicrobial and antibacterial potentials against a wide array of bacteria, as well as antiviral activities against HIV-I and HIV-II, HSV-I and II, measles virus, and human stomatitis virus etc. [53]. It is also thought to inform the protective properties of breast milk in neonates. In addition, antibacterial activity against medically important bacterial pathogens including *S. aureus, S. epidermidis, Streptococcus agalactiae, E. coli, Chlamydia pneumonia, Propionibacterium acne* due to this ester have been documented [54-55]. Lauric acid (5%) exhibited potent anti-*Streptococcus* sp. and *S. aureus* activity comparable with ciproxacin. The growth and reproduction of *Candida albicans* was also retarded by this compound *in vitro* [56-57]. In crop protection, this ester effectively impeded mycelial growth of *Rhizoctonia solani* and *Pythium ultimum in vitro* and barred expression of powdery mildew (*Blumeria graminisi f. sp hordei*) of bailey plants in the field [58]. These compounds may be the major reasons underscoring the fungitoxic and antimicrobial effects of this plant against ringworms and diarrhea amongst others in ethno-medicine.

Earlier investigations revealed the presence of hexadecenoic acid, octadecane and oleate in *A. flourubunda* [59] and hexadecenoic acid methyl ester, 1,2,3-propanetriiyl ester (12.8%) from oil fraction of *Alstonia boodei* [60-61]. These compounds were also identified in varying concentrations in this study. Several of the volatile compounds identified in this study such as phthalate, hexadecenoic acid methyl esther, cyclononasiloxane octadecamethyl, dichloroacetic acid (DCA), 6-Otadecenoic acid and lauryl peroxide possess significant antifungal activities (Table 2). Antifungal activity of some of these compounds against *Aspergillus fumigatus* has been reported to be due to oxidation of carbon-hydogen bond, altering radical chemistry, nucleophilic substitution and electrophilic ring opening [62]. Similarly, a recent evaluation of the volatile constituents of the seed oil attributed its antibacterial activity against species of *Kiebsiella* and *Salmonella* to 11-octadecenoic acid methyl ester [47].

Cyclononasiloxane octadecemethyl was identified in this study on *T. pachysiphon* (Peaks 19, 24, 29) The compound was associated with anti-cancer, anitioxidant, antibacterial, and antifungal activities [31,63]. Cyclononasiloxane octadecemethyl was implicated for impeding the growth and development of *Penicillium digitatum, Fusarium solani, Aspergillus niger* and *Borytis cinerea* by oil of *Marribium vulgare* [64].

GC/MS analysis of n-hexane extracts of neem leaf, stem and root barks besides affording 11-octadecenoic and 13-octadecenoic acids, also yielded dibutyl phthalate, and oxime. These compounds amongst others demonstrated significant antifungal activity [65-66].

Similarly, lauryl (benzoyl) peroxide is an anti-acne, bleaching and oxidizing agent [67]. It showed superior antibacterial activity against Proteus species, S. aureus and Pseudomonas aureginosa over gentamicin and phenoxyethanol which are known antibacterial agents [68]. Phthalic acid (ethyl pentyl ester) recorded in this study (Table 2) is associated with strong anti-enzyme and antioxidant activities. The compound in previous crop protection studies has shown anti-enzyme activity against several enzymes including chitinases responsible for degrading the chitin exo-skeleton during pre-molt phase in insects [69]. These workers found that diethyl phthalate and dibutyl phthalate were fungitoxic to Aspergillus sp., Fusarium spp., Candia spp., Drechslera haloides, Alternaria sp. and Rhizoctonia solani [70-72]. This activity was thought to be due to infringement of the actions of the extracellular enzymes of the fungus that played important roles in its nutrition and growth [69]. Similarly, the anti-enzyme activity against pyruvate dehydrogenase kinase (PDK), anti-cancer and antifungal activities against Rhizoctonia solani and F. oxysporum of dichloroacetic acid (DCA) recorded for T. pachysiphon (Table 2) have been reported. DCA has been identified from hexane extract of marine macroalgae [73]. The mechanism of action of this compound is linked to pyruvate dehydrogenase kinase inactivation, and fostering oxidative phosphorylation in target organs [63]. Its anticancer activity is suggested to hinge on depolarizing abnormal cells in the mitochondria of glioblastoma cancer cells leading to induced apoptosis amongst others [74]. The compound also played roles reducing lactate levels in Leigh syndrome patients. Z, Z, 8, 10-hexadecenoic acid (Table 2) has been reported as an important steroid compound, having similar activity to pheromones against insect pests [75-76]. As a result, it could be useful as eco-friendly insect attractant in integrated pest management (IPM) programs. Unfortunately, most of these compounds were identified at slightly sizeable or trace quantities in T. pachysipon. Medicinal plants contain a wide spectrum of chemical compounds known to act jointly or synergistically to bring about resultant effects that usually surpass total activity of any one single constituent [6,43]. Presence of some these compounds [77] in the test plant used in this study may have contributed to medicinal actions of the test plant in the indigenous medical practices of people of the Kingdom and its environs.

4. CONCLUSION

The results of this study indicate that *T. pachysiphon* is used in the herbal medicine practices of people of Ekebedi ancient Kingdom and its environs. The plant is used in the treatment of various diseases including fevers, pains, malaria, inflammatory conditions, diabetes, tumors, dementia, syphilis, scabies, and pathogenic infections etc.

Crude medicinal recipes are prepared in various forms, such as infusions, decoctions, alcoholic macerations in local gin (*kai kai*) or leaf juice mixed with honey in cases involving snake and centipede bites or scorpion stings. The study found that more women than men, farmers/traders, people with no education or that has primary education, orthodox Christians or traditional worshippers and grossly of Igbo or related ethnicity utilized *T. Pachysiphon* as medicine more. GC-MS signature carried out on the methanol leaf extract of the plant identified a large spectrum of fingerprinting consisting of 32 bioactive compounds in the leaf extract. To the best of our knowledge this is the first time that the spectrum of fatty acids components of leaf of *T. pachysiphon* is being reported. These fatty acid ingredients likely contribute to and underscore, at least in part, the plant's efficacy and relevance in the folk medicine practices of the Kingdom's people.

5. ACKNOWLEDGMENTS

The authors would like to express profound gratitude to all the traditional medicine men of Ekebedi ancient Kingdom and its environs who volunteered indigenous knowledge for this work and, the Staff members of the Analytical Chemistry Unit, FIIRO, Oshodi Lagos for carrying out GC-MS analysis of the plant samples.

REFERENCES

- Elia, J., 2006. Tabernaemontana pachysiphon Stapf. In: Schmelzer, G.H. & Gurib-Fakim, A. (Editors). Prota 11(1): Medicinal plants/Plantes médicinales 1. [CD-Rom]. PROTA, Wageningen, Netherlands.
- Pratchayasakul, W., Pongchaidecha, A., Chittipakorn N. and Chittipakorn, S. (2008). Ethnobotany and ethnopharmacology of Tabernaemontana divaricata. Indian Journal Medicinal Plant Research 127: 313-335.
- Omino, E.A., Kokwaro, J.O. (1993). Ethnobotany of apocynaecea species in Kenya. J. ethnopharmacol. 40(3): 167-180. DOI: 10.106/0378-8741(93)90065-D.
- Mansoor, T.A., Borrhalno, P.M., Dewanjee, S...Ferreira, M-J.U. (2013). Monoterpen bisindole alkaloids from the African medicinal plant *Tabernaemontana elegans*, induce apoptasis in HCT116 human colon cancer cells. J. Ethnopharmacol. 149(2); 463-470 DOI: 10.1016/j.jep.2013.06.051.
- Abubakar. I. B., Loh, H-S. (2016). A review of the ethnobotany, Pharmacology and phytochemistry of *Tabernaemontana corymbosa*. J. Pharm. Pharmacol. 68(4): 423-432. DOI: 10.1111/jphp.12523.
- 6. Naidoo, C.M., Naidoo, Y., Dewir, Y.H., Singh, M., Lin, J. (2023). Phytochemical composition and antibacterial evaluation of *Tabernaemontana ventricosa* Hochst Ex A. DC leaf, stem and latex extracts. South Afr. J. Bot. 152: 147-164. DOI: 10.1016/j.sajb.2022.11.026.
- Uwumarongie, O.H., Onwukaeme, D.N., Obasuiyi, O. (2007). Antimicrobial activity of methanolic leaf extract of *Tabernaemontana pachysiphon* Stapf. Nigerian Journal *Natural products Medicine* 11: 23-25. DOI: 10.4314/njnpm.v11i1.11872.

- Edinoff AN, Patel AS, Baker MW, Lawson J...Kaye AD. (2021). Conolidine: A novel plant extract for chronic pain. Ansth. Pain Med. 11(6): e121438 DOI: 10.5812/aapm.121438.
- 9. Duru, C.M., (2010). Mbata, T.I. The antimicrobial activities and phytochemical ethanolic leaf extracts screening of of Hedranthera bateri Hook and pachysiphon Stapf. Tabernaemontana J. Develop Biol. Tissue Engineering 2(1): 001-004.
- Ruttoh, E.K., Bli, C., Tarus, P.K., Karini, L.K., Okemo, P. (2009). Antifungal activity of *Tabernaemontana stapfiana* Britten organic extracts. Pharmacognosy Research 1: 387-391.
- Marathe, M.P., Rasane, M.H., Kumar, H., Patwardhan, A.A., Shouche, Y.S., Diswanan, S.S. (2013). *In vitro* antibacterial activity of *Tabernaemontana alternifolia* stem bark aqueous extract against clinical isolates of methicillin resistant *Staphylococcus aureus*. Annals of Clinical Microbiology and Antimicrobials 12: 26-33.
- Anyadoh-Nwadike, 12. Duru. C.M., S.O., Okechukwu, R.I. (2015). Antimicrobial activity and phytochemical analysis of aqueous and ethanolic of the bark extracts of Tabernaemontena pachysiphon Stapf. Scientific Journal of Public Health 3(1-5): 008-013.
- Hoft, M., Verporte, R., Beck, E. (1998). Leaf alkaloid contents of *Tabernaemontana* pachysiphon as influenced by endogenous and environmental factors in natural habitats. Planta Med. 64(2): 148-152. DOI: 10.1055/s-2006-957393.
- Beek, T. A., Kaujlaers, F. L. C., Thomassen, P. H. A. M. Verporte, R., Svendsen, A. B. (1984). Phytochemistry 23(8): 1771-1778.
- 15. Pallant, C.A., Cromarty, A.D., Steemkamp, V. (2012). Effect of an alkaloidal fraction of

Tabernaemontana elegans (Stapf.) on selectedmicro-organismsJournal140(2):398-404.Doi:10.1016/j.jep.2012.01.036.Doi:

- Piana, M., Boligin, A.A., de Brum, T.F., Zadra, M,...Boligan, A.A., Athayde, M.L. (2014). Phytochemical analysis and antioxidant capacity of *Tabernaemontana catharinensis* A. DC fruits and branches Anais de Academia Brasiliera de Ciencias 86(2): 881-888.
- Pallant. A. C. (2010). Bioactivity of alkaloidal fraction of *Tabernaemontana elegans* Stapf. Thesis (*MagisterScientiae*) submitted to the Department of Pharmacology, Faculty of Health Sciences, University of Pretoria, 2010.
- Naidoo, C.M., Naidoo, Y., Dewir, Y.H., Murthy, H.M., El-Tendawy, S., Al-Suhaibani, N. (2021). Major bioactive alkaloids and biological activities of Tabernaemontana Species. MDPI Plants 10: 313 Doi: 10.3390/plants.10020313.
- 19. Kalaimagal, C., Umamaheswari, G. (2015). Bioactive compounds from leaves of *Tabernaemontana divaricata* (L.). Int. Journal Recent Res. 6(4): 3520-3522.
- 20. Uwumaringie, H.O., Onwukaeme, D.N., Ighodaro I. (2018). GC-MS metabolite profiling, antnorciceptive antipyretic and activities of methanol stem bark extract of Tabernaemontana pachysiphon Stapf (Apocynaceae). Journal Sci. Practice 5(1): 228-230.
- 21. Kalaimagal C. (2019). Identification of bioactive compounds in flower of Tabernaemontana divaricata (L.) using GC-MS analysis. Asian J. Pharm. Clin. Res. 12(9): 129-132. Doi: 34559. 10. 22139/ajpcr.2019.v12i9.34559.
- 22. Kim, B-R., Kim. H.M., Jin, C.H., Kang, S-Y., Kim, J.B., Jean, Y.G., Park, K.Y., Lee, I-S., Han. A-R. (2020). Composition and antioxidant activities of volatile organic compounds in

radiation-bred Coreoppsis cultivars. MDPI Plants 9: 717 Doi: 10.3390/plants9060717.

- Mgbeahuruike, E.E., Yrjonen, T., Vourela, H., Holm, Y. (2017). Bioactive compounds from medicinal plants: focus on *PIper* species. South African J. Botany112: 54-59.
- Kadija, E.O, Adelfattah, E.M, Laila, B, Dalila B. (2021). Ethnobotanical study of medicinal plants used in the treatment of cancers in the city of Casablanca (West-Central Morocco). Tropical Journal Nat. Prod. Research 5(6): 1044-1054.
- 25. Antimicrobial Resistance Collaborators (2022): Global burden of antimicrobial resistance in 2019: A systematic analysis. Lancet 2022 399 (10325): 629-655 DOI: 10.1016/S0140-6736(21)027274-0. Erratumin: Lancet 2022 400(10358): 1102. DOI: 10.1016/S0140-6736(21)02653-2.
- 26. Santhyami, L., lina, A., Putri, A. (2024). Exploring urban ethnobotany: A case study of medicinal plants traded in Gede Hardjonagoro market, Surakata, Indonesia. Trop. J. Nat. Prod. Res. 8(4): 6839-6851.
- 27. Mgbeahuruike, E.E., Holm, Y., Vourela, H., Amandikwa, C., Fyrquist, P. (2018). An ethnobothanical survey and antifungal activity of *Piper guineense* used for the treatment of fungal infections in West African traditional medicine. J. Ethnopharmacology 229: 157-166.
- Okwu, D.E., Ukanwa, N. (2010). Isolation and characterization of flavonoids chalcones and anthocyanidines from Bridelia ferruginea Benth. Der Chemica Sinica 1(2): 21 – 28.
- Gopinath, S., Sakthidevi, G., Muthukumaraswamy, S., Mohan, V.R. (2013). GC-MS analysis of bioactive constituents of *Hypericum mysorense* (Hypericaceae). Journal of Current Chemistry Pharmaceutical Sciences: 3(1): 6-15.
- 30. Omotosho, A.E., Oluwafemi, E.O., Maikailu, S. (2014). Phytochemical analysis of *Cnidoscolus*

aconitifolius (Euphobiaceae) leaf with spectrometric techniques. Nigerian Journal of Pharmaceutical and Applied Research 3(1): 38-49.

- 31. USDA (United States Department of Agriculture) (1995). Names and structural partterns of phytochemicals. In: Dr Jim Dukes Phytochemical and Ethnobotanical Databases, UDSA, 1995. http://phytochemicals.nal.usda.gov, Accessed September 23, 2017.
- 32. NIST (National Institute for Standards and Technology). https://www.nist.gov/pml.
- Vineetha, M. S., Bhavya, J., Veena, S. M., Mirajkar, K. K., Muddapur, U., Ananthraju, K. S., Zarmeer, F., More, S. S. (2020): In vitro and in vivo inhibitory effects of *Tabernaemontana alternifolia* against Naja naja venom. Saudi Pharm. J. 28(6): 692-697 DOI: 10.1016/j.jsps.2020.04.010.
- Onyipat, J-L. E., Okoronkwo, I. L., Ogbonnaya, N. (2011). Complementary and alternative medicine use among adults in Enugu, Nigeria. BMC Complement. Alternative Med. 11: 19. DOI: 10.1186/1472-6882-11-19.
- 35. Aina, O., Guatam, L., Simkhada, P., Hall, S. (2020). Prevalence and determinants of knowledge about herbal medicine and nonhospital utilization in southwestern Nigeria: a cross sectional study. BMJ Open 10(9): DOI: 10.1136/bmjopen-2020-040769.
- 36. Li et al. (2020). Traditional medicine usage among adult women in Ibadan, Nigeria: A cross sectional study. BMC Complement. Med. Ther. 20: 93 DOI: 10.1186/s12906-020-02881-z.
- 37. Jackson, I. L., Akpan, M. R., Akwaowoh, A. E., Sampson, V. I. (2024). The attributes and determinants of herbal medicine use amongst pregnant women attendin antenatal clinics in Uyo, Nigeria. J. Herbal Med. 46: DOI: 10.1016/j.herbmed.2024.100891.

- 38. Poorima, K., Gopalakrishnan, V.K. (2014). Anticancer activity of *Tabernaemontana coronaria* against carcinogen induced clear cell renal cell carcinoma. Chinese J. Biol. 2014(1): 584074.
- 39. Puranik, S., Hiremath, M., Neril, R., Ghagane, S. (2018). Evaluation of in vitro antioxidant and anticancer activities of Tabernaemontana divaricata leaf extracts against T-24 human bladder cell lines. Int. J. Cancer Res. DOI; 10.3923/ijcr.2018.100.108.
- Boligon, A.A., Piana, M., Schawnz, T.G., Periera, R.P., Rocha, J.B.T., Athayde, M.L. (2014). Chromatographic and antioxidant capacity of *Tabernaemontana cathar*inensis. Nat Prod Comm 9 (1): 61-64.
- 41. Uwumarongie, O.H., Onwukaeme, D.N. Pharmacognostic and toxicity (2011). evaluation stem bark of the of Tabernaemoontana pachysiphon Stapf. (Apocynaceae). Nigeria Journal of Pharmaceutical Research 9(1): 63-71
- 42. Adulrahaman, A. A., Kolawole, O. M (2006). Traditional preparations and uses of maize in Nigeria. Ethnobotanical Leaflets 10: 219-227.
- Ambukkarasi, M., Thomas, P. A., Sundarajan, M., Pitchairaj G. (2016). GC-MS analysis and in vitro antioxidant activity of ethanolic extract of leaves of *Tabernaemontana divaricata*. Pharmacognosy J. 8(5): 451-458.
- Ezekwe, S.A,. Chikezie, P.C. (2017b). GC-MS analysis of aqueous extract of unripe fruit of *Carica papaya*. Journal Nutrition Food Sci. 7(3): 602-606.
- 45. Ezekwe, S.A., Chikezie, P.C. (2017a). GC-MS, hypoglycemic activity of aqueous extract of *Carica papaya* and its effect on blood lipid profile and hepatorenal tissues biomarkers of diabetic rats. Journal Diabetic Metabolism 8: 740. Doi: 10.4172/2156-6156.1000740.S.
- 46. Barathi, V., Anand, A.V., Anitha, P., Santhi, S. (2016). Chemical characterization of GC-MS

analysis of Tabernaemontana divaricata. Euro. J. Biomed. Pharm. Sciences 5(4): 451-454.

