11th ICNTC CONFERENCE
INTERNATIONAL CONFERENCE ON NEW TRENDS IN CHEMISTRY

11th INTERNATIONAL CONFERENCE ON NEW TRENDS IN CHEMISTRY
25 – 27 APRIL 2025

11th ICNTC BOOK OF PROCEEDINGS

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ICNTC Conference 2025

11th International Conference on New Trends in Chemistry

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Prof. Dr. Dolunay ŞAKAR

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Yıldız Technical University – Turkiye
Conference Chair

Prof. Dr. Yelda Yalcin Gurkan
Namik Kemal University – Turkiye
Chemistry Department

Dear Colleagues,

I am honoured to invite and send you this call for papers on behalf of Conference Organisation Board of “ **11th International Conference on New Trends in Chemistry**”, to be held in Bologna, Italy on the dates between April 25- 27, 2025.

Limited number of Papers and Posters with the below mentioned topics will be accepted for our conference:

- Analytical Chemistry
- Bio Chemistry
- Computational Chemistry
- Chemistry Education
- Environmental Chemistry
- Food Chemistry
- Forensic chemistry
- Inorganic Chemistry
- Material Chemistry
- Organic Chemistry
- Physical Chemistry
- Polymer Chemistry and Applications
- Pharmaceutical Chemsitry

The selected papers which are presented as oral in the conference will be published in an international peer-reviewed journal which is indexed by SCOPUS as Q4. Each manuscript will have DOI Numbers.

We kindly wait for your attendance to our conference to be held on 25-27 April 2025.

All informations are available in conference web site. For more information please do not hesitate to contact us. info@icntcconference.com

Respectfully Yours,

On Behalf of the Organization Committee of ICNTC Conference

Prof. Dr. Dolunay ŞAKAR
11th ICNTC 2023 | Conference Chair
Yıldız Technical University – Istanbul / Turkiye
Chemistry Department

25 APRIL 2025 FRIDAY
MADRID MEETING ROOM

Welcome Speech / 09:00 – 09:10
Prof. Dr. Dolunay ŞAKAR / Conference Chair
Yıldız Technical University - Istanbul , Turkiye

Keynote Speech / 09:10 – 09:40
Prof. Dr. Julia LU
Toronto Metropolitan University, Canada

Speech Title: Artificial Intelligence (AI) in Chemistry in Higher Education

SESSION A (09:40 – 10:40)
SESSION CHAIR: Prof. Dr. Sevil YÜCEL
MADRID MEETING ROOM

09:40 – 10:00
PAPER TITLE : Catalytic Cycloaddition of Carbon Dioxide and Epoxides by Chloro-Iron and Cobalt Complexes with Functionalized Quadridentate Ligands: Structural Effect and DFT Calculation
AUTHOR(S) : **Adnan S. Abu-SURRAH**, Haitham H. Al-SA'DONI, Hamzeh M. ABDEL-HALIM, Khaleel I. ASSAF

10:00 – 10:20
PAPER TITLE : Determining the Tracer Performance of $ZnFe_2$O**4** Modified with Different Costing Agents for Magnetic Particle Imaging
AUTHOR(S) : Gülsüm ÇALIŞKAN, Muhammad IRFAN, **Nurcan DOGAN**

10:20 – 10:40
PAPER TITLE : Rational Protacs Design by Modelling the Ternary Complex Formation
AUTHOR(S) : **Tommaso D'ANGELI**, Massimo BARONI, Tommaso PALOMBA, Gabriele CRUCIANI

TEA & COFFEE BREAK 10:40 – 11:00

25 APRIL 2025 FRIDAY

SESSION B (11:00 – 12:40)
SESSION CHAIR: Prof. Dr. Nurcan DOĞAN
MADRID MEETING ROOM

11:00 – 11:20
PAPER TITLE : Production of 48S Bioactive Glasses Enriched with Various Metal Ions and Evaluation of Their Bioactivity Properties
AUTHOR(S) : **Sevil YÜCEL**, Gözde KELEŞ, Ali Can ÖZARSLAN, Cem ÖZEL, İlkay Turhan KARA, Mine KUCAK

11:20 – 11:40
PAPER TITLE : The Efficiency of Cadmium Extraction from Various Soil Types and Model Systems with the Selected Environmental Friendly Chelators
AUTHOR(S) : **Pawel MISKOWIEC**, Anna STREKALOVSKAYA

11:40 – 12:00
PAPER TITLE : Enhanced Ultrafiltration Membranes: Fabrication of Polysulfone Membranes with Functionalized TiO_2 Nanotubes for Superior Permeability and Fouling Resistance
AUTHOR(S) **: Ibrahim Hotan ALSOHAIMI**