- 47. Orishapide, A.T., Ibekwe, N.N., Adesomoju, A.A., Okogun, J.I. (2012). Chemical composition and antibacterial activity of the seed oil extract of *Entandrophragma angolense* (Welw) C.D.C. African Journal of Pure and Applied Chemistry 6(13): 184-187.
- 48. Nazan, C., Yusuf, Z.K., Aygan, A. (2019). Investigation of bioactive compounds in relict endemic Ajuga relicta P. H. Davis (lamiaceae) from Turkey. Int. Journal Sec. Metabolite 6(3): 223-232.
- 49. Barwal, M. K., Ram, C., Gurjar, P. S...Kumar, P. (2022). The bioactive compounds and fatty acid profile of bitter apple seed oil obtained in hot, arid environments. Horticulturea DOI: 10.3390/horticurea8030259.
- Rajeswari, G. Murugan, M., and Moghan, V. R. (2012). GC-MS analysis of bioactive components of *Hugonia mystax* L. (Linaceae). Research Journal of Phamacy Biology and Chemical Science 3(4): 301-308.
- 51. Dayrit, F.M. (2014). The properties of Lauric acid and their significance in coconut oil. Journal of the American Oil Chemists' Society 92: 001-015.
- 52. De Roos, N.M., Schouten, E.G., Katan, M.B. (2001). Consumption of solid fat rich in lauric acid results in a more favourable serum lipid profile in healthy men and women than consumption of a solid fat rich in trans-fatty acids. The Journal of Nutrition 131(2): 242-245. Doi: 10.1093/jn/131.2.242.
- 53. Dayrit, F.M., Newport, M.T. (2020). The potential of coconut oil and its derivatives as effective and safe antiviral agent against the novel coronavirus (n-CoV-2019). icp.org.ph/2020/01/the-... Accessed June 13, 2021.
- 54. Nakatsuji, T., Kao, M.C., Fang, J-Y., Zouboulis, C.C., Zhang, L., Galo, R.L. Huang, C-M.

(2009). Antimicrobial property of lauric acid against propionibacterrium acnes: Its therapeutic potential for inflammatory acnes vulgaris. Journal Invest. Dermatol. 129(10): 2480-2488.Doi: 10.1038/jid.2009.93.

- 55. Matsue, M., Mori, Y., Nagase, S., Sugiyama, Y., Hirano, R., Ogai, K., Ogura, K., Kurihara, S., Okamoto, S. (2019). Measuring the antimicrobial activity of lauric acid against various bacteria and human gut microbiota using new method. Cell Transplant 28(12): 1528-1541. Doi: 10.1177/0963689719881366.
- 56. Ugbogu, O.C., Onyeagba, R.A., Chigbu, A.O. (2006). Lauric acid content and inhibitory effects of palm kernel oil on two bacterial and *Candida albicans*. African Journal of Biotechnology 5(11): 1045-1047.
- Nitibani, F.E., Siswanta, J.D., Solikhah, E.N. (2016). Isolation and antibacterial activity test of lauric acid from crude coconut oil (*Cocos nucifera* L). Procedia Chemistry 18: 132-140.
- Walters, D.R., Walker, R.L., Walker, K.C. (2003). Lauric acid exhibits antifungal activity against plant pathogenic fungi. J. Phytophathol. 151: 228-230.
- 59. Lifongo, L.L., Simoben, C.V., Ntie-Nkang, F., Babiaka, S.B., Judson, P.N. (2014). A bioactivity versus ethnobotanical survey of medicinal from Niger, West Africa. Natural Products Bioprospecting 4(1): 001-019.
- 60. Okwu, D.E., Ighodaro, B.U. (2010). GC-MS analysis of bioactive compounds and antibacterial activity of the oil fraction from the leaf of *Alstonia boonei* De Wild. Der Pharma Chemica 2(10: 261-272.
- Huang, C.Y., Hong, P., Zhang, Z.Y., Song, J. (2010). Evaluation of antioxidant and antitumor activities of lemon essential oil. Journal of Medicinal Plant Research 4(18): 910-915.
- 62. Salamus, J., Baird, M.S. (1995). Biologically active cyclopropanes and cyclopropenes. Journal of Medicinal Chemistry 2: 511-542.

- 63. Anand, C.S., Mehendale, L.M. (2014). Dichloroacetic acid (DCA). Sciencedirect.com/topics/medicine-anddentistry/dichloroacetic-acid... Accessed June 15, 2021.
- 64. Zaral, Z., Kadri, A., Choba, I.B., Mansour, R.B., Bekir, A., Mejdoub, H., Gharsallah, N. (2011). *In vitro* evaluation of antibacterial, antifungal, and cytotoxic properties of *Marrubium vulgare* L. essential oil grown in Tunisia. Lipids and Health 10: 161-168.
- 65. Akpuaka, A., Ekwenchi, M. M., Dashak, D. A., Dilda, A. (2013). Biological activities of characterized isolates of n-Hexane extract of *Azadirachta Indica* A. Juss (Neem) leaves. Nature and Science 11(5): 141-147.
- 66. Oshiobugie, M.J., Olaniyi, A.M., Rapael, A.O. (2017). AAS and GC-MS analysis of phytocomponents in the leaf, stem and root of *Azadirachta indica* A. Juss (Dongoyaro). British Journal Pharm. Research 15(4): 1-12.
- 67. Cherney, K., Kramer, O. (2019). Benzyol peroxide for acne: How to treat acne with benzyol peroxide. www.healthline.com/health/benzyol-peroxidefor-acne... Accessed June 5, 2021.
- Matsugo, S. Mizuno, M., Konishi, T. (1985). Free radical generating and scavenging compounds as a new type of drugs. Current Medicinal Chemistry 2(4): 763-780.
- 69. Premjanu, N., Jaynthy, C. (2014). Antimicrobial activity of diethyl phthalate: An *insilico* approach. Asian Journal of Pharmacy and Clinical Research 7(4): 141-145.
- 70. Mustafa, M.F.M., Alamri, S.A., Taha, T.H, Alrumman, S.A. (2013). In vitro antifungal activity of Argemone ochrolueca Sweet latex against some pathogenic fungi. Afr. J. Biotechnol. 12(1): 1132-1137.
- Mangamuri, U., Muvva, V., Poda, S., Naragani, K., Munaganti, R.K., Chitturi, B., Yenamanndra, V. (2016). Bioactive metabolites

produced by *Streptomyces cheonanensis* VUK-A from Coringa mangrove sediments: isolation, structure elucidation and bioactivity. Biotechnology 6: 63 Doi: 10.1007/s13205-016-0398-6.

- 72. Ahsan, T., Chen, J., Zhao, X., Irfan, M., Wu, Y. (2017). Extraction and identification of bioactive compounds (eicosane and dibutyl phthalate) produced by Streptomyces strains KX852460 for the biological control of *Rhizoctonia solani* AG-3f strain KX852461to control target spot disease in tobacco. AMB Express 7(1): 54 Doi: 10.1186/s13568-017-0351-z.
- 73. Shobier, A.H., Ghani, S.A.A., Khouloud, M., Barakat, K.B. (2016). GC/MS spectroscopic approach and antifungal potential of bioactive extracts produced by marine macroalgae. Egyptian Journal of Aquatic Research 42: 289-299. Doi.org/10.1016/j.ejar.2016.07.003 1687.
- 74. Tataranni, T., Piccoli, C. (2019). Dichloroacetic acid and cancers? An overview towards clinical applications. Homeostasis and Cancer Article ID.8201079. Doi: 1155/2019/8201079.
- 75. Wang, H-L., Brattstrom, O., Brakefield, P.M., Franke, W., Lofstedt, C. (2014). Identification and synthesis of novel male specific esters in the wings of the tropical butterfly, *Bycyclus martius sanaos*. J. Chem. Ecology 40: 549-559.
- 76. Wang, H-L., Geertsema, H., van Nieukerken, E.J., Lofstedt, C. (2015). Identification of the female-produced sex pheromone... of the leafminer *Holocacista capensis* infesting grapevine in South Africa. J. Chem. Ecology 41: 724-731.
- 77. Poorima, K., Perumal, P.C. (2014): Protective effects of ethanolic extract of *Tabernaemontana divaricata (L.)R. Br.* against DEN and Fe NTA induced liver necrosis in wistar rats. Biomed Res. Intern.2014, 1-9.

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

Bacteriocinogenic Activity of *Lysinibacillus fusiformis* NR_042072.1 Isolated from Cow Milk

Research Article

Majekodunmi Adedayo^{1,*} (10), Taofeeq Abdulkareem¹ (10)

¹Kwara State University, Faculty of Pure and Applied Sciences, Department of Microbiology, Malete, Nigeria

Author E-mails: majekodunmi.adedayo@kwasu.edu.ng taofeeqo93@gmail.com M. Adedayo ORCID ID: 0000-0002-4266-7298 T. Abdulkareem ORCID ID: 0000-0002-7430-7537

*Correspondence to: Majekodunmi Adedayo, Kwara State University, Faculty of Pure and Applied Sciences, Department of Microbiology, Malete, Nigeria

DOI: 10.38061/idunas.1491313

Received: 02.07.2024; Accepted: 30.11.2024

Abstract

The prevailing increase in the search for bio-preservatives in the food industry has raised global concern. Biological substances with health benefits and no toxicity are considered as alternatives to the chemicals in food processing. Bacteriocins, which are proteinaceous substances with preservative properties, are now gaining attention in this regard and the search for new organisms for production is becoming global. This study focuses on the analysis of bacteriocinogenic activity of a local strain of Lysinibacillus fusiformis NR 042072.1 isolated from fresh cow milk obtained from Gaa Mobolohunduro, Tanke, Ilorin, Kwara State, Nigeria. Lactic acid bacteria were isolated from cow milk, characterized and identified using standard microbiological methods. Bacteriocin was extracted from the isolate, partially purified, and characterized using standard methods; and the antibacterial activity against some foodborne bacteria which are Escherichia coli, Staphylococcus aureus, Pseudomonas aeruginosa and Bacillus subtilis was determined using agar-well diffusion method. The effect of certain physicochemical parameters on the antibacterial activity of the bacteriocin was also determined. The isolate was identified as Lysinibacillus fusiformis NR 042072.1. The bacteriocin showed antibacterial action against E. coli and P. aeruginosa with a zone of inhibition of 17.0±0.5 and 20.0±1.0 mm, respectively. The bacteriocin was active within temperatures of 4-50 °C, pH 5-7.5, bile salts concentration of 0.6-1.0 % and in the presence of the enzymes trypsin and pepsin. In conclusion, the bacteriocin produced by Lysinibacillus fusiformis NR 042072.1 could control food spoilage caused by the test organisms.

Keywords: Bacteriocin, Fresh Cow Milk, Lysinibacillus Fusiformis, Antimicrobial Action, Food Preservation.

1. INTRODUCTION

The menace of food spoilage and foodborne diseases with devastating effects on food production and consumers have become a global challenge. On the other hand, the method of food preservation with chemicals has been reported to be toxic and highly risky, hence, creating more problems than it solves. Research on food preservation has recently been focused on the search for alternative bio-preservatives to ameliorate the health challenges posed by chemicals used in food preservation.

Bacteriocins are protein substances with no toxic implications on humans but effective as an antibacterial agent. (Kaur-Sidhu and Nehra, 2021; O'Connor etal., 2020). They are reported to be selectively toxic on food spoilage microbes without self-destruction of the producing bacteria because they possess self-defensive proteins (Kaur-Sidhu and Nehra, 2021).

Bacteriocins produced by the lactic acid bacteria are generally considered as safe in food and have a high potential to inhibit proliferation and destroy other spoilage bacteria and pathogens in food and thus extend shelf life of food. They are also very stable in food (Johnson et al., 2018; Colombo et al., 2018; Kaur Sidhu and Nehra, 2021). Their bactericidal effect is through lysis of cell wall and membrane, destruction of DNA and prevention of protein synthesis (Meade et al., 2020; Yang et al., 2014). Bacteriocins have been naturally found and consumed in fermented foods throughout human history hence the renewed effort on their development and application in food (O'Connor et al., 2020). However, to date, there are few commercialized acceptable bacteriocins such as leucocin A, nisin, enterocins, pediocin and microgad (Kaur Sidhu and Nehra, 2021; Raj et al., 2021).

Lysinibacillus fusiformis is a member of the family Bacillaceae in the phylum Fermicutes. The bacterium is found in many habitats including soil, plants, and in animals (Hashmi et al., 2020). Species in the genus Lysinibacillus are notably recognized by the presence of a polyphasic cell wall peptidoglycan (Hashmi et al., 2020). Many Bacilli have been studied for their biotechnological potential; in the list are Pediococcus acidilactici, Lactococcus lactis, Streptococcus and Lactobacillus species have been employed in bacteriocin and dairy beverages production (Raj et al., 2021) but the antibacterial bacteriocin production potential of Lysinibacillus has received less attention. The current research is aimed at isolating and identifying bacteriocin-producing organisms for use as alternative to chemical in food bio-preservation.

2. MATERIALS AND METHODS

Source of BacteriaTest Strains

Four common foodborne bacteria strains, namely, *Staphylococcus aureus, Bacillus subtilis* (Gram positive) *Pseudomonas aeruginosa* and *Escherichia coli* (Gram negative) isolated from spoiled food were procured from the culture collection centre of the Microbiology Department, University of Ilorin, Ilorin, Kwara State, to study the antibacterial activity of the bacteriocin produced by the isolated strain.

Isolation and Identification of Lactic Acid Bacteria

Bacteria were isolated from raw milk samples using De Man, Rogosa and Sharpe (MRS) agar (Merck-GranuCult-110660). Plates were incubated at 37^oC for 48 hours. The pure colonies of isolates obtained were maintained on agar slants at 4^oC for further research (Sidhu and Nehra, 2020). Lactic acid bacteria obtained were subjected to molecular identification using 16S rRNA gene amplification technique. DNA was extracted from the isolate using a genomic DNA extraction kit, and universal primers (27F and 1492R) were used to amplify the conserved region of 16S rRNA gene (Sidhu and Nehra, 2020). The pure PCR product was to obtain a consensus sequence using Bioedit software (Hall, 1999). The isolate was confirmed by comparing the homology of the resulting consensus sequence with a database of NCBI using BLAST (Altschul, 1997; Altschul*et al.*, 1990), ascension number was obtained from NCBI for the isolate, and phylogenetic tree showing relatedness was also constructed using the BLAST results.

Extraction and Partial Purification of Bacteriocin

Crude bacteriocin was extracted from the isolate following the modified method of Yang *et al.* (2012). A 48-hour old culture of the isolate was subjected to centrifugation at 10, 000 rpm for 15 min at 4^oC to obtain cell-free supernatant (CFS). The pH of the CFS was adjusted to 6.0 using 1N NaOH. It was partially purified by precipitating crude bacteriocin-900 ml (prepared from 1 L MRS broth) with 429.96 g of ammonium sulphate up to 70% saturation levels overnight at 4^oC. The mixture was centrifuged at 4000 rpm for 1 hour and the surface and bottom pellicles were harvested and resuspended in sodium phosphate buffer (50 mM, p H 6.5) (Goyal *et al.*, 2018). The solution was filtered using a Millipore filter (0.22 μ m) paper.

Determination of Antibacterial Activity of Partially Purified Bacteriocin

The antibacterial screening of the partially purified bacteriocin produced by the isolate was carried out using Agar-well diffusion method (Chen *et al.*, 2019), Exactly 0.1 ml of one day old culture of the test bacteria were seeded on to already solidify Mueller Hilton agar plates after the cultures have been adjusted to 0.5 % Mcfarland turbidity (containing approximately 1.5 x 10^8 CFU/ml). Wells bored in the plates were filled with 50 µl of the bacteriocin. The plates were incubated at 37 °C for 18 hours. Zones of inhibition around the test organisms were measured in millimetres using a scale rule. Bacteriocin activity was determined in terms of the diameters of the zones of inhibition around the wells against the test bacteria.

Effect of Enzymes on Stability of Bacteriocin

The stability and activity of bacteriocins in the gut are affected by the presence of digestive enzymes secreted into the environment for proper digestion of food. Hence, the stability of the partially purified bacteriocin in the presence of some enzymes was determined to mimic gut condition as described by Zhang *et al.* (2018). The bacteriocin was treated with 1 mg/ml of trypsin, pepsin and α -amylase. The mixtures were incubated at 37^oC for 2 hours and heated for 10 min at 95 ^oC. The treated bacteriocin was screened for antibacterial potency against the test organisms using agar-well diffusion method.

Thermal Stability of Bacteriocin

The thermal stability of the bacteriocin was determined by incubating the solution at temperatures of $4 - 80^{\circ}$ C for 15minutes (Elayaraja *et al.*, 2014). Effect of temperature on the antibacterial potency of the bacteriocin was determined against the test organisms using agar-well diffusion method.

pH Stability of Bacteriocin

To study the effect of pH on the bacteriocin, the pH of the bacteriocin was adjusted to between 3.0 and 11.0 using 1 N HCl and 1 N NaOH. The mixture was incubated for 30 minutes at 37 0 C (Miao *et al.*, 2014). The effect of varying pH values on the antibacterial activity of the bacteriocin was determined against the test organisms using agar-well diffusion method.

Effect of Bile Salts on The Stability of The Bacteriocin

The effect of bile salts on the bacteriocin was studied by adding bile salts of concentrations of 0.1 to 0.6%. The solution was incubated for 30 minutes at 37° C. The effect of varying bile salts concentrations was determined against the test organisms using agar-well diffusion method. Bacteriocins without the bile salts were set as control (Tambekarand Bhutada, 2010).

Statistical Analysis

The data was shown as the mean \pm standard deviation (SD, n = 5). The results obtained was analysed using SPSS 18.0 program for Windows (Munich, Germany).

3. RESULTS

Isolation and Identification of Bacteriocin Producing Strain

The isolate was a Gram-positive, catalase- negative, and oxidase-negative bacillus. Sequencing of the amplified 16S rRNA gene and its homology search using BLAST identified the isolate to be *L. fusiformis* (Figure 1). The ascension number NR_042072.1 was assigned. The agarose gel electrophoresis of 16SrRNA amplified genes from *L. fusiformis* NR_042072.1 was presented in Figure 2.



Figure 1. Phylogenetic tree of 16S rRNA gene L. fusiformis NR_042072.1 strain



Figure 2. Agarose gel electrophoresis of 16SrRNA amplified genes from L. fusiformis NR_042072.1 (L5)

Antibacterial Activity of L. fusiformis NR_042072.1 on Test Organisms

The bacteriocin *L. fusiformis* NR_042072.1 was only potent against two of the test bacteria which were *E. coli* and *P. aeruginosa* (Table 1). A zone of inhibition of 20 mm was obtained against *P. aeruginosa* while 17 mm was recorded against *E. coli*.

 Table 1. Antibacterial Activity of Bacteriocin from L. fusiformis NR_042072.1 on Test Organisms Measured by the Diameter of Zones of Inhibition (mm)

Test Organisms	E. coli	S. aureus	P. aeruginosa	B. subtilis
Zone of Inhibition	17±0.5	-	20.0±1.0	-

Key: -=Negative

Characterization of Partially Purified Bacteriocin

Effect of Enzymes on Bacteriocin Produced by L. fusiformis NR 042072.1 on Test Organisms

The bacteriocin became active against *S. aureus* (Table 2) after treatment with enzymes. The presence of the enzymes enhanced the bactericidal activity of the proteinaceous bacteriocin.

Table 2. Effect of Enzymes on the Antibacterial Activity of Bacteriocin from Lysinibacillus fusiformis NR_042072.1 on Test

 Organisms Measured by the Diameter of Zones of Inhibition (mm)

Test Organisms		E. coli	S. aureus	P. aeruginosa	B. subtilis
	α –Amylase	21.5±0.5	-	19.0±1.0	-
Enzymes	Pepsin	-	23.0±1.0	21.0±1.0	-
	Trypsin	-	22.5±0.5	-	-

Key: - =No activity

Effect of Temperature on Bacteriocin Produced by L. fusiformis NR_042072.1 on Test Organisms

The bacteriocin produced by *L. fusiformis* NR_042072.1 was stable over a wide range of temperature (Table 3). The bacteriocin was still potent against the test bacteria at a relatively high temperature of 50° C; however, there was no activity at 80° C.

Table 3. Effect of Temperature on the Antibacterial Activity of Bacteriocin from *L. fusiformis* NR_042072.1 on Test Organisms Measured by the Diameter of Zones of Inhibition (mm)

Test Organisms		E. coli	S. aureus	P. aeruginosa	B. subtilis
	4	20.0±1.0	-	16.5±0.5	-
_	20	-	-	18.5±0.5	-
Temperature (⁰ C)	30	-	17.5±0.5	-	-
	50	20.0±0.0	21.5±0.5	-	20.5±1.5
	80	-	-	-	

Key: -=Negative

Effect of pH on Bacteriocin Produced by L. fusiformis NR_042072.1 on Test Organisms

The activity of the bacteriocin was better enhanced at pH 5 and 7.5. There was no effect at pH 9. The bacteriocinogenic activity was more pronounced at weakly acidic and near neutral pH (Table 4).

Table 4. Effect of pH on the Antibacterial Activity of Bacteriocin from *L. fusiformis* NR_042072.1 on Test Organisms Measured by the Diameter of Zones of Inhibition (mm)

Test Organi	sms	E. coli	S. aureus	P. aeruginosa	B. subtilis
	5.0	12.5±0.5	-	22.5±0.5	-
pН	7.5	18.0±0.0	-	25.0±1.0	-
	9.0	-	-	-	-

Key: - =No activity

Effect of Bile Salts on Bacteriocin Produced by L. fusiformis NR_042072.1 on Test Organisms

Antibacterial activity of bacteriocin produced by the *L. fusiformis* NR_042072.1 was stable after treatment with 0.6% and 1.0% of bile salts, though the activity was reduced (Table 5).

 Measured by the Diameter of Zones of Hillottion (hill)

 Staphylococcus
 Pseudomonas

 Test Organisms
 Escherichia coli

 aureus
 aeruginosa

 Table 5. Effect of Bile Salts on the Antibacterial Activity of Bacteriocin from L. fusiformis NR_042072.1 on Test Organisms

 Measured by the Diameter of Zones of Inhibition (mm)

				0	
	0.6	10.5±0.5	-	11.5±0.5	-
Bile	0.8	-	-		-
salt (%)	1.0	12.0±0.0	11.0±1.0	12.0±0.0	-

Key: - =No activity

4. DISCUSSION

The present investigation highlights the production, characterization, partial purification and antibacterial potency of bacteriocin from *Lysinibacillus* NR_042072.1. The presence of pathogenic organisms in food and their toxic metabolites have been variously reported as a major cause of food related illnesses worldwide. Infections from Foodborne pathogens have become more prevalent in recent decades due to industrialization in general and dependence on ready to eat street-vended foods.

Lactic acid bacteria have been positively identified as a reliable source of bacteriocins to prevent pathogens from surviving and multiplying in food (Zoghi *et al.*, 2021). The obtained phylogenetic tree proved that the isolated bacterium was close to *L. fusiformis* (NR_042072.1). Bacteriocin produced by the *Lysinibacillus* NR_042072.1 was potent on two of the test organisms (*E. coli* and *P. aeruginosa*) but less active against *S. aureus* and *B. subtilis*.

Bacteriocins are generally known to be more effective against closely related Gram-positive species; however, the findings of this study were contrary. The work of Sidhu and Nehra (2020); De-Giani *et al.* (2019) reported the possibility of similar trend antibacterial activity of bacteriocin from bacteria. The activity of bacteriocin through mechanisms such as creating pores in cell envelope, inactivating anionic carrier and enzyme activity, and pore formation in cell membrane could have aided the activity against the Gram-negative bacteria whose outer cell wall is resistance to the action of bacteriocin.

The bacteriocin became more potent after it was exposed to proteolytic enzymes. It became active against *S. aureus* (Table 2) against the bacteriocin not exposed to the enzymes. The presence of the enzymes supposedly contributed to its antibacterial effect. The bacteriocins and enzymes are proteinaceous material and a level of synergy in action is advantageous to bacteriocin's potency. The enzyme amylase, pepsin and trypsin are secreted naturally in the gut to aid food digestion, the bacteriocin must be relatively stable in the presence of these enzymes if there will be any activity. Contrary to this finding was the report of Sidhu and

Nehra (2020); Zhang *et al.* (2018); Elayaraja *et al.* (2014) on partial and complete loss of antibacterial potency of bacteriocins after exposure to enzyme treatment. The enhanced ability of the bacteriocin against the test organisms after treatment with the enzymes confirms its proteolytic nature.

The antibacterial potency of the bacteriocin was most prominent between temperatures of 4-50°C, with 50°C as the optimum. Though it was still stable at 80°C, there was no antibacterial potency at this very high temperature. Bacteriocins been protein are heat sensitive and are denatured at high temperature. The potency of the bacteriocin between 30-50°C is desirous because this shows that it will be active in the human digestive system and in food processing. A similar submission was made by Sidhu and Nehra (2020) on loss of potency at high temperatures. On the contrary, Zhang et al. (2018) reported that bacteriocin Lac-B23 retained its antibacterial effect at an extremely high temperature of 121°C. In another study, bacteriocin from species of Lactobacillus remained active after heat treatment (Moghaddam et al., 2006). The variation in activity observed could be based on the fact that bacteriocins are unique in their behavior, being protein, they are sensitive to heat and the fact that the test organisms were not the same coupled with varied environmental questions. However, the bacteriocin obtained in this study was still effective at 50°C and could therefore be used in food and dairy industry for food preservation at moderately high temperature. pH is notable among the factors that affect the antibacterial activity of bacteriocins. From the study, bacteriocin produced by L. NR 042072.1 was limited at high acidic and alkaline pH but more potent around pH 5 as the optimum and towards neutrality (Table 4). This observation is similar to the previous authors' report on the stability and activity of bacteriocin at pH around neutral (Sidhu and Nehra, 2020; Zhang et al., 2018). In another report, antibacterial activity of bacteriocin was found to vary indirectly with the increase in pH until zero activity was detected (Wang et al., 2018).

The gastrointestinal tract of man and other animals secretes bile salts; which is required for complete breakdown of lipids during digestion, bacteriocin must not be inhibited by the bile salts for effectiveness hence, the antibacterial activity of the synthesized bacteriocin was tested in the presence of bile salts at concentration similar to the gut; the bacteriocin however, was relatively stable and active against the test organisms in the presence of bile salts although zones of inhibition were reduced. This observation suggests that the bacteriocin could remain stable in the gut if consumed in food and be active against foodborne pathogens in the presence of bile salts secreted in the gut.

5. CONCLUSION

This study reports the extraction of bacteriocin from lactic acid bacteria *L. fusiformis* NR_042072.1 isolated from fresh cow milk. The bacteriocin displayed antibacterial activity against both Gram-positive and Gram-negative foodborne pathogens tested suggesting its potential for usability as a bio-preservative in food.

6. ACKNOWLEDGMENTS

We would like to thank the reviewers.

REFERENCES

- Altschul, S. (1997). Gapped BLAST and PSI-BLAST: A new generation of protein database search programs. Nucleic Acids Research, 25(17), 3389–3402. https://doi.org/10.1093/nar/25.17.3389.
- Altschul, S. F., Gish, W., Miller, W., Myers, E. W., & Lipman, D. J. (1990). Basic local alignment search tool. Journal of Molecular Biology, 215(3), 403–410. https://doi.org/10.1016/S0022-2836(05)80360-2.

- Chen, C.-C., Lai, C.-C., Huang, H.-L., Huang, W.-Y., Toh, H.-S., Weng, T.-C., Chuang, Y.-C., Lu, Y.-C., & Tang, H.-J. (2019). Antimicrobial Activity of Lactobacillus Species Against Carbapenem-Resistant Enterobacteriaceae. Frontiers in Microbiology, 10, 789. https://doi.org/10.3389/fmicb.2019.00789
- Colombo, M., Castilho, N. P. A., Todorov, S. D., & Nero, L. A. (2018). Beneficial properties of lactic acid bacteria naturally present in dairy production. BMC Microbiology, 18(1), 219. https://doi.org/10.1186/s12866-018-1356-8.
- De Giani, A., Bovio, F., Forcella, M., Fusi, P., Sello, G., & Di Gennaro, P. (2019). Identification of a bacteriocin-like compound from Lactobacillus plantarum with antimicrobial activity and effects on normal and cancerogenic human intestinal cells. AMB Express, 9(1), 88. https://doi.org/10.1186/s13568-019-0813-6.
- Elayaraja, S., Annamalai, N., Mayavu, P., & Balasubramanian, T. (2014). Production, purification and characterization of bacteriocin from Lactobacillus murinus AU06 and its broad antibacterial spectrum. Asian Pacific Journal of Tropical Biomedicine, 4, S305–S311. https://doi.org/10.12980/APJTB.4.2014C537.
- Goyal, C., Malik, R. K. & Pradhan, D. (2018). Purification and characterization of a broad spectrum bacteriocin produced by a selected Lactococcus lactis strain 63 isolated from Indian dairy products. Journal of Food Science and Technology, 55(9), 3683–3692. https://doi.org/10.1007/s13197-018-3298-4.
- Hashmi, I., Bindschedler, S. & Junier, P. (2020). Firmicutes. In Beneficial Microbes in Agro-Ecology (pp. 363–396). Elsevier. https://doi.org/10.1016/B978-0-12-823414-3.00018-6.
- Johnson, E. M., Jung, Dr. Y.-G., Jin, Dr. Y.-Y., Jayabalan, Dr. R., Yang, Dr. S. H. & Suh, J. W. (2018). Bacteriocins as food preservatives: Challenges and emerging horizons. Critical Reviews in Food Science and Nutrition, 58(16), 2743–2767. https://doi.org/10.1080/10408398.2017.134087

https://doi.org/10.1080/10408398.2017.134087 0.

10. Meade, E. Slattery, M. A. & Garvey, M. (2020). Bacteriocins, Potent Antimicrobial Peptides and the Fight against Multi Drug Resistant Species: Resistance Is Futile? Antibiotics, 9(1), 32. https://doi.org/10.3390/antibiotics9010032.

- Miao, J., Guo, H., Ou, Y., Liu, G., Fang, X., Liao, Z., Ke, C., Chen, Y., Zhao, L., & Cao, Y. (2014). Purification and characterization of bacteriocin F1, a novel bacteriocin produced by Lactobacillus paracasei subsp. Tolerans FX-6 from Tibetan kefir, a traditional fermented milk from Tibet, China. Food Control, 42, 48–53. https://doi.org/10.1016/j.foodcont.2014.01.041.
- Moghaddam, M. Z., M. S., A. M. M., & F. D. (2006). Inhibitory Effect of Yogurt Lactobacilli Bacteriocins on Growth and Verotoxins Production of Enterohemorrhgic Escherichia coli O157:H7. Pakistan Journal of Biological Sciences, 9(11), 2112–2116. https://doi.org/10.3923/pjbs.2006.2112.2116.
- O'Connor, P. M., Kuniyoshi, T. M., Oliveira, R. P., Hill, C., Ross, R. P., & Cotter, P. D. (2020). Antimicrobials for food and feed; a bacteriocin perspective. Current Opinion in Biotechnology, 61, 160–167. https://doi.org/10.1016/j.copbio.2019.12.023.

14. Sidhu, P. K., & Nehra, K. (2020). Isolation, screening and molecular Identification of bacteriocin producing lactic acid bacteria possessing wide spectrum antibacterial activity. Research Journal of Biotechnology, 15, 35–42.

 Sidhu, P. K. & Nehra, K. (2021). Bacteriocins of Lactic Acid Bacteria as Potent Antimicrobial Peptides against Food Pathogens. In M. K. Habib & C. Martín-Gómez (Eds.), Biomimetics. IntechOpen.

https://doi.org/10.5772/intechopen.95747.

- Tambekar, D. H., &Bhutada, S. A. (2010). An Evaluation Of Probiotic Potential Of Lactobacillus Sp. From Milk of Domestic Animals and Commercial Available Probiotic Preparations In Prevention of Enteric Bacterial Infections. 7.
- 17. Wang, Y., Zou, F., & Cegla, F. B. (2018). Acoustic waveguides: An attractive alternative for accurate and robust contact thermometry. Sensors and Actuators A: Physical, 270, 84–88. https://doi.org/10.1016/j.sna.2017.12.049.
- Yang, E., Fan, L., Jiang, Y., Doucette, C., & Fillmore, S. (2012). Antimicrobial activity of bacteriocin-producing lactic acid bacteria isolated from cheeses and yogurts. AMB

Express, 2(1), 48. https://doi.org/10.1186/2191-0855-2-48.

- Yang, S.-C., Lin, C.-H., Sung, C. T., & Fang, J.-Y. (2014). Antibacterial activities of bacteriocins: Application in foods and pharmaceuticals. Frontiers in Microbiology, 5. https://doi.org/10.3389/fmicb.2014.00241.
- Zhang, J., Yang, Y., Yang, H., Bu, Y., Yi, H., Zhang, L., Han, X., & Ai, L. (2018). Purification and Partial Characterization of Bacteriocin Lac-B23, a Novel Bacteriocin Production by Lactobacillus plantarum J23, Isolated from Chinese Traditional Fermented Milk. Frontiers in Microbiology, 9, 2165. https://doi.org/10.3389/fmicb.2018.02165.

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

The Effect of Tunçbilek Thermal Power Plant Waste Fly Ash on Mechanical Properties of Portland Cement

Research Article

Ahmad Hosseinpour Sheikhrajab^{1*}, Ahmet Erdal Osmanlıoğlu¹

¹Istanbul University, Faculty of Engineering, Department of Mining Engineering, Istanbul, Türkiye

Author E-mails: ahmad.hossein@ogr.iuc.edu.tr ahmet.osmanlioglu@iuc.edu.tr A. Sheikhrajab ORCID ID: 0009-0001-0132-3026 A. E. Osmanlıoğlu ORCID ID: 0000-0001-5547-7525

*Correspondence to: Ahmad Hosseinpour Sheikhrajab, Istanbul University, Faculty of Engineering, Department of Mining Engineering, Istanbul, Türkiye

DOI: 10.38061/idunas.1436316

Received: 13.02.2024; Accepted: 19.09.2024

Abstract

Cement production incorporates pozzolanic materials, with fly ash being a common waste utilized for its pozzolanic properties. Using fly ash has positive or negative effects on the cement, especially its physical and mechanical properties. In this study, the effect of substituting fly ash, a waste product from Tunçbilek thermal power plant, one of Turkiye's major power plants, for cement at 5%, 10%, 15%, 30%, and 50% by weight on the 2-, 7-, and 28-days compressive strength, 28-day ultrasonic property, and 150-day porosity of cement has been investigated. As a result, in comparison to the Portland cement, a decrease was observed in unit weight, ultrasonic properties, and 2-, 7- and 28-days compressive strength. Conversely, an increase was observed in porosity. According to the obtained results and EN 197-1 standards, the compressive strength of the samples with 5%, 10%, and 15% fly ash are between standard values.

Keywords: Cement, Fly Ash, Compressive Strength, Porosity.

1. INTRODUCTION

Cement is a fundamental building block of development. It is commonly mixed with concrete, a key material for constructing housing, roads, pipes, airports, and other infrastructures vital for supporting economic growth. Also, cement is used in the construction of factories, dams, hospitals, and schools, which are vital for the well-being, education, and health of society. After drinking water, cement is the second most widely used critical product globally, with over 4 billion tons consumed across various sectors annually. The cement industry, with a broad and multi-hued logistics network, contributes

significantly to the global economy, accounting for 5.4% of the global GDP and 7.7% of worldwide employment. Along with its multiple effects on employment and GDP, it plays a considerable role in achieving the United Nations Sustainable Development Goals [1].

Globally, there are more than 1,000 cement producers operating over 2,300 integrated cement manufacturing plants and more than 600 grinding stations. China has the largest cement production share at 57%, followed respectively by India with 7%, Vietnam with approximately 2.2%, and the United States with 2%, which collectively produce around three-quarters of the world's cement. Table 1 displays global cement production from 2010 to 2020 and key countries in global cement production [2].

Country	Production	Production	Production	Production
2	amount in 2010	amount in 2015	amount in 2019	amount in 2020
	(Million metric	(Million metric	(Million metric	(Million metric
	tons)	tons)	tons)	tons)
China	1,880	2,350	2,300	2,200
India	210	270	340	340
Vietnam	50	61	97	96
America	67.2	83.4	89	90
Indonesia	22	65	70	73
Türkiye	62.7	77	57	66
Iran	50	65	60	60
Brazil	59.1	72	54	57
Russia	50.4	69	56	56
Japan	51.5	55	53	53
Egypt	48	55	47	50
South Korea	47.2	63	50	50

Table 1. Major countries and production amounts in cement production worldwide from 2010 to 2020 [2]

According to the United Nations, by 2030, housing and basic urban infrastructure will be needed for 3 billion people, about 40% of the world's population. Therefore, supporting this sector ensuring the supply of essential materials like cement is crucial for meeting this fundamental need in developing economies. The International Finance Corporation (IFC) is one of the advocates for these sectors, having invested over \$3 billion in more than 25 countries over the last 15 years in such projects [3].

According to the materials mentioned in the above section, cement production is very important, so to produce cement, the supply of raw materials is also very important. In recent years, to prevent environmental pollution, also to reduce production costs, and increase economic productivity, cement producers have started incorporating waste materials such as pozzolanic materials. Mostly these pozzolanic materials are produced in other industries. One of these kinds of materials used in cement production is fly ash, a waste byproduct from coal-powered thermal power plants [4].

Coal-fired thermal power plants account for approximately 41% of global electricity production, and in some countries, this percentage may be even higher. Moreover, coal meets around 30% of the world's primary energy consumption [5]. With the increasing global demand for energy, coal consumption is

increasing too, leading to a significant increase in the production of fly ash (exceeding 600 million tons annually). The volume of fly ash produced depends on different elements such as the type of power plant, operational methods, type of coal burned, and combustion systems [6].

Generally, fly ash constitutes 10-15% of hard coal and 20-50% of lignite coal burned in thermal power plants. Approximately 75-85% of the fly ash, along with flue gases, exits the boiler, and this ratio may vary from year to year when using different alternative energy production systems. About 25% of the produced fly ash is utilized in various industries, such as construction, agriculture, and chemistry, for recycling purposes, while the remaining 75% is estimated to be disposed of as waste. The construction industry has a principal role in the recycling process of fly ash [7].

In Turkiye, coal-fired thermal power plants play a significant role in electricity production and are prevalent in most regions. These power plants predominantly use low-calorific value lignite coal and to a lesser extent, hard coal as their primary energy source. The ash content in these lignite coals typically ranges from 15% to 35%.

According to data released by the Turkish Statistical Institute, in 2022, thermal power plants in Türkiye produced 27.815548 million tons of waste, of which 10512 tons were hazardous. Of the total waste, 82.6% consisted of ash and slag waste, while 17% consisted of metal, paper, plastic waste, wastewater treatment sludge, and municipal and similar waste. Regarding waste disposal, 87.9% was sent to ash disposal areas/ash dams or controlled landfill sites, 11% was sent to waste management facilities with appropriate licenses and utilized in the backfilling of mines and quarries, and 0.7% was disposed of through other methods [8].

Statistically, a significant portion of the mineral waste produced in thermal power plants is fly ash. In Türkiye, the utilization of fly ash in various sectors and industries has been low. However, in recent years, with the growth of construction industry and the adoption of new cement and concrete standards from Europe, new efforts have emerged to increase the use and valorization of fly ash in the cement and concrete industries.

So far, the Tunçbilek thermal power plant is one of the Turkish very important thermal power plants. The location of Tunçbilek thermal power plant in Turkiye is shown in Figure 1.



Figure 1. Tunçbilek thermal power plant location in Türkiye

Turkiye is one of the leading cement producers in the world, as stated in Table 1. Many cement factories in the country are spread over almost all regions of the country. Considering the location of the-Tunçbilek thermal power plant in the country, it is seen that it is very close to the Marmara Region, where the population density of the country is highest. Fly ash, produced as the waste byproduct of the Tunçbilek thermal power plant, has an important place in terms of reducing environmental damage and sustainable valorization of industrial waste byproducts of the already high net carbon industry.