12:00 – 12:20
PAPER TITLE : Development of the Injectable Bone Grafts
AUTHOR(S) : **Cem ÖZEL**, Ali Can ÖZARSLAN, Sevil YÜCEL

12:20 – 12:40
PAPER TITLE : Encapsulating Bergamot and Ginger Essential Oils in Binary Blends of Zein and Casein Polymers
AUTHOR(S) : Cansu NAYIR, **Yasar Andelib AYDIN**

LUNCH BREAK 12:40 – 13:20

13:30 Departure from Conference Venue for Florence Tour and Conference Dinner

23:30 Back to Hotel After Conference Dinner & Tour

26 APRIL 2025 SATURDAY

SESSION C (09:00 – 10:40)
SESSION CHAIR: Assoc. Prof. Dr. İlknur KÜÇÜK
MADRID MEETING ROOM

Session C & D are parallel sessions!

09:00 – 09:20
PAPER TITLE : Computational Evaluation of Cymenes: Substituent Group Effect, Pharmacokinetics and Drug-likeness
AUTHOR(S) : **Goncagül SERDAROĞLU**

09:20 – 09:40
PAPER TITLE : Theoretical Examination of Paroxetine Hcl, the Active Ingredient of the Drug Marketed As Paxil, Used in Antidepressant Treatment, Using the Dft Method
AUTHOR(S) : **Bahar EREN**, Yelda YALCIN GURKAN

09:40 – 10:00
PAPER TITLE : Synthesis and Characterization of Copper (II) Complex and Its Catalytic Activity on C(sp^3)-H Oxidation Reactions
AUTHOR(S) : **Yalçın KILIÇ**, İbrahim KANİ

10:00 – 10:20
PAPER TITLE : Investigation of the Degradation Reaction Kinetics of Cefalexin by Hydroxyl Radical
AUTHOR(S) : **Seyda AYDOGDU**, Arzu HATIPOGLU

10:20 – 10:40
PAPER TITLE : Pesticides in Albanian Vegetable Farming: Challenges and Strategies for Sustainable Agriculture
AUTHOR(S) : **Matilda LIKAJ**, Elda MARKU, Ridvana MEDIU, Jonida TAHIRAJ, Sonila SHEHU

TEA & COFFEE BREAK 10:40 – 11:00

26 APRIL 2025 SATURDAY

SESSION D (09:00 – 10:40)
SESSION CHAIR: Prof. Dr. Azmi Seyhun KIPÇAK
OSLO MEETING ROOM

Session C & D are parallel sessions!

09:00 – 09:20
PAPER TITLE : Challenges in Icp-ms Analysis of Elemental Impurities in Pharmaceutical Products: Methodological and Analytical Considerations
AUTHOR(S) :**Biljana BALABANOVA**

09:20 – 09:40
PAPER TITLE : Development of a High-performance Liquid Chromatography (hplc) Method for Coumarin Quantification in Medicinal Plants Via Soxhlet Extraction
AUTHOR(S) : Sinan ŞİMŞEK, **Emel AKYOL**, İlknur KÜÇÜK

09:40 – 10:00
PAPER TITLE : Hydrogen Production from Sodium Borohydride in Continuous Hydrolysis Systems: the Effect of Recirculation
AUTHOR(S) : Emine ÇİFTÇİ, Ayça TÜKENMEZ, Esra BALKANLI ÜNLÜ, **Halit Eren FİGEN**

10:00 – 10:20
PAPER TITLE : Valorisation of Polycationic Metals from Acid Mine Drainage for Sustainable Wastewater Treatment: a Circular Economy Approach
AUTHOR(S) : Khathutshelo Lilith MUEDI, Job Tatenda TENDENEDZAI, Vhahangwele MASINDI, Nils Hendrik HANEKLAUS, **Hendrik Gideon BRINK**

10:20 – 10:40
PAPER TITLE : Assessment of the Effects of Iron (III) Oxide Nanoparticles and Iron (III) Chloride on Glutathione Reductase Activities
AUTHOR(S) **: Hasan KARADAĞ**, Özge FIRAT

TEA & COFFEE BREAK 10:40 – 11:00

26 APRIL 2025 SATURDAY

SESSION E (11:00 – 12:40)
SESSION CHAIR: Prof. Dr. Aysel KANTÜRK FİGEN
MADRID MEETING ROOM

Session E & F are parallel sessions!

11:00 – 11:20
PAPER TITLE : Utilization of Inductively Coupled Plasma (ICP) in Monitoring and Analysis of Some Heavy Metals in Jordanian Dams
AUTHOR(S) : **Ayman A. ISSA**, Mohammad H. ZAHLAN

11:20 – 11:40
PAPER TITLE : Effect of Different Drying Technics on Drying Characteristics of Celeriac
AUTHOR(S) : Cansın KURT, **İlknur KÜÇÜK**, İbrahim DOYMAZ

11:40 – 12:00
PAPER TITLE : Life Cycle Assessment of Magnetic Carbon Based Adsorbant Synthesis
AUTHOR(S) : **Berfin Ekin ÜLGEN**, Zeynep TAŞ, Muhammed İberia AYDIN, Bilge COŞKUNER FİLİZ, Aysel KANTÜRK FİGEN

12:00 – 12:20
PAPER TITLE : Application of Gas Foaming Technique for Improving Porous Properties of Chitosan-polyvinyl Alcohol (cs-pva) Nanofiber Based Biodegradable Scaffolds
AUTHOR(S) : **Gizem ÖZDEMİR**, Demetnur ERKOYUNCU, Aysel KANTÜRK FİGEN

12:20 – 12:40
PAPER TITLE : Improved Biobutanol Recovery Through Mixed-matrix Pvdf Membrane with Hydrophobic Maf-6 As Filler
AUTHOR(S) : Rüveyda ÖZDEMİR, **Derya ÜNLÜ**

LUNCH BREAK 12:40 – 13:20

26 APRIL 2025 SATURDAY

SESSION F (11:00 – 12:40)
SESSION CHAIR: Prof. Dr. Goncagül SERDAROĞLU
OSLO MEETING ROOM

Session E & F are parallel sessions!

11:00 – 11:20
PAPER TITLE : Blanching and Osmotic Dehydration Effects on Lyophilised Shrimp
AUTHOR(S) : Zehra Özden ÖZYALÇIN, **Azmi Seyhun KIPÇAK**

11:20 – 11:40
PAPER TITLE : Nanomaterial-modified Carbon Paste Electrode-based Biosensor for the Investigation of Apigenin in Real Samples
AUTHOR(S) : Said Nur KAYRAN, **Hasret SUBAK**

11:40 – 12:00
PAPER TITLE : Eucalyptol-based Liquid-liquid Microextraction Coupled with Gas Chromatography for the Determination of Phthalates in Bottled Water Samples
AUTHOR(S) : **Natalia JATKOWSKA**

12:00 – 12:20
PAPER TITLE : Synthesis, Characterization, and Usage of Graphitic Carbon Nitride ($g-C_3N_4$) Species in pH-Controlled Drug Delivery
AUTHOR(S) : **Ümran GÜNTER,** Esra Maltas ÇAĞIL,Talha KURU, Emre ASLAN, Imren HATAY PATIR

12:20 – 12:40
PAPER TITLE : A Novel Perspective on Lateral Flow Assays: Fluorescence Protein-peptide Aptamers
AUTHOR(S) : Canan ÖZYURT, Serap EVRAN, Burhan BORA, İnci ULUDAĞ ANIL, **Mustafa Kemal SEZGİNTÜRK**

LUNCH BREAK 12:40 – 13:20

26 APRIL 2025 SATURDAY

SESSION G (13:20 – 15:00)
SESSION CHAIR: Prof. Dr. Emel AKYOL
MADRID MEETING ROOM

Session G & H are parallel sessions!

13:20 – 13:40
PAPER TITLE : Evaluation of Smart Packaging Functions of Black Carrot Extract with Polysaccharide-based Films
AUTHOR(S) : **Özde İPSALALI**, Filiz UĞUR NİGİZ

13:40 – 14:00
PAPER TITLE : In Silico Insight on Hyaluronic Acid and Boron-hyaluronate
AUTHOR(S) : **Goncagül SERDAROĞLU**, Melda BOLAT, Dursun Ali KÖSE, Zekeriya ÖZTEMÜR, Nihat KARAKUŞ

14:00 – 14:20
PAPER TITLE : Infrared Drying of Aronia Berries: the Effect of Sustainable Pretreatments on Drying Behavior
AUTHOR(S) : Berfin SALIK, Ata TAN, **Ekin KIPÇAK**

14:20 – 14:40
PAPER TITLE :The Impact of Metal Industry Waste on the Agricultural Development and Yield Increase of Tea Plants
AUTHOR(S) : Burcu Didem ÇORBACIOĞLU, **İlknur KÜÇÜK**

14:40 – 15:00
PAPER TITLE :Synthesis, Characterization and Photocatalytic Performance Of Modified $Cd_{0.7}Zn_{0.3}S$
AUTHOR(S) : İbrahim KABA, **Özge KERKEZ KUYUMCU**

TEA & COFFEE BREAK 15:00 – 15:30

26 APRIL 2025 SATURDAY

SESSION H (13:20 – 15:00)
SESSION CHAIR: Assoc. Prof. Dr. Halit Eren FİGEN
OSLO MEETING ROOM
Session G & H are parallel sessions!