Some studies have been done in the literature on the Tunçbilek thermal power plant waste fly ash and some studies observing the outcome of using the fly ash on the mechanical properties of Portland cement [9-25]. However, no consensus has been reached regarding the effect of utilizing fly ash in cement and concrete on compressive strength through these studies. In addition to compressive strength parameter, for determining the suitability of cement for use in different construction departments, the porosity at long period, ultrasonic properties of the cement and the correlation between these and comprehensive strength are of great importance, but these parameters and correlations were not taken into account thoroughly in these studies and

When the general scope and experimental details of previous studies are examined, this research stands out by addressing these gaps. In this research, the effect of substituting the Tunçbilek thermal power plant fly ash instead of cement in proportions of 5%, 10%, 15%, 30% and 50% by weight on parameters such as unit weight, 2-, 7- and 28-day compressive strength, ultrasonic properties,150-day porosity of cement, as well as the correlation between these parameters. Furthermore, the fly ash substitution ratios in this study differ from those used in earlier research. Therefore, this study aims to contribute to the development of sustainable materials by filling these gaps in the literature and aid us to figure out how this unwanted industrial by-product affects the performance of construction materials by comprehensively examining the mechanical properties of Portland cement containing fly ash, such as compression strength and related parameters. In doing so, it aims to foster industrial cooperation and facilitate technology transfer, offering new insights into engineering fields such as sustainable material use, waste management, and the enhancement of mechanical performance.

2. MATERIALS AND METHODS

2.1 Raw Materials

Cement holds a significant role in advancing the construction and industrial sectors, which has a noteworthy importance in the development of Turkiye's economy. The same can be said with almost all the other countries of the world. The cement market, which is used as the foundation stone of large-scale construction, infrastructure, and tunnels, especially in Turkiye, is constantly expanding. The continuous increase in demand for cement leads to the employment of thousands of people as facilities to expand to boost cement production.

Additionally, Turkiye is one of the leading cement producers worldwide. Turkiye's prominent position in the international market is due not only to the quantity of its production but also to the quality of its cement. Moreover, the sector significantly contributes to Turkiye's foreign trade volume. In this context, the cement sector makes substantial contributions to Turkiye's industrial development, economic growth, and sustainable construction goals. Cement samples produced in Turkiye are going to be used in this study.

PÇ 42.5 type cement produced in AkçanSA Cement factory, one of the biggest cement manufacturers located in Turkiye, was used in all experiments. The most important feature of PÇ 42.5 type cement is its high strength, making it suitable for structures where strength is the most important requirement, such as bridges and high-rise buildings. Fly ash or other pozzolanic waste materials are not used in the production of this type of cement. So, by adding fly ash to them, we can study its effects on the properties of the cement accurately. The chemical properties of cement, specific gravity, grain density, fineness, and specific surface are presented in Tables 2 and 6.

-	Constituents	Test Result	Unit	Test Method [26]
-	SO3	3.09	%	TS EN
				15309
	A12O3	4.15	%	TS EN
				15309
	CaO	66.48	%	TS EN
				15309
	MgO	1.09	%	TS EN
				15309
	Fe2O3	3.17	%	TS EN
				15309
	Na2O	0.61	%	TS EN
				15309
	SiO2	16.26	%	TS EN
				15309
	SrO	0.066	%	TS EN
				15309
	BaO	< 0.010	%	TS EN
-				15309
Table 2 (continue)	s)			
-	Cr2O3	< 0.010	%	TS EN
				15309
	K2O	0.79	%	TS EN
				15309
	MnO	0.091	%	TS EN
				15309
	P2O5	0.28	%	TS EN
				15309
	TiO2	0.28	%	TS EN
				15309

Table 2. Chemical analysis of PC 42.5 Portland cement

In this study, there were no changes in the type or granulometry of the aggregate. CEN standard sand, selected according to TS EN 196-1, was supplied by the LIMAK Trakya Cement Factory, which also conducted particle size analysis and measured the moisture content of the sand (Table 3). CEN reference sand is a siliceous, standard sand composed of rounded particles with at least 98% silica content. Therefore, no chemical analysis was performed on the sand [29]. The specific gravity of the sand is shown in Table 3.

Square Mesh Size (mm)	Test Results (%)	Moisture (%)
2,00 mm	0.00	
1,6 mm	7.80	
1,00 mm	32.76	0.10
0,05 mm	66.32	0.10
0,16 mm	87.54	
0,08 mm	98.98	

Table 3. CEN standard sand grain size distribution and moisture

In the experiments, fly ash, a byproduct of the Tunçbilek thermal power plant, was used. This fly ash is classified as Class F according to ASTM C 618 (Table 4). The chemical properties and class of the fly ash are shown in Table 5, while grain density and fineness tests were conducted and summarized in Table 6.

 Table 4. Tunçbilek fly ash classification [35]

Fly ash	S+A+F (%)	CaO (%)	Class
Tunçbilek uçucu	82.27	<10	F (Düşük Ca)
kül			

 Table 5. Chemical composition of Tunçbilek fly ash [34]

Oxide (%)	Tunçbilek fly ash
SiO2	54.79
A12O3	18.58
Fe2O3	8.90
CaO	2.81
MgO	5.90
K2O	1.65
Na2O	0.21
TiO2	0.93
P2O5	0.23
MnO	0.15
Cr2O3	ND
LOI	2.88

Material	Parameter	Test Result	Unit	Test Method
	Specific gravity (SGR 03)	2.69	g/cm3	Pycnometric Density Pulp
	Grain density	3.10	g/cm3	TS EN 196-6: 2020 [27]
PO 42.5 Portland	Fineness determination Sieve residue (90 µm)	0.5	%	TS EN 450-1: 2015 [31]
cement	Specific surface: Determination of fineness-Air permeability (Blaine method)	3270	cm2/ g	TS EN 196-6: 2020 [27]
CEN Standard sand	Specific gravity (SGR 03)	2.29	g/cm3	Pycnometric Density Pulp
Tunçbilek	Grain density	2.20	g/cm3	TS EN 196-6: 2020 [27]
fly ash	Fineness, Wet sieving method (45 Micron)	19	%	TS EN 450-1: 2015 [31]

Table 6. PC 42.5 Portland cement specific gravity, particle density, fineness, and specific surface, CEN standard sand specific gravity values, and Tunçbilek fly ash grain density and fineness

For these tests, city water (tap water) has been used to produce samples.

2.2 Preparation and Testing of Cement Samples

In this study, 30 samples were produced in the laboratory. The amount of cement was calculated according to TS EN 196-1, and 0%, 5%, 10%, 15%, 30%, and 50% of Portland cement was replaced with an equivalent weight of fly ash. Water and sand proportions remained constant in compliance with TS EN 196-1 standards [29]. The classification of the samples produced, their mixture ratios, and sizes are shown in Table 7.

In the reference process, the mortar prepared by mechanical mixing was compressed in the mold using a standard shaking machine. The samples in the mold were kept in a humid atmosphere for 24 hours, afterwards, they are pulled out from the mold were held inside water until the specified tests were carried out.

In the experiments, prismatic samples were produced in two different sizes (40 mm x 40 mm x 160 mm and 100 mm x 100 mm) and samples with dimensions of 100x100x100 mm were produced for the ultrasound velocity experiment [32].

Sample Name	% Cement + %Fly ash (% by weight) (gr)	Sand (Fixed amount by weight) (gr)	Water (Fixed amount by weight) (gr)	Sample Size (mm)	
CUKD0	% 100 Cement (150 gr) + % 0 Fly ash	450	75	40x40x160	
CUKD5	% 95 Cement (142.5 gr) + % 5 Fly ash (7.5 gr)	450	75	40x40x160	
CUKD10	% 90 Cement (140 gr) + % 10 Fly ash (10 gr)	450	75	40x40x160	
CUKD15	% 85 Cement (127.5 gr) + % 15 Fly ash (22.5 gr)	450	75	40x40x160	
CUKD30	% 70 Cement (105 gr) + % 30 Fly ash (45 gr)	450	75	40x40x160	
CUKD50	% 50 Cement (75 gr) + % 50 Fly ash (75 gr)	450	75	40x40x160	
CUKK0	% 100 Cement (586 gr) + % 0 Fly ash	1758	293	100x100x100	
CUKK5	% 95 Cement (556.7 gr) + % 5 Fly ash (29.3 gr)	1758	293	100x100x100	
CUKK10	% 90 Cement (527.4 gr) + % 10 Fly ash (58.6 gr)	1758	293	100x100x100	
CUKK15	% 85 Cement (498 gr) + % 15 Fly ash (88 gr)	1758	293	100x100x100	
CUKK30	% 70 Cement (410.2 gr) + % 30 Fly ash (175.8 gr)	1758	293	100x100x100	
Table 7 (continues)					
CUKK50	% 50 Cement (293 gr) + % 50 Fly ash (293 gr)	1758	293	100x100x100	

Table 7. Nomenclature of produced samples, mixture ratios, and sizes

3. RESULTS AND DISCUSSION

3.1 Fresh Mortar Test

Unit Weight Test [28]:

A unit weight test was conducted on the fresh mortar. Since the unit weight of fresh mortar is not dependent on size, the test was applied to a cast mortar with dimensions of $100 \times 100 \times 100$ mm. The results of the unit weight test are shown in Table 8.

Sample Name	Unit Weight (N/m3)
CUKK0	21378.5
CUKK5	21143.13
CUKK10	21035.26
CUKK15	20996.03
CUKK30	20917.58
CUKK50	20790.09

Table 8. Fresh mortar unit weight test results

In the study involving weight substitution between cement and fly ash, the lower specific gravity of fly ash leads to an increase in the volume of the binding material. As the volume of the binder increases, there's a decrease in the unit weight of the fresh concrete mix. The correlation between unit weight and fly ash content can be seen in Figure 2. As illustrated, while the use of fly ash increases, the unit weight decreases.



Figure 2. Correlation between unit weight and fly ash ratio

3.2. Hardened Concrete Test

Compression Strength Test:

The compression strength values of the samples were determined using the UTEST Compression Testing machine. In Table 9 the averages of the experiment results for all specimens are presented. Also, the percentages in parentheses, which are listed vertically beside the compression strength test results in this table, assume that the compression strength of the sample without fly ash (control concrete) is 100%. The other variables in the column are evaluated according to the percentage ratios of the test results of this sample. In Table 10, the test results for all samples on the 28th day are assumed to be 100 MPa, and the percentage difference created by the time each sample was kept until testing is investigated for each test result.

Sample	2 Day	7 Day Average	28 Day Average
Name	Average	Results (MPa)	Results (MPa)
	Results		
	(MPa)		
CUKK0	32.1 (100%)	41.4 (100%)	53.0 (100%)
CUKK5	28.9 (90%)	38.4 (92%)	50.3 (95%)
CUKK1	25.4 (79%)	36.0 (87%)	48.9 (92%)
0			
CUKK1	23.6 (73%)	33.0 (80%)	47.1 (89%)
5			
CUKK3	15.7 (49%)	27.4 (66%)	40.9 (77%)
0			
CUKK5	9.5 (29%)	17.3 (41%)	27.9 (53%)
0			

Table 9. Average results of compression strength experiments conducted on all specimens and the comparison of the percentage values of compression in comparison with the control concrete sample (*)

*The table displays the average values of 18 specimens.

Table 10. Comparison of the percentage values of compression strength obtained from the test results with obtained from the test results with respect to the 28-day concrete (*)

Sample	2 Day	7 Day Average	28 Day Average						
Name	Average	Results (MPa)	Results (MPa)						
Indiffe	Posulta	Results (Ivil a)	Results (Ivil a)						
	(MD _a)								
	(MPa)								
CUKK0	60	78	100						
	Table 10 (continues)								
		. ,							
CUKK5	57	76	100						
CUKK1	52	73	100						
0									
CUKK1	50	70	100						
5	20	10	100						
5	38	67	100						
CUKK3									
0									
	35	62	100						
CUKK5									
0									

*The table displays the average values of 18 specimens.

The interpretation of the Table 9 results reveals a negative correlation between fly ash to concrete percentage in the sample and the compression strength in concrete. Additionally, as the fly ash ratio increases, it positively correlates with proportionally higher compression strength results in concrete samples cured for an extended period. The reduction in compression is linked to a reduction in the ratio of fly ash mass increase, resulting from a reduction in cement content. Despite the expected decrease in strength due to the reduced cement, the faster hydration of lime in the cement content plays a contributing role. The pozzolanic reaction of fly ash taking longer than lime results in a decrease in lime content as the fly ash ratio increases. Consequently, samples with higher fly ash ratios, exposed to the same conditions for the same duration, exhibit lower compression strengths compared to the 28-day concrete.

In Table 10, the values of compression strength test results on the 28th day are assumed to be 100 MPa for each column, and the averages of strengths on the 2nd and 7th days are processed as percentage ratios. Considering the findings on the table, it can be seen as the curing days increase homogeneously, the strength values in the test results also increase. Aside from this, as the fly ash ratio increases, a greater percentage increase in compression strengths with more days of curing in comparison with the samples with smaller fly ash content is inspected. The reason for this is anticipated to be because the hydration time of fly ash is longer than that of cement. In samples where cement is relatively less, it can be interpreted that to achieve maximum strength efficiency in concrete with higher fly ash content, it will need to be cured for a longer period due to the lower hydration and lime transformation capabilities of the contents of cement. The correlations between compression strength, curing days, fly ash content, and unit weight are illustrated in Figures 3 to 5.



Figure 3. Average compression strengths of samples for various curing times



Figure 4. Correlation between average compressive strength and fly ash ratio at different curing times



Figure 5. Correlation between unit weight and average 28th-day compression strength

Ultrasonic Velocity Test:

According to the TS EN 12504-4 standard [32], Ultrasonic velocity tests were conducted on six cubeshaped samples with dimensions of 100x100x100 mm after 28 days of production. Proceq Pundit Lab+UTC-3050 Ultrasonic Wave Velocity Testing Device was used for measurements. The findings of the tests conducted on all samples can be seen below in Table 11.

Table 11. S	amples of	ultrasonic	velocity t	est results
-------------	-----------	------------	------------	-------------

Sample Name	Ultrasonic Velocity m/s			
CUKK0	4367			
CUKK5	4365			
CUKK10	4348			
CUKK15	4292			
CUKK30	4202			
CUKK50	4032			

Based on the percentage of fly ash, it has been examined over time that samples with lower void content allow ultrasonic waves to pass more quickly. An increase in ultrasonic velocity is positively correlated with higher compressive strength and the favorable effect of fly ash on void content in humid surroundings [34, 36].

Table 11 shows that concrete with fly ash has lower ultrasonic velocity values at 28 days compared to control mortar (0% fly ash). This suggests that samples with high fly ash content may not be as durable under environmental conditions at early ages. It can be stated that high fly ash content negatively affects the ultrasonic velocity values of hardened mortar.

Upon examining the ultrasonic tests in previous studies, it is observed that the ultrasonic velocity of fly ash samples increases with age. This is caused by the pozzolanic reaction slowing down the occurrence of lime [34, 36].

It has been seen that fly ash has a positive effect on void content in moist environments in advancing ages. When considering that lime is released as a result of cement hydration and then dissolves in water, leaving voids in the concrete, the reduction in void content associated with lime can be explained during the pozzolanic reaction of fly ashes. The positive effect of fly ashes on void content can be related to this way and considering that void content is closely related to durability under environmental conditions, it can be anticipated that the utilization of fly ash in small percentages is expected to have a positive impact on durability. The correlations between ultrasonic velocity and fly ash ratio, unit weight, and compression strength, are to be found in the figures below (6-7-8).



Figure 6. Correlation between ultrasonic velocity and fly ash ratio



Figure 7. Correlation between ultrasonic velocity and unit weight



Figure 8. Correlation between ultrasonic velocity and average compression strength at the 28th day

Porosity Test:

According to the TS 9179 standard [33], porosity tests were conducted on six samples with dimensions of 160x40x4 mm after 150 days of curing. For this test, the samples in the mold were kept in a humid setting for 24 hours, and then the samples pulled out of the mold were kept in water for 28 days. Again, these samples have been taken out of the water and stored in a normal environment for 132 days. The Auto Pore Test Device was used for the measurements. Table 12 demonstrates the findings of the tests conducted on all samples.

Table 12.	Sample	porosity	test results
-----------	--------	----------	--------------

Sample Name	Total Intrusion Volume (mL/g)	Total Pore Area (m²/g)	Median Pore Diameter (Volume) (nm)	Median Pore Diameter (Area) (nm)	Average Pore Diameter (4V/A) (nm)	Bulk Density at 0.52 psia (g/mL)	Apparent (skeletal) Density (g/mL)	Porosity (%)	Stem Volume Used (%)
CUKK0	0.0664	5.825	102.3	14.7	45.6	2.1448	2.5008	14.2340	39
CUKK5	0.0679	7.019	101.0	8	38.7	2.1351	2.4973	14.5021	38
CUKK10	0.0809	8.610	95.8	8.9	37.6	2.0704	2.4871	16.7552	48
CUKK15	0.0854	8.984	188.4	6.6	38	2.0632	2.5046	17.6223	48
CUKK30	0.0905	10.559	243.3	5.9	34.3	2.0441	2.5083	18.5075	50
CUKK50	0.1005	10.481	380.7	5.4	38.4	2.0113	2.5211	20.2217	53

Hardened concrete's crucial characteristics are tied to pores and hydration products, influencing strength, shrinkage, swelling, and permeability. Examining concrete's pore structure and mineral composition is beneficial.

Fresh concrete pores are filled with water or gas. In initial mixing, water separates cement particles and gravel, creating water-filled spaces. This space allows for cement hydration product formation. As hydration advances, the void volume, larger than the original unhydrated cement, decreases as hydration products form a continuous matrix, binding residual cement particles in time. The original water-filled area not covered by hydration products contributes to the cement paste's pore system, typically with the
biggest pores. Hydration decreases the dimensions and volume of capillary voids; if small, the gel's bulk volume eventually fills the void, producing a paste without capillary voids [36].

The volume of water-filled voids in fresh cement paste must surpass the total volume of cement or leave a portion of the original cement without water. Consequently, even after complete cement hydration, hydration products are insufficient to occupy the original water space, resulting in pastes with relatively large capillary pores.

Researches on hardened cement pastes reveal that the size of the largest pores is primarily related to the level of moisture. For young cement pastes with minimal hydration, the size is approximately 1 micrometer, decreasing to around 0.1 micrometers in fully hydrated mature pastes [34, 36]. During ongoing curing, as voids are filled with hydration products, most capillary voids become isolated, and separated by the gel, provided the initial water/cement ratio is sufficiently low. This gel is primarily calcium silicate hydrate with a non-solid part, forming a porous solid [34, 36].

In compliance with the findings in Table 12, as the volume of fly ash added instead of cement increases, Total Pore Area, Total Intrusion Volume, Average Pore Diameter (4V/A), Median Pore Diameter (Volume) Visible (skeletal) Density, and Porosity increase, while at 0.52 psia Median Pore Diameter (Area) and Mass Density decrease.

The correlations between Porosity, fly ash ratio, unit weight, compression strength on the 28th day, and ultrasonic velocity can be observed in the Figures below (9 to 13).



Figure 9. Correlation between porosity and fly ash ratio







Figure 11. Correlation between porosity and compression strength on the 28th day



Figure 12. Correlation between porosity and ultrasonic velocity



Figure 13. Correlation between porosity, average compression strength on the 28th day, and ultrasonic velocity

In the preceding part, it was explained that the pozzolanic reaction of fly ash increases with age because of delays in lime formation. Fly ash positively influences void content, vital for durability under environmental conditions, and curing time significantly affects porosity. Therefore, to simulate real construction environments and obtain more precise results, the samples were cured at 25 degrees Celsius outside water under normal conditions for 132 days more, and then porosity tests were performed.