13:20 – 13:40
PAPER TITLE : Evaluation of Different Fillers on Glass Fiber Reinforced Polymer Composites for the Automotive Industry
AUTHOR(S) : **Miray ÖZBAKIŞ**, Pınar TERZİOĞLU

13:40 – 14:00
PAPER TITLE : Mechanical Performance of Glass Fiber / Polyester Composites Containing Biofiller for the Automotive Applications
AUTHOR(S) : Miray ÖZBAKIŞ, **Pınar TERZİOĞLU**

14:00 – 14:20
PAPER TITLE : Obtaining Valuable Components from Various Citrus Product Wastes by Different Extraction Methods
AUTHOR(S) : **Deniz UYGUNÖZ**, Gamzenur ÇİFÇİ, Özgür Ozan DEMİRCİ, Emek MÖRÖYDOR DERUN

14:20 – 14:40
PAPER TITLE : Fabrication and Filtration of Arabic Gum Doped Electrospun Pla Membrane for Rejection of Gray Water Pollutants
AUTHOR(S) : **Seniyecan KAHRAMAN**, Ayşenur KATIRCI, Filiz UĞUR NİGİZ

14:40 – 15:00
PAPER TITLE : Green Tea-boron Nitrite Incorporated Pumpkin Pectin-alginate Food Packaging Film Preparation and Characterization
AUTHOR(S) : **Özde İPSALALI**, Filiz UĞUR NİGİZ

TEA & COFFEE BREAK 15:00 – 15:30

26 APRIL 2025 SATURDAY

SESSION I (15:30 – 17:10)
SESSION CHAIR: Assoc. Prof. Dr. Pınar TERZİOĞLU
MADRID MEETING ROOM

Session I & J are parallel sessions!

15:30 – 15:50
PAPER TITLE : The Effect of Modifiers on the Microstructure of Road Bitumen and Strength of Asphalt Concrete
AUTHOR(S) : **Yuliya BYZOVA,** Antonina Dyuryagina, Kirill Ostrovnoy, Tatyana Shirina

15:50 – 16:10
PAPER TITLE : Electrospun CuMOF/PLA Nanofibers as Biodegradable Antibacterial Membranes for Biomedical Applications
AUTHOR(S) : **Bilge Nur MEKTEPLİ**, Dilek DALGAKIRAN, Sennur DENİZ

16:10 – 16:30
PAPER TITLE : Development of L-asparaginase-enzyme Immobilized Cu(ii)-nanoparticles and Characterization of Binding Process Via Spr Sensor
AUTHOR(S) : **Monireh BAKHSHPOUR-YÜCEL**, Kübra KAYA, Samir Abbas Ali NOMA, Bilgen OSMAN

16:30 – 16:50
PAPER TITLE : Anticancer Drug Release from Graphene Oxide/bacterial Celluloses
AUTHOR(S) : **Melike KÜÇÜK**, Monireh BAKHSHPOUR-YÜCEL, Sine ÖZMEN TOĞAY, Emel TAMAHKAR IRMAK, Elif TÜMAY ÖZER, Bilgen OSMAN

16:50 – 17:10
PAPER TITLE : $NiCo_2O_4$/S,N-Codoped Graphene Oxide/Nafion/GCE Nanocomposite Electrode Material for Energy Storage Applications
AUTHOR(S) : **Nilüfer KOÇYİĞİT**, Melih Beşir ARVAS

TEA & COFFEE BREAK 17:10 – 17:45

26 APRIL 2025 SATURDAY

SESSION J (15:30 – 17:30)
SESSION CHAIR: Asst.Prof.Dr.Ekin KIPÇAK
PARIS MEETING ROOM

Session I & J are parallel sessions!

15:30 – 15:50
PAPER TITLE : Investigating the Properties of Recycled and Virgin Poly (ethylene terephthalate) Textured Yarns: Effect of Different Blending Ratios
AUTHOR(S) : **Simay ÖZBAKIŞ**, Ayça AYDIN, Pınar TERZİOĞLU

15:50 – 16:10
PAPER TITLE : Freeze Drying of Squid: A Study to Investigate the Effect of Different Pre-Treatments
AUTHOR(S) : **Zehra Özden ÖZYALÇIN**, Azmi Seyhun KIPÇAK

16:10 – 16:30
PAPER TITLE : Drug Analysis With Molecularly Imprinted Polymers From Ion Pair Complexes
AUTHOR(S) : **Ayşegül GÖLCÜ**

16:30 – 16:50
PAPER TITLE : Investigation of Metal Organic Framework Based Electrodes for Flow Capacitive Deionization Systems
AUTHOR(S) : Eren ÖZBAY, Ali Furkan ALBORA, Sadullah ÖZTÜRK, Arif KÖSEMEN, **Şahika Sena BAYAZIT**