The results, after 150 days of curing and hydration, compared to the control mortar (0% Fly ash), indicate that porosity increases with higher fly ash ratios. This shows that samples with high fly ash

content may be less durable and mechanically strong under environmental conditions, as elevated fly ash content adversely affects porosity values in hardened mortar.

Conversely, using fly ash in low percentages is deemed beneficial for compression strength and durability, so moderation in fly ash content is of great importance.

4. CONCLUSION

This study reveals the following conclusions regarding the substitution of cement with Tunçbilek fly ash:

- According to the relation between specific gravity and volume, the lower specific gravity of fly ash leads to an increased volume of the binding material, and the result is a lower unit weight in the fresh concrete mix as the binder's volume expands.
- A negative correlation exists between the fly ash-to-cement ratio and compressive strength in concrete samples cured for short periods. The reduction in compressive strength is attributed to the lower cement content as fly ash replaces cement. Despite this, the faster hydration of lime in the cement contributes to early strength. Samples with higher fly ash content exhibit greater percentage increases in compressive strength with extended curing, emphasizing the need for longer curing periods due to the slower hydration of fly ash compared to cement.
- Ultrasonic Velocity tests indicated that the examines with lower void content allow ultrasonic waves to pass more quickly. The increase in ultrasonic velocity positively correlates with higher compressive strength and the favorable effect of fly ash on void content in a moist environment and concrete with high fly ash content shows lower ultrasonic velocity values at 28 days, suggesting potential durability concerns in certain surrounding conditions, especially at early ages.
- In this study the crucial role of pore structure and hydration products in influencing concrete properties such as strength is emphasized. Generally, fly ash has a positive effect on void content in a moist environment, reducing voids associated with lime during the pozzolanic reaction of fly ashes over an extended period. According to the 28th-day compressive strength test results, the samples with 5%, 10%, and 15% fly ash are between standard values. Therefore, it can be inferred that the valorization of fly ash at low percentages has the potential to further improve the compressive strength within a longer period, signifying its improving effect on concrete properties.
- Curing time significantly impacts porosity, and samples cured for 150 days in this study demonstrate increased porosity with higher fly ash ratios, suggesting potential durability concerns.
- In conclusion, based on the comprehensive results acquired with this research, utilizing low percentages of fly ash is deemed advantageous for enhancing concrete properties. This underscores the significance of maintaining moderation in fly ash content to achieve optimal performance in real-world construction applications.

REFERENCES

- Schlorke S., Li T., Stec M., Mallagray J.V., Kaleem H., 2020, The Impact of COVID-19 on the Cement Industry International, Finance Corporation (IFC), https://www.ifc.org/wps/wcm/connect/c015a cbf-8465-4f8e-95e8-857511f10bbb/202008-COVID-19-impact-on-cementindustry.pdf?MOD=AJPERES&CVID=ngx QLJQ, [accessed 12 March 2021].
- Garside M., 2021, Major Countries in Worldwide Cement Production 2010-2020,https://www.statista.com/statistics/267 364/world-cement-production-by-country/, [accessed 5 May 2021].
- UN-Habitat, 2023, The Challenge, https://unhabitat.org/topic/housing #:~:text=By%202030%2C%20UN%2DHabi tat%20estimates,accessible%20housing%20 units%20every%20day, [accessed 5 April 2023].
- Yetiş Ü., 2017, Republic of Türkiye, Ministry of Environment, Urbanization and Climate Change, Department of European Union Investments, Technical Assistance Project to Strengthen the Capacity of the Ministry of Environment and Urbanization in the Field of EIA,

http://www.hlccevre.com/images/PDF/sektor el-kilavuzlar/g19-cimento-fabrikalar.pdf, [accessed 3 March 2021].

- World Coal Association, 2012, Coal Energy for Sustainable Development, UK, https://sustainabledevelopment.un.org/getW SDoc.php ?id=996, [accessed 4 June 2023].
- Elmas S., 2020, The Effect of Thermal Power Plant Fly Ash in Granite Body On Microstructure And Technical Properties, Journal of Scientific Perspectives, Volume 4, Issue 2, pp. 147-156 E - ISSN: 2587-3008,

URL: https://ratingacademy .com.tr/ojs/index.php/jsp,DOİ: https://doi.org/10.26900/jsp.4.012.

- Akgül Ç.M., Yener A.P., Bayramtan M., 2018, Life Cycle Analysis for Fly Ash Concretes in Turkey, Istanbul Bulten, no.148, pp. 4-12.
- Turkish Statistical Institute, 2022, Waste Statistics, https://data.tuik.gov.tr/Bulten/Index?p=Atik-Istatistikleri-2022-49570, [accessed 3 December 2023].
- Yetgin Ş., Çavdar A., 2005, The effects of trass addition ratio on strength, setting time and soundness properties of trass-cement, Fırat University, Journal of Science and Engineering Sciences, Volume: 17, Issue: 4, pp.687-692.
- Biricik, H., 1999, Reducing Water Permeability with Pozzolan Material, Yıldız Technical University, Istanbul, Turkey.
- Özturan T., 1991, Effectiveness of Mineral Additives in High Strength Concrete Production, Boğaziçi University, Istanbul, Turkey.
- Yeğinobali A, Ertün T., 2011, Standards and Mineral Additives in Cement, Turkish Cement and Cement Products Assembly (TÇMB / AR-GE), Ankara, Turkey.
- American Coal Ash Association,2003, Fly Ash Facts for Highway Engineers. Federal Highway Administration (U.S. Department of Transportation), https://www.fhwa.dot.go v/pavement/recycling/fach01 .cfm, [accessed 2 April 2021].
- 14. Dwivedi A., Kumar Jain M., 2014, Fly Ash Waste Management and Overview: A Review. Recent Research in Science and Technology, 6(1): 30-35 ISSN: 2076-5061,

http://recent-science.com, [accessed 13 June 2021].

- 15. Güler G, Güler E, İpekoğlu Ü, Mordoğan H, 2005, Properties and Usage Areas of Fly Ashes, Turkey 19th International Mining Congress and Fair, IMCET 2005, Dokuz Eylül University, İzmir, Turkey.
- Ghazali N., Muthusamy K., Wan Ahmad S., 2019, Utilization of Fly Ash in Construction, IOP Conference Series: Materials Science and Engineering, 601 (2019) 012023, doi:10.1088/1757-899X/601/1/012023.
- Özcan U, Güngör S., 2019, A Sustainable Method / The Use of Pozzolan In Concrete, European Journal of Science and Technology, No. 15, pp. 176-182.
- 18. Bhatta A, Priyadarshinia b S., Acharath Mohanakrishnana A., Abria A., Sattlera M., Techapaphawit S., 2019, Physical, Chemical, and Geotechnical Properties of Coal Fly Ash: A Global Review. Case Study in Construction Materials, Volume 11, e00263.
- 19. Leloğlu S., 2020, Coal and Energy Report 2020, TMMOB Chamber of Mining Engineers, Annual report, https://enerji.mmo.org.tr/wpcontent/uploads/2020/09/MADEN-M.O-K%C3%96M%C3%9CR- VR-ENERJ%C4%B0-RAPORU-2020.pdf, [accessed 12 Mart 2021].
- 20. Yao Z. T, Ji X.S, Sarker P.K, Tang J.H, Xia M.SXi ,Y.Q., 2015, A Comprehensive Review on the Applications of Coal Fly Ash, Earth-Science Reviews, Volume 141, Pages 105-121.
- Önal G., Doğan Z., Yüce H., Coal Consumption-Thermal Power Plants, Proceedings of the 8th Coal Congress of Turkey,

https://www.maden.org.tr/resimler/ekler/773 75f945f272a2_ek.pdf, [accessed 18 April 2021].

- 22. Karayigit A.I., Celik Y., 2010, Mineral Matter and Trace Elements in Miocene Coals of the Tuncbilek-Domanic Basin, Kütahya, Turkey.
- 23. Tunçbilek Thermal Power Plant, https://www.enerjiatlasi.com /komur/tuncbilek-termik-santrali.htm, [accessed 30 August 2022].
- 24. Yılmaz A., Energy Atlas, https://www.enerjiatlasi.com/komur/ tuncbilek-termik-santrali.html, [accessed 23 November 2022].
- 25. Güner H.T., 2019, Early Miocene palaeoclimatic reconstruction of Tunçbilek basin, Turkish Forestry Magazine, 2019, 20(2): 93-100 https://dergipark.org.tr/tr/download/article-file/753528 [accessed 24 November 2022].
- Technical Board, 2008, Characterization of waste and soil - Determination of elemental composition by X-ray fluorescence, TS EN 15309, Turkish Standard institution, Turkey.
- 27. Technical Board, 2020, Methods of testing cement Part 6: Determination of fineness, TS EN 196-6, Turkish Standard institution, Turkey.
- Construction Technical Committee, 2019, Testing fresh concrete - Part 6: Density, TS EN 12350-6, Turkish Standard institution, Turkey.
- Technical Board, 2016, Methods of testing cement - Part 1: Determination of strength, TS EN 196-1, Turkish Standard institution, Turkey.
- Technical Board, 2012, Cement- Part 1: Compositions and conformity criteria for common cements, TS EN 197-1, Turkish Standard institution, Turkey.
- Construction Materials Technical Committee, 2015, Fly ash for concrete - Part 1: Definition, specifications and conformity criteria, TS EN 450-1, Turkish Standard institution, Turkey.

- 32. Technical Committee, 2021, Testing concretePart 4: Determination of ultrasonic pulse velocity, TS EN 12504-4, Turkish Standard institution, Turkey.
- Petroleum Specialization Group, 2015, Determination of pore volume distribution of catalysts- Mercury instrusion porosimetry method, TS 9179, Turkish Standard institution, Turkey.
- 34. Aydaşgil A., 2003, Use of Tunçbilek Thermal Energy Plant's fly ashes in stoneware body and glazes, Master's Thesis, Anadolu of University Institute Science and Technology, Department of Ceramic Engineering Supervisor: Assoc. Prof. Bekir KARASU 2003, 61 pages.
- 35. Kursun I., Eskibalci M. F., Terzi M., Ozdemir O., 2019, Investigation of the Relationship between Electrokinetic Properties and Classification of Coal Fly Ashes, IMPC Eurasia Conference, Antalya, Turkey.
- 36. Hsin Hung H., 1997, Properties of High-Volume Fly Ash Concrete, PHD's Thesis, Sheffield University, Department of Mechanical Engineering, 241 pages.

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

The Heat Problem with Non-Local Boundary Conditions

Research Article

İrem Bağlan 🗓

Kocaeli University, Faculty of Arts and Sciences, Department of Mathematics, Kocaeli, Türkiye

Author E-mail: isakinc@kocaeli.edu.tr İ. Bağlan ORCID ID: 0000-0002-1877-9791

*Correspondence to: İrem Bağlan, Kocaeli University, Faculty of Arts and Sciences, Department of Mathematics, Kocaeli, Türkiye DOI: 10.38061/idunas.1566513

Received: 13.10.2024; Accepted: 12.11.2024

Abstract

In this article, the two-dimensional inverse nonlinear parabolic problem is discussed. The most important feature of the problem is its solution with the Fourier approach. The solution was obtained by Fourier implicit and iteration methods [1]-[6].

Keywords: Heat problem, Fourier iterative method, nonlocal conditions, implicit finite-difference methods.

1. INTRODUCTION

The inverse two-dimensional parabolic problem finds applications in many fields, including diffusion applications, heat transfer, population, medicine, electrochemistry, engineering, chemistry, plasma physics.

2. MATERIALS AND METHODS

To solve this inverse problem, a combination of the Fourier method, Picard successive approximations, and the finite difference method was employed.

3. THE EXISTENCE OF SOLUTIONS

$$\frac{\partial \upsilon}{\partial \tau} = \frac{\partial^2 \upsilon}{\partial \xi^2} + \sigma(\tau) \frac{\partial^2 \upsilon}{\partial \eta^2} + \theta(\xi, \eta, \tau, \upsilon), \tag{1}$$

$$\upsilon(\xi,\eta,0) = \phi(\xi,\eta), \xi \in [0,\pi], \eta \in [0,\pi]$$
⁽²⁾

$$\begin{aligned}
\upsilon(0,\eta,\tau) &= \upsilon(\pi,\eta,\tau), \eta \in [0,\pi], \tau \in [0,T] \\
\upsilon(\xi,0,\tau) &= \upsilon(\xi,\pi,\tau), \xi \in [0,\pi], \tau \in [0,T] \\
\upsilon_{\xi}(0,\eta,\tau) &= \upsilon_{\xi}(\pi,\eta,\tau), \eta \in [0,\pi], \tau \in [0,T] \\
\upsilon_{\eta}(\xi,0,\tau) &= \upsilon_{\eta}(\xi,\pi,\tau), \xi \in [0,\pi], \tau \in [0,T] \\
\varsigma(t) &= \int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \nu(\xi,\eta,\tau) d\xi d\eta, \tau \in [0,T],
\end{aligned}$$
(3)

Here, (1) represents the inverse coefficient problem, (2)-(3) define the initial and periodic boundary conditions of the problem [8] and (4) is the integral condition for the inverse coefficient [7].

The following solution is obtained with the Fourier Method:

$$\begin{split} \upsilon(\xi,\eta,\tau) &= \frac{1}{4} \left(\phi_0 + \frac{4}{\pi^2} \int_0^t \theta_0(\tau,\upsilon) d\tau \right) \\ &+ \sum_{m,n=1}^{\infty} \left(\phi_{emn} + \frac{4}{\pi^2} \int_0^t e^{-\int_{\tau}^t \left[(2m)^2 + \sigma(\iota)(2n)^2 \right] d\iota} \theta_{emn}(\tau,\upsilon) d\tau \right) \\ &- \cos(2m\xi) \cos(2n\eta) \\ &+ \sum_{m,n=1}^{\infty} \left(\phi_{esmn} + \frac{4}{\pi^2} \int_0^t e^{-\int_{\tau}^t \left[(2m)^2 + \sigma(\iota)(2n)^2 \right] d\iota} \theta_{esmn}(\tau,\upsilon) d\tau \right) \\ &- \cos(2m\xi) \sin(2n\eta) \\ &+ \sum_{m,n=1}^{\infty} \left(\phi_{semn} + \frac{4}{\pi^2} \int_0^t e^{-\int_{\tau}^t \left[(2m)^2 + \sigma(\iota)(2n)^2 \right] d\iota} \theta_{semn}(\tau,\upsilon) d\tau \right) \\ &- \sin(2m\xi) \cos(2n\upsilon) \\ &+ \sum_{m,n=1}^{\infty} \left(\phi_{smn} + \frac{4}{\pi^2} \int_0^t e^{-\int_{\tau}^t \left[(2m)^2 + \sigma(\iota)(2n)^2 \right] d\iota} \theta_{semn}(\tau,\upsilon) d\tau \right) \\ &- \sin(2m\xi) \cos(2n\upsilon) \\ &+ \sum_{m,n=1}^{\infty} \left(\phi_{smn} + \frac{4}{\pi^2} \int_0^t e^{-\int_{\tau}^t \left[(2m)^2 + \sigma(\iota)(2n)^2 \right] d\iota} \theta_{smn}(\tau,\upsilon) d\tau \right) \\ &- \sin(2m\xi) \sin(2n\eta) \end{split}$$

Here,

$$\begin{split} \phi_{0} &= \upsilon_{0}(0), \\ \phi_{cmn} &= \upsilon_{cmn}(0)e^{-\int_{0}^{t} \left[(2m)^{2} + \sigma(\iota)(2n)^{2}\right]d\iota}, \\ \phi_{csmn} &= \upsilon_{csmn}(0)e^{-\int_{0}^{t} \left[(2m)^{2} + \sigma(\iota)(2n)^{2}\right]d\iota}, \\ \phi_{scmn} &= \upsilon_{scmn}(0)e^{-\int_{0}^{t} \left[(2m)^{2} + \sigma(\iota)(2n)^{2}\right]d\iota}, \\ \phi_{smn} &= \upsilon_{smn}(0)e^{-\int_{0}^{t} \left[(2m)^{2} + \sigma(\iota)(2n)^{2}\right]d\iota}. \end{split}$$

Let the following rules apply:

(C1)
$$\varsigma(t) \in C^{1}[0,T]$$

 $\phi(\xi,\eta) \in C^{1,1}([0,\pi] \times [0,\pi]),$
(C2) $\phi(0,\eta) = \phi(\pi,\beta), \phi_{\xi}(0,\eta) = \phi_{\xi}(\pi,\eta), \int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \phi(\xi,\eta) d\xi d\eta = \varsigma(0),$
 $\phi(\xi,0) = \phi(\xi,\pi), \phi_{\eta}(\xi,0) = \phi_{\eta}(\xi,\pi),$
(C3) (1) Let $\theta(\xi,\eta,\tau,\upsilon)$ have the following properties:

$$\begin{split} & \left| \frac{\partial \theta(\xi,\eta,\tau,\upsilon)}{\partial \xi} - \frac{\partial \theta(\xi,\eta,\tau,\overline{\upsilon})}{\partial \xi} \right| \leq \chi(\xi,\eta,\tau) \left| \upsilon - \overline{\upsilon} \right|, \\ & \left| \frac{\partial \theta(\xi,\eta,\tau,\upsilon)}{\partial \eta} - \frac{\partial \theta(\xi,\eta,\tau,\overline{\upsilon})}{\partial \eta} \right| \leq \chi(\xi,\eta,\tau) \left| \upsilon - \overline{\upsilon} \right|, \\ & \left| \frac{\partial \theta(\xi,\eta,\tau,\upsilon)}{\partial \eta \partial \xi} - \frac{\partial \theta(\xi,\eta,\tau,\overline{\upsilon})}{\partial \xi \partial \eta} \right| \leq \chi(\xi,\eta,\tau) \left| \upsilon - \overline{\upsilon} \right|, \\ & \text{where } \chi(\xi,\eta,\tau) \in L_2(D), \chi(\xi,\eta,\tau) \geq 0. \end{split}$$

(2)
$$\theta(\xi, \eta, \tau, \upsilon) \in C^{2,2,0}[0, \pi], \tau \in 0, T],$$

$$\int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \upsilon_{t}(\xi, \eta, \tau) d\xi d\eta = \varsigma'(\tau), 0 \le t \le T.$$

$$\sigma(\tau) = \frac{\varsigma'(\tau) - \int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \theta(\xi, \eta, \tau, \upsilon) d\xi d\eta - \frac{\pi^{3}}{2} \upsilon_{\eta}(\pi, \tau)}{\frac{\pi^{3}}{2} \upsilon_{\xi}(\pi, t)}.$$

Definition 1.

$$\{v(\tau)\} = \{v_0(\tau), v_{c\alpha\beta}(\tau), v_{cs\alpha\beta}(\tau), v_{sc\alpha\beta}(\tau), v_{s\alpha\beta}(\tau)\} \text{ of continuous functions on } [0, T].$$

Banach norm:

$$\max_{0 \le t \le T} \frac{|\upsilon_0(\tau)|}{4} + \sum_{\alpha,\beta=1}^{\infty} \left(\frac{\max_{0 \le t \le T} |\upsilon_{c\alpha\beta}(\tau)| + \max_{0 \le t \le T} |\upsilon_{cs\alpha\beta}(\tau)|}{+ \max_{0 \le t \le T} |\upsilon_{s\alpha\beta}(\tau)| + \max_{0 \le t \le T} |\upsilon_{s\alpha\beta}(\tau)|} \right) < \infty$$

$$\begin{aligned} \left\| \upsilon(\tau) \right\| &= \max_{0 \le t \le T} \frac{\left| \upsilon_0(\tau) \right|}{4} \\ &+ \sum_{\alpha,\beta=1}^{\infty} \left(\max_{0 \le t \le T} \left| \upsilon_{c\alpha\beta}(\tau) \right| + \max_{0 \le t \le T} \left| \upsilon_{c\alpha\beta}(\tau) \right| \right) \\ &+ \max_{0 \le t \le T} \left| \upsilon_{s\alpha\beta}(\tau) \right| + \max_{0 \le t \le T} \left| \upsilon_{s\alpha\beta}(\tau) \right| \right). \end{aligned}$$

Theorem 1. The problem has a solution under conditions (C1) -(C3).

Proof. Let's iterate the coefficients:

$$\begin{split} \upsilon_{0}^{(N+1)}(\tau) &= \phi_{0} + \frac{4}{\pi^{2}} \int_{0}^{t} \theta_{0}(\tau, \upsilon) d\tau \\ \upsilon_{cmn}^{(N+1)}(\tau) &= \phi_{cmn} + \frac{4}{\pi^{2}} \int_{0}^{t} e^{-\int_{\tau}^{t} [(2m)^{2} + \sigma(\iota)(2n)^{2}] d\iota} \theta_{cmn}(\tau, \upsilon) d\tau \\ \upsilon_{cmn}^{(N+1)}(\tau) &= \phi_{cmn} + \frac{4}{\pi^{2}} \int_{0}^{t} e^{-\int_{\tau}^{t} [(2m)^{2} + \sigma(\iota)(2n)^{2}] d\iota} \theta_{csmn}(\tau, \upsilon) d\tau \\ \upsilon_{scmn}^{(N+1)} &= \phi_{scmn} + \frac{4}{\pi^{2}} \int_{0}^{t} e^{-\int_{\tau}^{t} [(2m)^{2} + \sigma(\iota)(2n)^{2}] d\iota} \theta_{scmn}(\tau, \upsilon) d\tau \\ \upsilon_{smn}^{(N+1)} &= \phi_{smn} + \frac{4}{\pi^{2}} \int_{0}^{t} e^{-\int_{\tau}^{t} [(2m)^{2} + \sigma(\iota)(2n)^{2}] d\iota} \theta_{scmn}(\tau, \upsilon) d\tau \end{split}$$

According to the assumptions, we get $v_0^{(0)}(\tau) \in B$, $t \in [0, T]$. To find the approximation principle from 1 to N, Cauchy, Hölder, Bessel inequalities and Lipschitzs condition were employed, respectively. These tools are utilized to demonstrate the existence of the solution. Finally, we arrive at:

$$\begin{split} \left\| \upsilon^{(N+1)}(\tau) \right\| &= \max_{0 \le \tau \le T} \frac{\left\| \upsilon^{(N)}_{_{0}}(\tau) \right\|}{4} + \\ &\sum_{m,n=1}^{\infty} \max_{0 \le \tau \le T} \left\| \upsilon^{(N)}_{_{mn}}(\tau) \right\| + \max_{0 \le \tau \le T} \left\| \upsilon^{(N)}_{_{smn}}(\tau) \right\| + \max_{0 \le \tau \le T} \left\| \upsilon^{(N)}_{_{smn}}(\tau) \right\| + \max_{0 \le \tau \le T} \left\| \upsilon^{(N)}_{_{smn}}(\tau) \right\| \\ &\leq \frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi} \left\| \gamma \right\| \left\| \upsilon^{(0)}(\tau) \right\| + \frac{\left(\sqrt{9T\pi} + 16\sqrt{T}\right)M}{3\pi} \\ &\text{we get } \upsilon^{(N)}(\tau) \in B. \\ &\left\{ \upsilon(\tau) \right\} = \left\{ \upsilon^{(N)}_{_{0}}(\tau), \upsilon^{(N)}_{_{smn}}(\tau), \upsilon^{(N)}_{_{smn}}(\tau), \upsilon^{(N)}_{_{smn}}(\tau), \upsilon^{(N)}_{_{smn}}(\tau), m, n = 1, 2, ... \right\} \in B \\ & \cdot \end{split}$$

By using the same operations, we obtain:

$$\left\|\sigma^{(N+1)}(\tau)\right\| \leq \frac{\pi}{2} + \frac{1}{\left\|\upsilon^{(N)}(\tau)\right\|}$$

we get $\sigma^{(N)}(\tau) \in \mathbf{B}$.

Let us show that the convergence of solution:

$$\begin{split} & \left\| \upsilon^{(N+1)}(\tau) - \upsilon^{(N+1)}(\tau) \right\| \\ \leq & \left(\frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi\sqrt{N!}} \right)^{(N)} \left\| \gamma \right\|^{(N)} \left\| \upsilon^{(N)}(\tau) \right\| \\ & \left\| \sigma^{(N+1)}(\tau) - \sigma^{(N)}(\tau) \right\| \leq \frac{\pi}{2} + \left(\frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi\sqrt{N!}} \right)^{(N)} \left\| \gamma \right\|^{(N)} \left\| \upsilon^{(N)}(\tau) \right\| \\ & \text{Then } \upsilon^{(N+1)}(\tau) \to \upsilon^{(N)}(\tau), \sigma^{(N+1)}(\tau) \to \sigma^{(N)}(\tau), N \to \infty. \\ & \left\| \upsilon^{(N+1)}(\tau) - \upsilon(\tau) \right\| \end{split}$$

$$\begin{split} &\| \mathbf{0}^{(1)} - \mathbf{0}(\tau) \| \\ &\leq \left(\frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi\sqrt{N!}} \right)^{(N)} \| \boldsymbol{\gamma} \|^{(N)} \operatorname{x} \exp \left(\frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi} \right)^2 \| \boldsymbol{\gamma} \|^2 \\ & \upsilon^{(N+1)}(\tau) \to \upsilon(\tau), \sigma^{(N+1)}(\tau) \to \sigma(\tau), N \to \infty. \end{split}$$

Applying Cauchy inequality, Hölder Inequality, Lipschitzs condition, and Bessel inequality to the difference, we obtain

$$\left\|\upsilon(\tau) - \upsilon(\tau)\right\| \le 0x \exp\left(\frac{\sqrt{9T\pi} + 16\sqrt{T}}{3\pi}\right)^2 \left\|\gamma\right\|^2$$

 $v(\tau) = v(\tau), \sigma(\tau) = \rho(\tau)$. Thus, we proved the uniqueness of the solution.

.

4. THE EXAMPLE FOR NUMERICAL METHOD

The linearize of the problem:

$$\begin{split} \upsilon_{t}^{(n)} &= \upsilon_{xx}^{(n)} + \sigma(\tau)\upsilon_{yy}^{(n)} + \theta(\xi, \eta, \tau, \upsilon^{(n-1)}), \\ \upsilon^{(n)}(\xi, \eta, 0) &= \phi(\xi, \eta), \xi \in [0, \pi], \eta \in [0, \pi] \\ \upsilon^{(n)}(0, \eta, \tau) &= \upsilon^{(n)}(\pi, \eta, \tau), \eta \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}(\xi, 0, \tau) &= \upsilon^{(n)}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \text{Let us } \upsilon^{(n)}(\xi, \eta, \tau) &= \psi(\xi, \eta, \tau). \\ \psi_{\tau} &= \psi_{\xi\xi} + \sigma(\tau)\psi_{\eta\eta} + \theta(\xi, \eta, \tau, \psi), \\ \upsilon^{(n)}(\xi, \eta, 0) &= \phi(\xi, \eta), \xi \in [0, \pi], \eta \in [0, \pi] \\ \upsilon^{(n)}(0, \eta, \tau) &= \upsilon^{(n)}(\pi, \eta, \tau), \eta \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}(0, \eta, \tau) &= \upsilon^{(n)}(\chi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\chi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ \upsilon^{(n)}_{\eta}(\xi, 0, \tau) &= \upsilon^{(n)}_{\eta}(\xi, \pi, \tau), \xi \in [0, \pi], \tau \in [0, T] \\ [0, \pi]^{2} \times [0, T] \text{ is divided into a } M^{2} \times N \text{ mesh with the step size } h = \pi/M, \ \tau = T/N \end{split}$$

Using implicit finite-difference method for the problem:

$$\begin{split} & \frac{1}{\tau} \Big(\psi_{i,j}^{k+1} - \psi_{i,j}^{k} \Big) \\ & = \frac{1}{h^2} \begin{bmatrix} \left(\psi_{i-1,j}^{k+1} - 2\psi_{i,j}^{k+1} + \psi_{i+1,j}^{k+1} \right) \\ & + \sigma^k \left(\psi_{i,j-1}^{k+1} - 2\psi_{i,j}^{k+1} + \psi_{i,j+1}^{k+1} \right) \end{bmatrix} \\ & + \theta_{i,j}^{k+1}, \end{split}$$

$$\psi_{i,j}^{0} = \varphi_{i},$$

$$\psi_{0,j}^{k} = \psi_{M+1,j}^{k}, \psi_{M+1,j}^{k} = \frac{\psi_{1,j}^{k} - \psi_{M,j}^{k}}{2}$$

$$\begin{split} \psi_{i,0}^{k} &= \psi_{i,M+1}^{k}, \psi_{i,M+1}^{k} = \frac{\psi_{i,1}^{k} - \psi_{i,M}^{k}}{2} \\ \sigma(\tau) &= \frac{\varsigma'(\tau) - \int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \, \theta(\xi, \eta, \tau) d\xi d\eta - \frac{\pi^{3}}{2} \psi_{\eta}(\pi, \tau)}{\frac{\pi^{3}}{2} \psi_{\xi}(\pi, \tau)} \\ \sigma^{k+1} &= -\frac{\left(\left(k^{k+2} - k^{k}\right)/\tau\right)}{\left(\frac{\pi^{3}}{2} \psi_{\xi}(\pi, \tau)\right)^{k}} \\ \frac{-\left(\int_{0}^{\pi} \int_{0}^{\pi} \xi \eta \, \theta(\alpha, \beta, \tau) d\xi d\eta\right)^{k}}{\left(\frac{\pi^{3}}{2} \psi_{\xi}(\pi, \tau)\right)^{k}} \\ -\left(\frac{\pi^{3}}{2} \psi_{\xi}(\pi, \tau)\right)^{k} \end{split}$$

where k = 0, 1, ..., N.

From Simpson's scheme,

$$\begin{aligned} &\frac{1}{\tau} \Big(\psi_{i,j}^{k+l(s+1)} - \psi_{i,j}^{k+l(s)} \Big) \\ &= \frac{1}{h^2} \begin{bmatrix} \left(\psi_{i-1,j}^{k+l(s+1)} - 2\psi_{i,j}^{k+l(s+1)} + \psi_{i+1,j}^{k+l(s+1)} \right) \\ + \sigma^{k(s+1)} \left(\psi_{i,j-1}^{k+l(s+1)} - 2\psi_{i,j}^{k+l(s+1)} + \psi_{i,j+1}^{k+l(s+1)} \right) \end{bmatrix} \\ &+ \theta_{i,j}^{k+1}, \\ &\psi_{0,j}^0 = \phi_i, \\ &\psi_{0,j}^{k+l(s)} = \psi_{M+1,j}^{k+l(s)}, \psi_{M+1,j}^{k+l(s)} = \frac{\psi_{1,j}^{k+l(s)} - \psi_{M,j}^{k+l(s)}}{2} \\ &\psi_{i,0}^{k+l(s)} = \psi_{i,M+1}^{k+l(s)}, \psi_{i,M+1}^{k+l(s)} = \frac{\psi_{i,1}^{k+l(s)} - \psi_{i,M}^{k+l(s)}}{2} \end{aligned}$$

 $\psi_{i,j}^{k+1(s+1)}$ is found.

5. CONCLUSION

The inverse problem for the periodically bounded nonlinear two-dimensional parabolic equation was investigated. Two important investigations were carried out. These are determined by both theoretical and numerical examination. The Fourier method and finite difference method were used as methods.

6. ACKNOWLEDGMENTS

We would like to thank the reviewers.

REFERENCES

1. Sharma, P.R., Methi, G. (2012). Solution of twodimensional parabolic equation subject to non-local conditions using homotopy Perturbation method, Jour. of App.Com., 1, 12-16.

2. Cannon, J. Lin, Y. (1899). Determination of parameter p(t) in Hölder classes for some semilinear parabolic equations, Inverse Problems, 4, 595-606.

3. Dehghan, M. (2005). Efficient techniques for the parabolic equation subject to nonlocal specifications, Applied Numerical Mathematics, 52(1), 39-62,2005.

4. Dehghan, M. (2001). Implicit Solution of a Two-Dimensional Parabolic Inverse Problem with Temperature Overspecification, Journal of Computational Analysis and Applications, 3(4). 5. Bağlan, I., Kanca, F. (2020). Solution of the boundary-value problem of heat conduction with periodic boundary conditions, Ukrainian Mathematical Journal, 72(2), 232-245.

6. Bağlan, I., Kanca, F. (2020). Two-dimensional inverse quasilinear parabolic problems with periodic boundary conditions of the boundary-value problem of heat conduction with periodic boundary conditions, Applicable Analysis, 98(8), 1549-1565. 7. Ionkin, N.I. (1977). Solution of a boundary value problem in heat conduction with a nonclassical boundary condition, Differential Equations, 13, 204-211.

8. Hill G.W. (1886), On the part of the motion of the lunar perigee which is a function of the mean motions of the sun and moon, Acta Mathematica, 8,1-36.

IDUNAS

NATURAL & APPLIED SCIENCES JOURNAL

2024 Vol. 7 No. 2 (69-85)

Determination of the Optimum Laser Processing Parameters Required to Obtain a Stable Hydrophobic Surface on the Surface of AA1050 Aluminum Alloy

Research Article

Satılmış Ürgün^{1*}, Mustafa Özgür Bora ², Sinan Fidan², Şeref Tosunoğlu^{3,4}, Timur Canel⁵

¹ Kocaeli University, Faculty of Aeronautics and Astronautics, Department of Aviation Electrics and Electronics, Kocaeli, Türkiye ² Kocaeli University, of Aeronautics and Astronautics, Department of Airframe & Powerplant Maintenance, Kocaeli, Türkiye ³ Kocaeli University, Institute of Educational Sciences. 41380 Umuttepe, Kocaeli, Türkiye.

⁴ BS Petrol A.Ş., Kocaeli, Türkiye

⁵ Kocaeli University, Faculty of Art and Sciences, Department of Physics, Kocaeli, Türkiye

Author E-mails: urgun@kocaeli.edu.tr ozgur.bora@kocaeli.edu.tr sfidan@kocaeli.edu.tr bspetrol@bspetrol.com tcanel@kocaeli.edu.tr S. Ürgün ORCID ID: 0000-0003-3889-6909 M. Ö. Bora ORCID ID: 0000-0003-4385-4981 Ş. Tosunoğlu ORCID ID: 0009-0004-2607-1584 T. Canel ORCID ID: 0000-0002-4282-1806

*Correspondence to: Satılmış Ürgün, Kocaeli University, Faculty of Aeronautics and Astronautics, Department of Aviation Electrics and Electronics, Kocaeli, Türkiye DOI: 10.38061/idunas.1569710

Received: 18.10.2024; Accepted: 12.11.2024

Abstract

In this study, the surfaces of sheets made of AA1050 aluminum alloy, which has high aluminum purity and high strength, were roughened with a fiber laser. The aim of the study was to obtain a surface with a highly stable contact angle. For this purpose, it was necessary to minimize the change in the contact angle with time. Three different laser processing parameters were used. The effects of the laser parameters on the stability of the contact angle were investigated. According to the data obtained from the study, the type of texture created on the surface was calculated as the most effective parameter to obtain a stable surface. The rate of influence of the texture type parameter on the result is 61.61%. The parameter with the least effect on the result was calculated as laser-scanned factor with 15.31%. The parameter that moderately affected the result was calculated as laser power with 23.08%. In addition, ANOVA calculations suggested that a more stable surface pattern could be obtained by experimental parameters.

Keywords: Surface Texturing, Fiber Laser Machining, AA1050 Aluminum Alloy, Taguchi Optimization, Hydrophobic Surface.

1. INTRODUCTION

A metal alloy is a substance made of two or more metals, or a metal and non-metallic elements mixed to improve the metal's characteristics for a particular use. Beyond what is achievable with pure metals, alloys are utilized to increase mechanical strength, durability, corrosion resistance, conductivity, and heat tolerance. Typical examples are brass (copper and zinc), bronze (copper and tin), and steel, which is an alloy of iron and carbon. These alloys have qualities that make them invaluable in a variety of industrial fields. For example, steel is perfect for the construction, automotive, and shipbuilding industries because of its increased strength and resistance to wear, whereas stainless steel, which has chromium, is used for kitchenware and medical devices because it resists corrosion.

Aluminum alloys are frequently utilized in the aerospace sector due to their high strength-to-weight ratio and low density, which are essential for decreasing the weight of spacecraft and airplanes [1]. Due to its extreme strength and heat resistance, titanium alloys are indispensable in biomedical and aerospace applications, including implants and prosthetic limbs. Superalloys based on nickel are essential for hightemperature settings, such as gas turbines and jet engines, where oxidation and heat resistance are required. Because of their excellent electrical conductivity, copper alloys are frequently employed in electronics for electrical wiring and connectors [2]. Because of their adaptability, metal alloys are used extensively in a variety of industries, including the energy sector, where they are used in oil rigs, nuclear reactors, and pipelines because they can withstand high temperatures and pressures [3]. In the automotive industry, for example, advanced high-strength steels improve safety and fuel efficiency [4]. Because alloys may be customized to meet specific requirements, they are essential to contemporary industrial processes and technologies.

With an aluminum percentage of 99.5% or more, AA1050 aluminum alloy is a commercially pure aluminum alloy from the 1000 series [5]. Because of its high purity, AA1050 has several numbers of beneficial qualities, including as good electrical, thermal, and corrosion resistance as well as workability [6]. Since AA1050 is a pliable and soft substance, it can be easily stamped, drawn, or shaped, which makes it perfect for applications where formability is crucial [7]. Nevertheless, compared to other aluminum alloys, its mechanical strength is comparatively low, which prevents it from being used in structural applications that call for great strength.

The electrical industry is one of the main sectors that benefits from AA1050's characteristics [8]. This alloy is widely used for busbars, conductors, and electrical cables where efficiency in current transmission is crucial because of its high electrical conductivity. Because of the alloy's superior resistance to corrosion, it can be used in places like heat exchangers and chemical processing plants where other metals could corrode due to exposure to chemicals or moisture. With exceptional thermal conductivity, it is also a well-liked material for cooling components like heat sinks, which are crucial for HVAC and electrical systems [9]. Because AA1050 is non-toxic, resistant to contamination, and simple to mold into thin sheets, it is frequently used in the packaging sector to produce food containers and aluminum foil [10]. Another noteworthy characteristic of the alloy is its reflectivity, which qualifies it for use in the renewable energy industry for solar panel frames, reflectors, and lighting fixtures. Moreover, AA1050 is employed in architectural applications, including cladding, roofing, and decorative panels where aesthetics, durability, and weather resistance are required. Although AA1050's lower strength precludes its usage in load-bearing applications, its high conductivity, resilience to corrosion, and ease of manufacturing make it a flexible material suitable for a wide range of applications in industries, including environmental protection, food

packaging, and electrical and building systems [11]. Because surface treatment improves a material's mechanical, chemical, and physical qualities without changing its bulk characteristics, it is an essential technique in many sectors [12]. To enhance a product's resistance to corrosion, wear and tear, adhesion, electrical conductivity, and aesthetic appearance—all of which contribute to its durability and effectiveness in particular environments—material surface modification is frequently the preferred approach [13]. Surface treatment is economically necessary to prevent degradation, as industry data estimates that corrosion costs alone account for 3-4% of GDP in many industrialized nations [14]. To overcome these difficulties, techniques such as anodizing, plating, coating, and surface hardening are frequently employed. Furthermore, surface treatments are necessary to increase the lubricity of machine parts, lower wear and friction, and prolong the life of vital components [15]. For example, research indicates that in extremely abrasive environments, surface-hardened steel can increase part life by up to three or five times.