16:50 – 17:10
PAPER TITLE : Development and Validation of HPLC Method Using Multivariate Optimization for the Simultaneous Determination of Niflumic Acid and Its Impurities
AUTHOR(S) : **Evridiki PİNGO**, Bürge AŞÇI

17:10 – 17:30
PAPER TITLE : Spectrophotometric Kinetic Assessment of Ruthenium (III) Catalyzed Oxidation of Aspirin by Hexacyanoferrate (III) in Alkaline Medium - A Mechanistic Pathway
AUTHOR(S) : Beena GUPTA, **Riya SAILANI**

TEA & COFFEE BREAK 17:30 – 17:45

26 APRIL 2025 SATURDAY

POSTER SESSION K (17:45 – 18:30)
SESSION CHAIRS: Prof. Dr. Julia LU, Prof. Dr. Sevil YÜCEL, Prof. Dr. Ayşegül GÖLCÜ
OSLO MEETING ROOM

Session K & L are parallel sessions!

PAPER TITLE : A New SPE Method Based on Polymeric Sorbent for the Determination of Organophosphorus Pesticides
AUTHOR(S) : **Gülfere ARSLAN**, Melike KÜÇÜK, Bilgen OSMAN, Elif TÜMAY ÖZER

PAPER TITLE : Development of Carbon Electrodes for Capacitive Deionization (cdi) Process
AUTHOR(S) : **Demet AÇIKGÜL**, Dilek DURANOĞLU DİNÇER

PAPER TITLE : New Environmentally Friendly Approach to Speciation Analysis of Selenium in Environmental Waters
AUTHOR(S) : **Malgorzata GRABARCZYK**, Cecylia WARDAK, Marzena FIALEK, Edyta WLAZLOWSKA

PAPER TITLE : Photocatalytic Degradation of Methylene Blue by Using Magnetic Photocatalyst
AUTHOR(S) : **Zeliha Betül KOL**, Dilek DURANOĞLU DİNÇER

PAPER TITLE : Removal of Oncology Group Epirubicin and Methotrexate Drugs from Wastewater by Dft Method
AUTHOR(S) : Mustafa Ernur BİLTEKİN, Burak GÜRKAN, **Yelda YALÇIN GÜRKAN**

PAPER TITLE : Synthesis of Hard Carbon from Different Biomass Sources As Electrode Material for Energy Storage Systems
AUTHOR(S) : **Derya ALTINTAŞ**, Yüksel BAYRAM, Murat ATEŞ, Ozan YÖRÜK

PAPER TITLE : Unlocking Soil Health: Carbon Determination in Agricultural Soils
AUTHOR(S) : **Biljana BALABANOVA**, V. ILIEVA, S. MITREV, K. PANEV, I. DONEV, A. PIPEREVSKI

PAPER TITLE: CO_2 As A Building Block İn The Synthesis Of Steroidal Oxazolidinones
AUTHOR(S) : **David ISPAN**, Aron KÜZDÖ, Reka GECSE, Rita SKODA-FÖLDES

PAPER TITLE : New Methoxy And Hydroxy Substituted Pyridyl Benzamides As Potential Ph Sensors
AUTHOR(S) : **Antonija MAMIC**, Robert VIANELLO, Kristina BUTKOVIC, Iva KULUSIC and Marijana HRANJEC

PAPER TITLE : The Effect of Alkali Pretreaments on the Drying Kinetics Of Blueberries
AUTHOR(S) : Beril GÜNORAL, Buse BEŞİKCİ, **Ekin KIPÇAK**

PAPER TITLE : Synthesis and Spectroscopic Investigation of Host-Guest Interactions in Ferrocenylpyrimidines
AUTHOR(S) : **Mark VARADI**, Matyas J. GAZSI, Rita SKODA-FÖLDES

PAPER TITLE: Preparation of 2-Oxazolidinone Derivatives Using Carbon Dioxide as C1 Building Block in Ionic Liquid Solvent
AUTHOR(S) : **Donat Adrian GACOV**, Rita SKODA-FOLDES

PAPER TITLE: Capillary Electrophoresis and Chemometrics
AUTHOR(S) : Andreas ZEMANN

PAPER TITLE Development and Validation of a RP-HPLC Method for Simultaneous Determination of Chlorhexidine Gluconate, Benzydamine Hydrochloride, and Cetylpyridinium Chloride
AUTHOR(S) : Gülşah ÇEBİ, **Evridiki PİNGO,** Bürge AŞÇI

PAPER TITLE:"Computational Study of Natural Substances in the Active Principles of Drugs: Reaction Between Berberine and Cisplatin – DFT and QSAR Investigations"
AUTHOR(S) : **BENSIRADJ Nour El Houda**, KARICHE Sihem, TIDJANI-RAHMOUNI Nabila

26 APRIL 2025 SATURDAY

POSTER SESSION L (17:45 – 18:30)
SESSION CHAIR: Prof.Dr. Adnan S. Abu-SURRAH, Prof.Dr.Emel AKYOL, Assoc.Prof.Dr.İlknur KÜÇÜK
MADRID MEETING ROOM

Session K & L are parallel sessions!