Surface treatments like thermal spraying and chemical vapor deposition (CVD) are frequently used in the automotive and aerospace sectors to improve resistance to high temperatures and mechanical strains. In the electronics industry, surface treatments are also used to enhace adhesion properties since materials like circuit boards and semiconductors need changed surfaces to promote coating and adhesive bonding [16]. Industries can customize the surface qualities for particular applications, lowering total material costs while attaining improved performance, by concentrating on surface modification rather than changing the core material. Reducing material waste and extending product life are economic methods and help to improve manufacturing sustainability and energy efficiency.

The process of altering a material's surface qualities with a high-energy laser beam is called laser surface treatment. This process modifies the surface morphology, microstructure, and chemical composition of a material by directing a laser beam onto a particular region of its surface to cause localized heating, melting, or vaporization. The precision and controllability of this method are its main advantages. The depth of penetration and the intensity of the heat applied can be precisely controlled by adjusting the laser's power, wavelength, and pulse duration. This allows for targeted change without changing the material's bulk properties. Techniques such as laser surface melting (LSM), laser surface alloying (LSA), laser cladding, and laser hardening are frequently used in the laser surface treatment process. For instance, in laser hardening, the material's surface is heated by the laser beam to a temperature higher than its critical point, and then the treated region rapidly self-quenches, improving the treated area's hardness and wear resistance [17, 18]. When a material is melted by a laser and applied to a surface, it may take the form of wire or powder that is coated with a protective layer. These procedures are carried out in a regulated setting, frequently with inert gasses present, to avoid oxidation and other unwanted chemical reactions.

In industrial applications, laser surface treatment is particularly advantageous due to its various better advantages over traditional surface treatment technologies [19]. Lasers offer a number of advantages, one of which is localized treatment. Because the laser beam can be precisely targeted, there can be fewer heat-affected zones and nearby materials won't sustain thermal damage. This high degree of precision lowers the possibility of microstructural alterations or distortion to the bulk material, which is crucial for high-performance parts with precise mechanical requirements.

Furthermore, laser surface treatments are incredibly effective and adaptable. Processing durations can be shortened and manufacturing efficiency raised by conducting the procedure quickly and automatically. Additionally, a variety of materials, such as metals, ceramics, and composites, may be treated by lasers, which makes them useful in a variety of sectors. Another significant benefit is the ability to customize surface attributes. Manufacturers can customize surface properties like hardness, roughness, and corrosion resistance to match particular application needs by varying the laser's parameters. Furthermore, compared to conventional techniques like electroplating or chemical coatings, laser treatments frequently use fewer

chemical agents, lowering hazardous waste. This makes them environmentally benign. Because laser surface treatment is precise, adaptable, and may improve surface qualities without sacrificing material integrity, it is favoured in many scientific study domains as well as many enterprises [20].

The automobile industry is one of the main applications for laser surface treatment, where it is used to harden crankshafts, gears, and engine components [21]. Critical automotive parts have a longer lifespan thanks to laser hardening, which lowers maintenance costs and improves vehicle performance by increasing wear resistance and fatigue life. Similar techniques are used in the aerospace sector to reinforce and repair parts subjected to harsh environments, like landing gear and turbine blades, which undergo high levels of stress and temperature cycling. These techniques include laser cladding and surface alloying. Surfaces treated with lasers function better at high temperatures and against oxidation and wear.

Laser surface treatment is a commonly employed technique in the tooling and manufacturing industry to improve the longevity of cutting tools, dies, and molds [22]. By increasing the surface hardness of tools through laser hardening, machining procedures can be performed with less wear and tear. Furthermore, laser texturing is employed to generate precise surface patterns on molds, an essential step in achieving superior surfaces in metal forming and plastic injection molding. In order to improve mold performance and product aesthetics, laser surface treatment is crucial since it allows for precise control over surface roughness and texture [23]. In the biomedical industry, laser surface treatment is also widely utilized to alter the surfaces of medical implants and equipment [24]. To improve osseointegration-the process by which bone grows into an implant-on the surface of orthopaedic implants, for example, laser texturing can produce microstructures. This increases the implants' long-term durability [25]. In the oil & gas and power generation industries, in particular, laser cladding is the material of choice for repairing and strengthening vital parts including drilling equipment, turbine blades, and pipelines. In abrasive settings, the treatment helps prevent erosion, corrosion, and wear. Laser surface treatment is utilized in science to create cutting-edge coatings and materials. Researchers examine how surface alteration affects material performance in harsh situations, including nuclear reactors or space exploration, using lasers. Additionally, laser treatments are used in research on nanotechnology and photonics, where the development of sensors, photonic devices, and functional surfaces with distinctive optical properties requires controlled surface alteration at the micro- and nanoscale. Overall, laser surface treatment is favoured across multiple industries and research fields for its ability to enhance surface properties, improve component performance, and contribute to innovation in material science and engineering [26].

When a material surface repels water, it is said to be hydrophobic. This is indicated by a high contact angle, usually more than 90 degrees, between the surface and the water droplet [27]. This characteristic stems from the surface's poor affinity for water molecules, which is frequently brought about by particular chemical compositions or surface microstructures that lessen the surface-water interaction. Originating from the Greek words "hydro" (water) and "phobos" (fear), the phrase "hydrophobic" means "waterfearing." Water tends to form droplets rather than spreading out when it comes into contact with a hydrophobic surface, which reduces the amount of surface area that the water and the material have in contact. Natural examples of this behaviour include the lotus plant's leaves, which maintain their cleanliness and dryness because to their inherent. Artificial methods for achieving hydrophobicity include surface treatments, chemical coatings, and the fabrication of certain micro- and nanostructures that improve water repellency. Additionally, these surfaces may have the ability to self-clean, with dirt and pollutants being removed by water droplets that roll off the surface. Not only do hydrophobic surfaces repel water, but they are also very desirable in industrial applications because they can show resistance to fouling, corrosion, and biological contamination. This idea is furthered by superhydrophobic surfaces, which have nearly zero water adhesion and even more extreme water-repellent qualities (contact angles above 150 degrees) [28]. Artificial methods for achieving hydrophobicity include surface treatments, chemical coatings, and the fabrication of certain micro- and nanostructures that improve water repellence. Additionally, these surfaces may exhibit self-cleaning properties, where dirt and pollutants are removed by water droplets that roll off the surface. Not only do hydrophobic surfaces repel water, but they are also very desirable in industrial applications because they can show resistance to fouling, corrosion, and biological contamination. This idea is furthered by superhydrophobic surfaces, which have nearly zero water adhesion and even more extreme water-repellent qualities (contact angles above 150 degrees) [29].

Hydrophobic coatings are used in the automotive and aviation sectors to improve aerodynamic performance by lowering drag brought on by water buildup on vehicle surfaces [30]. Hydrophobic coatings are especially useful for windows and windshields because they increase visibility by keeping water from sticking to them and producing droplets that might obscure eyesight. Hydrophobic coatings can also stop ice, frost, or hydrate deposits from forming on pipelines and equipment, which could otherwise interrupt operations in the oil and gas sector. In biotechnological and medical applications, hydrophobic surfaces are also very important because they help stop biological materials like bacteria, proteins, and cells from adhering to them [31]. This anti-fouling characteristic is especially crucial for devices like implants, surgical equipment, and catheters where reducing the danger of contamination is essential.

Furthermore, hydrophobic surfaces are favoured in uses where water-resistant packaging is necessary, including in the food and pharmaceutical sectors, where it's critical to maintain dry and moisture-free items. Hydrophobic surfaces are used in many different industrial domains because of their exceptional capacity to reject water, withstand fouling, and preserve hygienic conditions. Hydrophobic coatings are frequently used on side mirrors, paint, and windshields in the car industry to increase visibility during rainy weather and lessen cleaning requirements. These coatings improve driver safety by keeping water from sticking to the surface and lessen wiper wear. Hydrophobic coatings can also be applied to vehicle's exterior, creating self-cleaning surfaces that prevent dirt buildup and extending the life of the paint job.

Hydrophobic coatings are applied to aircraft wings, fuselages, and windows in the aviation and aerospace sectors to lessen drag brought on by water accumulation [32]. This can increase fuel economy and lessen the weight load of ice building. These coatings are also necessary for de-icing applications, which stop ice from forming on important surfaces while the aircraft is in flight. This is essential for averting mishaps and guaranteeing the aircraft's performance and safety in a range of environmental circumstances.

Hydrophobic surfaces are also highly beneficial to the construction sector, especially for building materials like concrete, glass, and ceramics [33]. Building facades are shielded from water penetration by hydrophobic coatings, which over time prevent efflorescence (salt deposits), water damage, and cracking. These coatings increase the longevity of infrastructure and buildings by making surfaces water-repellent, especially in areas with high humidity or frequent rains.

In the electronics sector, hydrophobic coatings are applied to wearables, tablets, and smartphones to enhance water resistance [34]. Maintaining the performance of consumer devices that are frequently exposed to moisture—like those used outside or accidentally spilled—requires doing this. Hydrophobic films are also applied to some electronic parts, such printed circuit boards (PCBs), to stop water-induced corrosion and extend their lifespan.

In the marine industry, where exposure to seawater can cause quick corrosion and biofouling, hydrophobic surfaces are especially crucial [35]. Ship hulls, offshore platforms, and subsea equipment are coated with hydrophobic materials to stop water seepage, corrosion, and the growth of organisms like algae and barnacles, which can have a detrimental effect on operating effectiveness. These coatings help lessen drag on the hulls of vessels, increasing fuel economy and requiring less maintenance.

Hydrophobic surfaces are used in the medical and healthcare sectors to stop bacterial development and contamination on implants, surgical tools, and other medical equipment. Catheters, stents, and other invasive devices with hydrophobic coatings minimize the adhesion of biological materials, therefore lowering the risk of infection. Hydrophobic coatings in pharmaceutical packaging shield medications from moisture, maintaining their stability and potency for longer.

All things considered, hydrophobic surfaces are essential to many different industries, offering advantages including improved performance, longevity, and safety by guarding against water-related damage and

contamination. Hydrophobic treatments are vital in scientific and industrial applications because they can provide surfaces that are self-cleaning, water-repellent, and anti-fouling. By modifying the surface chemistry and topography, different surface modification techniques can convert the surfaces of aluminum alloys AA1050 into hydrophobic surfaces that promote water repellency.

Combining surface texturing with chemical treatments is one of the most successful strategies. In order to achieve hydrophobicity, texturing techniques are designed to provide micro- or nanoscale roughness on the surface. This limits the surface's area of contact with water droplets. For this, laser surface texturing—which involves using a laser to create a patterned microstructure on the aluminum surface—is frequently utilized. The resultant hierarchical patterns resemble the surfaces found naturally in hydrophobic phenomena, such the leaf of a lotus plant.

Chemical etching is another technique for making micro-rough surfaces. Using acidic or alkaline solutions, material is removed selectively to produce a rough surface with micro- and nanostructures [36]. The next stage is to alter the surface chemistry to improve the water-repelling qualities of the surface once the suitable texture has been obtained. Low-surface-energy coatings are usually applied to achieve this, as they reduce the interaction between water molecules and the surface. For this reason, fluoropolymer coatings and silane-based coatings are frequently employed since they impart hydrophobic functional groups, including methyl groups (-Si(CH₃)₃) or fluoro groups (-CF₃), into the aluminum surface. Because of the considerable surface energy reduction provided by these coatings, water droplets are able to roll down the surface in beads rather than spreading out.

Apart from chemical treatments and laser texturing, anodization is a further technique that can be utilized to improve the hydrophobicity of aluminum alloys AA1050 [37]. The aluminum surface develops a thick, porous oxide layer during anodization, which can be further functionalized with hydrophobic substances. When paired with chemical treatments, the anodized surface can display superhydrophobic qualities, which are defined by water contact angles greater than 150 degrees.

Whether applied individually or in combination, these methods enable the surfaces of AA1050 aluminum alloys to become extremely water-repellent. This property makes the alloys suitable for use in outdoor construction materials, automotive components, and marine environments where resistance to corrosion and moisture is crucial.

In this study, an attempt was made to optimize the laser process parameters required to obtain a stable hydrophobic surface. The process parameters used to obtain more stable hydrophobic surface. For this purpose, pure water was applied to the surfaces obtained with different process parameters. The contact angle was measured for the first 10 seconds after the water was dripped. Contact angles were measured every second in the first 10 seconds.

The value with the least deviation from the average of the contact angles in the first 10 seconds was accepted as the most stable value. Accordingly, the value with the smallest standard deviation was accepted as the most stable hydrophobic surface and the smallest standard deviation was characterized as the best value. A statistical tool used to quantify the degree of variation or dispersion in a set of data values is the standard deviation. It sheds light on how widely distributed a dataset's values are around the mean (average). Whereas a large standard deviation suggests that the values tend to be close to the mean. The square root of the variance, or the average of the squared deviations from the mean, is what is known as the standard deviation in mathematics. Equation 1 provides the formula for the standard deviation (σ) of a population:

$$\sigma = \sqrt{\frac{\Sigma(x_i - \mu)}{N}} \tag{1}$$

Where:

 σ = population standard deviation

 x_i = each data point

 μ = population mean N = number of data points

Since the standard deviation gives a numerical depiction of data variability, it is a crucial statistical tool. It is essential for comprehending the consistency and dependability of datasets in domains like finance, economics, quality control, and research.

Standard deviation comes in two primary varieties, each with a distinct function:

Population Standard Deviation (σ): This kind of standard deviation is applied when the study takes into account every member of a group or population. It is a representation of the dataset's overall variability. In domains such as biology or engineering, where the complete collection of measurements is accessible, comprehending the general variance within the population is crucial. The population standard deviation proves especially useful in these scenarios.

Sample Standard Deviation (s): When there is only a sample of the population available, the sample standard deviation (s) is utilized. The sample standard deviation aids in estimating the variability of the population from which the sample is obtained, as a sample is merely a subset of the entire population. When utilizing a sample, the formula divides by N-1, where N is the total number of observations in the sample, to account for any bias. This is known as Bessel's correction, ensures that the sample provides an unbiased estimate of the population variance.

The precision and dependability of standard deviation computations are impacted using both types, which vary based on whether the dataset represents the full population or merely a sample. Standard deviation is a crucial tool in engineering for dependability analysis, risk management, and quality control. It aids engineers in comprehending measurement, process, or system variability, which is essential for creating reliable and consistent products. There are various applications of standard deviation in engineering.

Quality Control: Standard deviation is essential for keeping an eye on product consistency in manufacturing and production. For example, engineers monitor the standard deviation of important measurements when making components that need to meet certain tolerances to make sure the process stays within allowable bounds. In a production process, a low standard deviation means that the majority of the products fall within the intended parameters; on the other hand, a high standard deviation could suggest flaws or differences in the procedure.

Reliability Engineering: Standard deviation is used in the field of reliability engineering to evaluate the lifespan and robustness of systems or goods. Through the assessment of performance indicators including fatigue life, failure rates, and time-to-failure variability, engineers are able to forecast the degree of system reliability under various scenarios. A lower standard deviation suggests more predictable performance, which is essential in systems where reliability is critical, such as aerospace or automotive industries.

Risk and Safety Analysis: Standard deviation is another tool used by engineers in risk and safety analyses. For example, in structural engineering, the standard deviation can be used to assess the variability in material strengths or load-bearing capacities in order to forecast possible dangers or failure modes. Standard deviation aids engineers in creating safe structures that can resist changing loads or environmental circumstances.

Process Control and Optimization: Standard deviation is a tool used in process engineering to find variations in processes. It is used by engineers to assess a process's consistency over time. Process engineers can create a more stable and optimized process that is more cost-effective and efficient by lowering the standard deviation.

In the field of engineering, a distribution with a small standard deviation signifies limited variability within the dataset, as the data points are tightly packed around the mean. This implies that the system or process under analysis has a high degree of precision and that the observed values or results are consistent. In the manufacturing industry, for example, a minimal standard deviation in a produced part's dimensions indicates that the production process is well-controlled, and the parts are almost similar, which results in improved quality and reliability. This consistency is frequently desired in engineering design since it increases system performance predictability and lowers the possibility of failures or outliers. Furthermore, a low standard deviation lessens the requirement for significant safety factors or contingencies in design and analysis by giving engineers more confidence that the measured values accurately reflect the system's performance or behaviour. In general, decreasing variability is essential to ensuring efficiency, safety, and precision in many engineering applications.

In light of this, standard deviation is an effective statistical instrument that helps engineers comprehend, manage, and maximize variability in systems, procedures, and measurements—thereby enhancing the dependability, security, and caliber of engineering endeavors and output.

2. MATERIALS AND METHODS

2.1 Optimization Method

In scientific and engineering research, optimization techniques are crucial because they facilitate the methodical and effective search for the best answers to challenging issues. In engineering, many design procedures, such as lowering energy consumption in systems, optimizing material utilization, or enhancing the performance of machines, require judgments among several factors. Finding the best balance between conflicting goals would take a lot of time and resources if optimization techniques weren't used. In scientific research, optimization algorithms are essential for data analysis, simulation, and model fitting. They enable scientists to fine-tune parameters and produce results that are dependable and accurate. This is particularly beneficial in domains like computational biology, physics, and chemistry, where optimization aids in the design of experiments and in solving difficult equations. Furthermore, sophisticated optimization techniques like gradient-based methods, heuristic approaches, and genetic algorithms offer significant computational advantages, making it possible to solve problems that would otherwise be insurmountable as they become more complex with larger datasets or more variables. Therefore, optimization plays a crucial role in enhancing the efficacy and efficiency of scientific and engineering undertakings, which in turn spurs innovation and discovery.

Genichi Taguchi created the Taguchi method, a statistical technique for enhancing process efficiency and product design with an emphasis on lowering variability and raising quality. The Taguchi technique is centered on resilient design, which tries to improve consistency and reliability by lessening the sensitivity of processes or products to fluctuations brought about by outside influences (noise). Multiple factors can be evaluated simultaneously with fewer experiments thanks to a technique that uses orthogonal arrays to systematically design experiments. This efficiency in experimental design is one of its key advantages, since it cuts time and costs while still generating valuable insights into the impact of many factors. The Taguchi approach also incorporates a loss function, which measures the financial deviation from the target performance and promotes not only satisfying the requirements but also minimizing the impact of any deviation.

The Taguchi technique has several benefits, such as being easy to use, affordable, and capable of handling several variables without requiring a lot of processing power. Moreover, it works especially effectively in scenarios where standard optimization techniques would be hard to use, including complicated variable

interactions in processes or costly or impracticable real-world testing. The Taguchi method has a wide range of applications. It is widely used in industrial industries, especially in the electronics, automotive, and aerospace sectors, to improve process efficiency and product quality. It has been used, for instance, to create high-performance materials, enhance production procedures, and optimize machining parameters. The Taguchi technique has been applied outside of manufacturing, in areas including chemical processing, environmental engineering, and biotechnology, where it is crucial to optimize experimental conditions and reduce variability. All things considered, the Taguchi technique offers an organized, effective method of optimization, which makes it useful in many different fields of science and industry.

In the Taguchi method, three different characteristics are used according to the purpose of the experiments. If the largest value is to be obtained in the experiments, 'larger the better' characteristic is used, if the smallest value is to be obtained, 'smaller the better' characteristic is used. If the experiments are intended to approach a predetermined numerical target, the 'nominal the best' characteristic is used. According to these three different characteristics, three different calculation formulae are used. These are

For Larger the better characteristic;

$$S_{N_i} = -10 \log_{10} \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{y_{ij}^2} \right]$$
(2)

For Smaller the better characteristic;

$$S_{N_{i}} = -10 \log_{10} \left[\frac{1}{n} \sum_{i=1}^{n} y_{ij}^{2} \right]$$
(3)

For Nominal the best characteristic;

$$S_{N_{NB}} = -10 \log_{10} \left[\frac{1}{n} \sum_{i=1}^{n} (y_i - m)^2 \right]$$
(4)

This study aims to obtain the most Stable Hydrophobic Surface. For this purpose, the "smaller the better" characteristic was used.

2.2 Material and Experimental

In this study, the surfaces of 2 mm thick AA1050 aluminum sheets were machined with different machining parameters given in Table 1. A high precision fiber laser was used to process the surfaces.

In this study, fiber laser technology was used to engrave various geometric patterns onto the surface of an aluminum alloy from the AA1050 series. Four distinct laser power levels (40 W, 60 W, 80 W, and 100 W) and four distinct "Theoretical laser scanned area factors (%)" (80%, 60%, 40%, and 20%) were applied to each of the four geometric designs (Square, Diamond, Hexagon, and Circle) that were examined. These studies were carried out to investigate the response of laser-processed surfaces to different laser parameters and surface geometries in terms of surface energy, microstructure characteristics, and surface roughness.

The shapes of the laser-applied patterns were used to categorize the textures, and for every geometric design, a number of experimental situations were investigated. The aim of this research is to examine how laser processing parameters affect aluminum surfaces and identify the prerequisites for achieving ideal surface characteristics.

High-intensity laser beams can be produced using dependable, effective fiber laser systems. They find extensive application in micromechanical applications, materials processing, and marking procedures. Fiber lasers are notable for their long lifespan, high beam quality, little maintenance, and energy efficiency. For medium-to-high power applications, a fiber laser with a 100W power output is perfect. It works well for metal cutting, marking, and surface modification.

In the experiments, the 3 parameters given in Table 1 were analysed at 4 different levels. Two of these parameters are numerical parameters and the other one is the classification of the patterns formed on the surface.

Pattern Type	Scanned Area factor (%)	Laser Power (Watt)
Square	80	40
Diamond	60	60
Hexagonal	40	80
Circle	20	100

Table 1. Three machining parameters and their fours of levels.

3. RESULTS AND DISCUSSION

Traditional experimental design requires 3^4 (=81) experiments when three factors with four levels are studied. When optimization techniques are applied, fewer experiments are required to yield effective results. This inquiry used the Taguchi method, which produces good findings in many scientific and technical disciplines. To reduce the number of experiments, the Taguchi technique suggests that selecting the appropriate orthogonal array should be done first. According to the Taguchi approach, in order to reduce the number of tests, selecting the appropriate orthogonal array should be done first. Because three parameters and four levels were used in this experiment, the L₁₆ orthogonal array was used. The laser machining sets specified in Table 1 were arranged in accordance with the L₁₆ orthogonal array, and experiment sets were built. Table 2 lists the experimental configurations and the means of the standard deviations of the contact angle values of the surfaces obtained using these configurations.

Table 2. Sets of experiments produced by the Taguchi method, arranged according to the L_{16} orthogonal array.

	Texture	Scanned area factor (%)	Power (W)	Standard deviation of contact angles	S/N values of contact angle
1	Square	80	40	2.16	-6.68
2	Square	60	60	2.03	-6.16

3	Square	40	80	3.55	-11.00
4	Square	20	100	4.58	-13.22
5	Diamond	80	60	12.22	-21.74
6	Diamond	60	40	6.17	-15.81
7	Diamond	40	100	7.24	-17.19
8	Diamond	20	80	4.20	-12.46
9	Hexagon	80	80	NA	NA
10	Hexagon	60	100	NA	NA
11	Hexagon	40	40	9.03	-19.12
12	Hexagon	20	60	5.54	-14.87
13	Circle	80	100	10.36	-20.30
14	Circle	60	80	8.25	-18.33
15	Circle	40	60	40.14	-32.07
16	Circle	20	40	2.75	-8.79

Table 2 reveals that reliable data could not be obtained with the 9th and 10th test sets. This may be due to the inhomogeneity of the material surface. The contact angles of the textured surfaces obtained using the test sets were made every second for the first 10 seconds after the droplet was dropped on the surface. The contact angles of the surfaces generally show a decreasing trend. This indicates that the liquid dripped over time is dispersed on the surface. In this study, it is aimed to obtain the machining parameters required to obtain the surface with the least change in the contact angle or the least change in the contact angle. For this purpose, the standard deviations of the contact angles measured in the first 10 seconds were calculated and the results are presented in Table 2. Signal to noise ratios of these standard deviation values were calculated according to Taguchi method. The obtained signal to noise ratio values were given in Table 2. As seen in Table 2, the lowest standard deviation was obtained with the experiment set number 2. The largest standard deviation value was obtained with the experiment set numbered 16. Figure 1 shows the image of the droplet on the surface where (a) the smallest and (b) the largest standard deviation values are obtained. The largest value of the contact angle of the droplet in Figure 1a is 165.22° and the largest value of the contact angle of the droplet in Figure 1a is 107.16°.



Figure 1. Images obtained during the measurement of contact angles of the surfaces giving the (a) largest and (b) smallest standard deviation values.

The ANOVA table presented in Table 3 shows the sum of the squares (SSi) of the means of the S/N values for each level and the variance of these values. The table shows that according to the Taguchi method, the standard deviation of the contact angles measured in the first 10 seconds of the textured surface to be obtained in the experiment with the experimental set with texture type square, scanned factor 20 % and laser power 40 W is 1.43. The ANOVA table also shows that the factor with the greatest influence on the standard deviation is the texture type with 61.61%. The second most influential factor was laser power with 23.08% and the least influential factor was scanned factor with 15.31%.

	Average S/N									
	1st	2nd	3rd	4th						
Factors	level	level	level	level	А	В	С	D	Е	F
Texture	-9.26	-16.80	-16.99	-19.87	4	247.46	61.87	61.61	1	1
Scanned Factor (%)	-16.24	-13.43	-15.77	-12.33	4	61.50	15.38	15.31	4	20%
Power (W)	-12.60	-18.71	-13.93	-16.90	4	92.68	23.17	23.08	1	40 W
		-15.55								
Total						401.65		100.00		
Optimum S/N										-3.09
Minimum standard deviation										1.43

Table 3. ANOVA table prepared using the Taguchi technique. A- Degree of freedom, B- Sum of squares (SSi), C- Average of sum of squares (variance), D- Effect of factors (%), E- Optimum Levels, F- Optimum Values (calculated).

Main Effect Plots

Main effect plots are an essential tool in statistical analysis, particularly when talking about experimental design and analysis of variance (ANOVA). These plots provide a visual representation of the average response of a dependent variable across different values of a single independent variable while all other parameters are maintained constantly. To make it easier to analyse experimental data, main effect plots are mainly used to examine and illustrate the relationship between independent and dependent variables. One of the key benefits of main effect plots is their ability to show the presence and magnitude of main effects, or the influences of various independent factors on the dependent variable.

By plotting the mean answer for each level of the independent variable, main effect plots allow researchers to assess the overall effect of an independent variable on a dependent variable, regardless of the levels of other variables. This facilitates the identification of trends, patterns, and variances in the dependent variable across different independent variable levels. The main effect plot is a helpful diagnostic tool for determining potential interactions between independent variables. An interaction occurs when the amount of one independent variable has a varied effect on the dependent variable. By examining main effect plots in conjunction with interaction plots, researchers can get insight into the complex dynamics of the system they are studying. Specifically, they can ascertain whether the relationship between the independent and dependent variables varies across levels of other independent variables. Main effect plots are helpful in assessing experimental results and are necessary for model validation and selection. By visually analysing the main effect plots, researchers can ascertain whether the linearity and homoscedasticity assumptions that form the basis of the statistical model are satisfied.

As can be seen from the main effect plots given in Figure 2, although "texture type" does not express a numerical value, all three plots show similar characteristics. Since the aim of the study was to obtain a stable surface considering the contact angle, the smallest standard deviation values were obtained when the texture

type was square. The largest standard deviation values were obtained when the texture type was circle. Considering the scanned factor, the smallest standard deviation value is obtained at the smallest value of the scanned factor. An increase in the standard deviation is observed until the scanned factor reaches 40 %. After this value, a relatively slow decrease is observed until the scanned factor reaches 60 %. However, when the scanned factor increased from 60% to 80%, the standard deviation increased rapidly again. Considering the laser power, the smallest standard deviation values were obtained at 40 % and 80 %. As can be seen in this graph, when the laser power increased from 40 W to 60 W, a rapid increase in the standard deviation was observed, but when the power was increased from 60 W to 80 W, the standard deviation decreased at the same rate. When the power was increased from 80 W to 100 W, a very small increase in the standard deviation was observed.



Figure 2. Main effect plots for; (a) Texture Type, (b) Scanned Factor and (c) Laser Power.

Regression Equation

A regression equation represents the relationship between one or more independent variables and one or more dependent variables mathematically. Based on the known values of the independent variables, it is used to forecast or estimate the value of the dependent variable. The dependent variable is expressed as a linear combination of the independent variables plus an error term in the most basic form, known as linear regression. Nonlinear regression models can be used in more complicated situations to capture more complex interactions. Numerous scientific and engineering domains make extensive use of regression equations. They are used in structural modelling, material performance study, and load prediction in mechanical and civil engineering. They are employed in electrical engineering to represent system behaviour in signal processing and control systems. Regression models are used in environmental research to evaluate the effects of climate change and predict the dispersion of pollutants. In the biological sciences, regression analysis is also essential for simulating population dynamics or the spread of disease. Regression equations provide the advantages of being able to forecast outcomes, model real-world occurrences, and infer correlations between variables. The use of error terms allows scientists and engineers to account for

uncertainties when analysing massive data sets, gaining actionable insights, and optimizing systems. Equations 5-8 present the regression equation obtained using Minitab 21.1.1 for regression analysis.

For Circle	$Std. Dev. = 15.6 + (0,011 \times SF) - (0,011 \times P)$	(5)
For Diamond	$Std. Dev. = 7.7 + (0.011 \times SF) - (0.011 \times P)$	(6)
For Hexagonal	$Std. Dev. = 7.5 + (0,011 \times SF) - (0,011 \times P)$	(7)
For Square	$Std. Dev. = 3.3 + (0,011 \times SF) - (0,011 \times P)$	(8)

Table 4 provides the minimal standard deviation values determined by the regression equation, as well as the standard deviation values and error rates derived from the data. Table 5 shows that the estimated values of the standard deviation agree rather well with the findings of the measurements.

 Table 4. Calculated standard deviations.

Exp No	Texture	Scan	Р	Calculated
	Туре	Factor	(W)	Std. Dev.
		(%)		With
				regression
				Equations
1	Square	80	40	3,74
2	Square	60	60	3,3
3	Square	40	80	2,86
4	Square	20	100	2,42
5	Diamond	80	60	7,92
6	Diamond	60	40	7,92
7	Diamond	40	100	7,04
8	Diamond	20	80	7,04
9	Hexagonal	80	80	*
10	Hexagonal	60	100	*
11	Hexagonal	40	40	7,5
12	Hexagonal	20	60	7,06
13	Circle	80	100	15,38
14	Circle	60	80	15,38
15	Circle	40	60	15,38
16	Circle	20	40	15,38

4. CONCLUSION

The paper provides a systematic analysis of how to optimize the parameters of a fiber laser to produce patterns on AA1050 aluminum alloy plates that have various geometric properties. The ideal parameters for three important variables were found using the Taguchi technique. These three parameters are laser power, scanned factor, and texture type. The surface patterns' geometries are directly impacted by these characteristics. The study yielded data that indicated that the most beneficial parameter for achieving a stable surface was the texture type that was formed on the surface. The percentage of the outcome that is impacted by the texture type parameter is 61.61%. Laser scanned factor was shown to have the least impact on the outcome, accounting for 15.31% of the total. Laser power was found to have a moderate impact on the outcome, accounting for 23.08% of the parameter.

Experimental systems were constructed with the L16 orthogonal array. As a result, it was able to effectively explore the parameter space and produce precise results while avoiding resource waste. ANOVA calculations also revealed that a more stable surface pattern might be produced in an experiment with different parameters than those used in the actual studies.

Strong correlations between the experimental data and the anticipated model were also revealed by the regression analysis. This demonstrated the validity of the Taguchi technique for enhancing laser processing parameters. By presenting solid proof that these ideas are well-established and outlining the benefits that can already be obtained by figuring out the ideal conditions, this study adds to the ongoing discussion in this area.

5. ACKNOWLEDGMENTS

The authors like to thank Sezgin Sac Ltd. Sti. for their help in providing the materials used.

REFERENCES

- Raj, R. J., Selvam, P., & Pughalendi, M. (2021). A review of aluminum alloys in aircraft and aerospace industry. J. Huazhong Univ. Sci. Technol, 1671, 4512.
- 2. Czerwinski, F. (2024). Aluminum alloys for electrical engineering: a review. *Journal of Materials Science*, 1-46.
- Khalid, M. Y., Umer, R., & Khan, K. A. (2023). Review of recent trends and developments in aluminum 7075 alloys and metal matrix composites (MMCs) for aircraft applications. *Results in Engineering*, 101372.
- Masters, I. G., Williams, D. K., & Roy, R. (2013). Friction behaviour in strip draw test of pre-stretched high strength automotive aluminum alloys. *International Journal of Machine Tools and Manufacture*, 73, 17-24.
- 5. Zaraska, L., Sulka, G. D., Szeremeta, J., & Jaskuła, M. (2010). Porous anodic alumina

formed by anodization of aluminum alloy (AA1050) and high purity aluminum. *Electrochimica Acta*, 55(14), 4377-4386.

- 6. Garcia-Garcia, F. J. (2009). Effect of Magnesium, Nickel and Titanium on the Electrochemical Behaviour of AA1050 Aluminum Alloys in Nitric Acid. The University of Manchester (United Kingdom).
- Witkowska, M. (2013). Interogation of the Manufacturing Route of Aluminium Aa 1050 Used in Lithographic Application. The University of Manchester (United Kingdom).
- 8. Czerwinski, F. (2024). Aluminum alloys for electrical engineering: a review. *Journal of Materials Science*, 1-46.
- 9. Czerwinski, F. (2024). Aluminum alloys for electrical engineering: a review. *Journal of Materials Science*, 1-46.

- 10. Eslami, M. (2019). Surface Treatments to Protect Conventional and Rheo-High Pressure Die Cast Al-Si Alloys from Corrosion (Doctoral dissertation, University of Trento).
- 11. Ononiwu, N. H. (2021). Machinability studies and characterization of aluminium matrix reinforced with fly-ash and carbonized eggshells (Doctoral dissertation, University of Johannesburg).
- 12. Ebnesajjad, S., & Ebnesajjad, C. (2013). Surface treatment of materials for adhesive bonding. William Andrew.
- 13. Surface treatment is economically necessary to prevent degradation, as industry data estimates that corrosion costs alone account for 3-4% of GDP in many industrialized nations.
- 14. Wood, R. J., & Lu, P. (2024). Coatings and surface modification of alloys for tribo-corrosion applications. *Coatings*, 14(1), 99.
- Kuang, W., Miao, Q., Ding, W., & Li, H. (2022). A short review on the influence of mechanical machining on tribological and wear behavior of components. *The International Journal of Advanced Manufacturing Technology*, *120*(3), 1401-1413.
- Khanna, V. K. (2010). Adhesion–delamination phenomena at the surfaces and interfaces in microelectronics and MEMS structures and packaged devices. *Journal of Physics D: Applied Physics*, 44(3), 034004.
- 17. Steen, W. M., & Watkins, K. G. (1993). Coating by laser surface treatment. *Le Journal de Physique IV*, 3(C9), C9-581.
- Jeyaprakash, N., Yang, C. H., & Kumar, D. R. (2020). Laser surface modification of materials. *Practical Applications of Laser Ablation*, 61-81.
- Montealegre, M. A., Castro, G., Rey, P., Arias, J. L., Vázquez, P., & González, M. (2010). Surface treatments by laser technology. *Contemporary Materials*, 1(1), 19-30.
- 20. Holmberg, K., & Matthews, A. (2009). Coatings tribology: properties, mechanisms, techniques and applications in surface engineering. Elsevier.
- 21. Selvan, J. S., Subramanian, K., & Nath, A. K. (1999). Effect of laser surface hardening on

En18 (AISI 5135) steel. Journal of Materials Processing Technology, 91(1-3), 29-36.

- 22. Asnafi, N. (2021). Tool and die making, surface treatment, and repair by laser-based additive processes. *BHM Berg-und Hüttenmännische Monatshefte*, 166(5), 225-236.
- Masato, D., Piccolo, L., Lucchetta, G., & Sorgato, M. (2022). Texturing Technologies for Plastics Injection Molding: A Review. *Micromachines*, 13(8), 1211.
- 24. Bose, S., Robertson, S. F., & Bandyopadhyay, A. (2018). Surface modification of biomaterials and biomedical devices using additive manufacturing. *Acta biomaterialia*, *66*, 6-22.
- Wang, Q., Zhou, P., Liu, S., Attarilar, S., Ma, R. L. W., Zhong, Y., & Wang, L. (2020). Multiscale surface treatments of titanium implants for rapid osseointegration: a review. *Nanomaterials*, 10(6), 1244.
- Arulvel, S., Jain, A., Kandasamy, J., & Singhal, M. (2023). Laser processing techniques for surface property enhancement: Focus on material advancement. *Surfaces and Interfaces*, 42, 103293.
- 27. Liu, Y., Chen, X., & Xin, J. H. (2009). Can superhydrophobic surfaces repel hot water?. *Journal of Materials Chemistry*, 19(31), 5602-5611
- Roach, P., Shirtcliffe, N. J., & Newton, M. I. (2008). Progess in superhydrophobic surface development. *Soft matter*, 4(2), 224-240.
- Jeevahan, J., Chandrasekaran, M., Britto Joseph, G., Durairaj, R. B., & Mageshwaran, G. J. J. O. C. T. (2018). Superhydrophobic surfaces: a review on fundamentals, applications, and challenges. *Journal of Coatings Technology and Research*, 15, 231-250.
- Bai, Y., Zhang, H., Shao, Y., Zhang, H., & Zhu, J. (2021). Recent progresses of superhydrophobic coatings in different application fields: An overview. *Coatings*, 11(2), 116.
- Pavithra, D., & Doble, M. (2008). Biofilm formation, bacterial adhesion and host response on polymeric implants—issues and prevention. *Biomedical Materials*, 3(3), 034003.

- Keley, M. M. (2017). Super–Hydrophobic Surfaces Based On Fluorinated Carbon And Its Application On Avoiding Ice– Accretion (Doctoral dissertation, Universidade Federal do Rio de Janeiro).
- 33. Buczkowska, K. (2023). Hydrophobic Protection for Building Materials.
- Yu, Q., Xiong, R., Li, C., & Pecht, M. G. (2019). Water-resistant smartphone technologies. *IEEE* Access, 7, 42757-42773.
- 35. Ferrari, M., & Benedetti, A. (2015). Superhydrophobic surfaces for applications in seawater. *Advances in colloid and interface science*, 222, 291-304.
- 36. Aisswarya, K. (2016). Rough surface preparation through chemical etching for superhydrophobicity/Aisswarya Kumaran (Doctoral dissertation, University of Malaya).
- 37. Kozhukharov, S., Girginov, C., Tsanev, A., & Petrova, M. (2019). Elucidation of the anodization and silver incorporation impact on the surface properties of AA1050 aluminum alloy. *Journal of The Electrochemical Society*, *166*(10), C231.