PAPER TITLE : Farma-bant: Obtaining a Wound Covering Membrane from Calendula Officinalis, Centella Asiatica and Carthamus Tinctorius Plant Extract Mixtures
AUTHOR(S) : **Merve Duru EVCİMEN**, Öznur YAŞA ŞAHİN

PAPER TITLE : An Overview on Chemical Profile of Salvia Officinalis Population from Llogara Mountain Area, South Albania
AUTHOR(S) : **Jonida SALIHILA**, Edvina ÇIPI, Aida DAMA, Kleva SHPATI, Aurora NAPUCE, Aurel NURO

PAPER TITLE :Calculation of Electronic Properties of Adrenaline, Dopamine, Melatonin, and Serotonin Using Dft
AUTHOR(S) :Barış KOÇANA, Şeyda AYDOĞDU, **Arzu HATİPOĞLU**

PAPER TITLE :Cucurbit[10]uril Binding of Heteroleptic Iridium(iii) Complexes : Synthesis and Photophysical Characterization
AUTHOR(S) :**Lubna ALRAWASHDEH**, Khaleel I. ASSAF, Shoruq MARABEH, Lynne WALLACE, Anthony DAY

PAPER TITLE :Determination of Fatty Acid Composition of Some Plant Oils and Solid Soap Production with These Oils
AUTHOR'S :**Temine SABUDAK**, Elif OZCAN

PAPER TITLE : Modification of Some Bioorganic Products with Organosilicon Compounds for Use in Industrial Production
AUTHOR'S : **Lali TABATADZE**, Darejan IREMASHVILI, Qetevan EBRALIDZE, Irine BOTCHORISHVILI, Natia SHENGELIA

PAPER TITLE :New Trends in Design of Potentiometric Sensors
AUTHOR(S) : **Cecylia WARDAK,** K. MORAWSKA, M. GRABARCZYK

PAPER TITLE :The Inhibitory Effect of Hicaz Pomegranate Peel Extract on Enzymatic Browning in Potatoes: a Natural Solution
AUTHOR(S) : **Hatice PALÜZAR,** Şebnem MUTLU, Aysen SUEKİNCİ YILMAZ, Şebnem SELEN İŞBİLİR

PAPER TITLE : Ring Opening Copolymerization of Exoxides with CO2 and Organic Anhydrides Promoted by Dinuclear [OSSO]-type Metal Complexes
AUTHOR(S) : **Fatemeh NIKNAM**

PAPER TITLE : Synthesis and Biological Activity of New Benzoxazole Iminocoumarines As pH Sensors

AUTHOR(S) : **Marina GALIC,** Robert VIANELLO, Leentje PERSONS, Dirk DAELEMANS, Mihailo BANJANAC, Tea BRUKETA, Ela GASPARIC and Marijana HRANJEC

PAPER TITLE: Synthetic Flavonoid BrCl-flav-an Alternative Solution to Combat ESKAPE Pathogens
AUTHOR(S) : **Laura Gabriella SARBU**

PAPER TITLE : Dibenzofuran and Dihydrobenzodioxin-Based Hosts for Improved Stability Blue TADF OLEDs
AUTHOR(S) : **Eigirdas SKUODIS**, Goda GRYBAUSKAITE, Kristupas BAGDONAS, Domantas BERENIS, Dovydas BANEVICIUS, Gediminas KREIZA, Karolis KAZLAUSKAS

PAPER TITLE : Design of an Electrochemical Biosensing Platform Based on Metal-Doped Carbon Quantum Dots for Detecting Gastric Cancer-Related Genes
AUTHORS : **Dilek ÖZTÜRK**, Mahmut DURMUŞ

PAPER TITLE : The Cytotoxic Properties of Some Tricyclic 1,3-Dithiolium Flavonoids
AUTHORS : **Mihail Lucian BIRSA**

PAPER TITLE **:** Adsorption Investigation of Disperse Orange 30 Dye on H_2SO_4 Functionalized Activated Carbon
AUTHOR(S) : Kübra TOPAÇ, Hüsnü Kemal GÜRAKIN, **Dolunay ŞAKAR**

PAPER TITLE **:** Kinetic Studies of Disperse Orange 30 Dye Removal via H_2SO_4-Activated Scrap Tyre Particles
AUTHOR(S) : Sinem KARAGÖZ, Hüsnü Kemal GÜRAKIN**, Dolunay ŞAKAR**

27 APRIL 2025 SUNDAY

SOCIAL PROGRAM

Contents

Catalytic cycloaddition of carbon dioxide and epoxides by chloro-iron and cobalt complexes with functionalized quadridentate salophen ligands

Adnan S. Abu-Surrah[1]*, Haitham H. Al-Sa'doni [2], Hamzeh M. Abdel-Halim[1], and Khaleel I. Assaf [3]

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Abstract

Transition metal complexes with quadridentate salophen ligands showed to be highly efficient as catalyst precursor toward the synthesis of cyclic carbonate via the coupling reaction of carbon dioxide and epoxides. Herein, a series of *penta*-coordinated complexes of iron(III) and cobalt(III) (**5-8**) with quadridentate Schiff bases bearing electron withdrawing (EWG)/ electron releasing (ERG) substituents in the backbone and the terminal phenyl groups were evaluated as catalysts for the coupling reaction of carbon dioxide with styrene oxide in the presence of Bu_4NBr under solvent-free conditions. Furthermore, the cobalt(III) complex **7**, which showed the best activity toward the catalytic reaction, was utilized for the coupling of 1-butene oxide and cyclohexene oxide with CO_2. The complex showed a notable activity for the coupling of 1-butene oxide with carbon dioxide.

Keywords: *Iron(III) complexes, cobalt(III) complexes, Schiff bases, cyclic carbonate, carbon dioxide*

1. Introduction

The synthesis of cyclic carbonates (CCs) has garnered significant attention during the last decades. [1,2,3,4,5,6,7] The coupling reaction of epoxides with carbon dioxide (CO_2) using various catalytic systems has been employed for CC synthesis. Salen complexes of different transition metals such as chromium, [8,9] cobalt, [10,11] ruthenium, [12] zinc, [13,14] aluminum, [15] and tin [16] have exhibited high catalytic activity in this reaction. Also, Co(III)- [17,18,19] and Cr(III)-based complexes [8] with multidentate Schiff bases and/or porphyrin ligands as catalytic systems for the production of both CCs and polycarbonates were reported.

Remarkably, most of the previously stated have square pyramidal geometry, with a halide ligand occupying the apical position. The applicability of the commonly used cobalt and chromium complexes may be limited due to their toxicity. Therefore, there is still a growing need for the development of safer, more effective catalytic systems that can balance energy efficiency and sustainability in the cyclic carbonate manufacturing process.

Some iron-based catalysts have been explored for the coupling reaction of epoxides and CO_2. [20,21,22] Octahedral iron(II) complex with tetradentate nitrogen ligand, N, N-bis(quinolin-2-ylmethylene)-1,2-cyclohexanediamineiron(II) chloride has shown promising results for the production of cyclic propylene carbonate from propylene oxide and CO_2. The catalyst system showed fairly high yields (82–100%, at 100°C and 15 bar of CO_2).[20] Bis(phenoxyiminato)Fe(III)-based complexes as catalysts for coupling of CO_2 with different epoxides using DMF as solvent have also been reported [21].

Previously, we reported on different transition metal complexes with flexible tetradentate salen ligands bearing diverse functionalities over the ligand backbone. The complexes have been used as catalyst precursors for the coupling of epoxides with CO_2. The iron(III)-based catalysts bearing electron-withdrawing substituents on the axial positions of the complexes showed the highest catalytic activity towards the coupling reaction of styrene oxide and CO_2 under similar reaction conditions.[23] The cost-efficiency and lower toxicity of iron complexes make them greener alternatives to toxic metal complexes used as catalyst precursors.

In the present study, we described the application of some iron(III) and cobalt(II) complexes as catalyst precursors for the coupling of styrene oxide with carbon dioxide. In these compounds, the metal center is coordinated with Schiff base ligands (**1-4**) containing electron withdrawing (EWG)/ electron releasing (ERG) substituents on both the backbone and the terminal positions. Compared to the conventional Schiff base salen ligands, these selected functionalized ligands may enable us to determine which has greater influence on the catalytic activity: the substituents present in the backbone of the ligand or those located on the axial positions. In addition, the catalytic coupling of the epoxides, 1-butene oxide and cyclohexene oxide with CO_2 was investigated.

2. Experimental

2.1. Materials and instrumentation

Synthetic reactions were performed using Schlenk techniques. iron(II) acetate, cobalt(II) acetate, 4,5-dimethyl-o-phenylenediamine, 4,5-dichloro-o-phenylenediamine, 5-chlorosalicylaldehyde and 4-(diethylamino)salicyl-aldehyde were acquired from Aldrich. The ligands **1-4** and their corresponding iron(III) (**5** and **6**) and cobalt(III) (**7** and **8**) complexes were prepared following a previously published procedure.[i] All chemicals were utilized just as supplied. Elemental analysis was carried out using HEKAtech EuroEA elemental analyzer. A Bruker ARX300 spectrometer was utilized to record the ^{1}H- and ^{13}C-NMR spectra using $CDCl_3$ as a solvent with TMS as an internal standard. Both measurements were carried out at room temperature

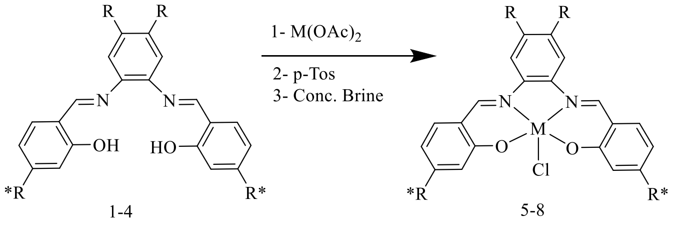
2.2. Coupling reactions

All reactions were carried out in a 300 ml autoclave. The catalyst, co-catalyst, and styrene epoxide (1:1:2556, respectively) were introduced into the vessel. Then the reactor was pressurized with 6 bars of CO_2 and heated to 120°C for 6 hours. After that, the reactor was allowed to cool, and the gas was released. The resulting residue was dissolved in CH_2Cl_2 and the remaining catalyst residues was removed by passing over a silica gel column. Evaporation of the solvent yielded the cyclic product.

Anal. Calcd. For: C 65.85, H 4.91; Found: C 65.92, H 4.61. GC/MS: one signal (164.8, 46%); IR (cm-1): 1776 (ν CO). ^{1}H NMR (CDCl3), 4.34 (t, 1H, CH_2), 4.80 (t, 1H, CH_2), 5.68 (t, 1H, CH), 7.37–7.43 (m, 5 H, arom.) ppm. ^{13}C NMR ($CDCl_3$), 71.1 (CH_2), 77.9 (CH), 154.8 (CO) ppm (CO).

3. Results and discussion

The free ligands (**1**–**4**) and the complexes (**5**-**8**) were prepared as previously described (Scheme 1). [23] The complexes were activated utilizing Bu_4NBr and used, under solvent-free conditions, to catalyze the cycloaddition reaction of carbon dioxide with styrene oxide. The results are summarized in Table 1.



1,5: M=Fe; R=CH$_3$; R*= Cl
2,6: M= Fe; R= Cl; R*= Cl
3,7: M= Co; R= CH$_3$;R*= Cl
4,8: M= Co; R= Cl; R*= Cl

Scheme 1: Structure of the *penta*-coordinated iron(III) (**5** and **6**) and cobalt(III) (**7** and **8**) complexes.

In general, differences in the metal and ligand substituents affect catalytic activity. The activities of the catalysts (TOF: mol product/mol catalyst.h) were recorded after purification of the isolated products (Table 1). The catalyst system is highly selectivity toward the production of cyclic carbonate at reasonably low pressure (6 bars).

The 1H NMR spectrum of the cyclic product shows resonances at 4.34 (1H, t) ppm, 4.80 (1H, t) ppm, 5.68 (1H, t) ppm and 7.37–7.43 (5H, m) ppm, corresponding to the CH, CH_2, CH_2 and aromatic groups, respectively. The ^{13}CNMR spectrum shows signals at 71.1, 77.9 and 154.8 ppm which can be attributed to the CH_2, CH, CO, respectively.

The parent cobalt(III) complex (**7** and **8**) showed higher activity (TON = 1280 and 1120, respectively) than the corresponding iron(III) complex (**3** and **4**, TON = 950 and 850, respectively). Upon addition of electron-donating groups (Me) to the phenyl group in the backbone of the salen ligands, a slight increase in the activity of the catalyst system was observed (Table 1, entries 1 and 3). This could be due to the increased nucleophilicity of the metal center in the presence of these groups. However, the presence of electron-withdrawing Cl groups (Table 1, entries 2 and 4) decreased the catalytic activity of both the iron(III)- and the cobalt(III)-based catalyst.

Table 1:Results of coupling reactions of carbon dioxide and styrene oxide using selected Fe(III)- and Co(III)-based complexes[a)]

Entry	*Complex code*	*Complex no.*	*Monomer*	*Productweight (g)*	*TON*	*TOF*
1	[(Me2-Salen-Cl2)FeCl]	5	SO	6.2	950	158.3
2	[(Cl2-Salen-Cl2)FeCl]	6	SO	5.1	850	140.4
3	[(Me2-Salen-Cl2)CoCl]	7	SO	8.3	1280	214.9
4	[(Cl2-Salen-Cl2)CoCl]	8	SO	6.7	1120	186.7
5	[(Me2-Salen-Cl2)CoCl]	7	BuO	5.6	1072	179
6	[(Me2-Salen-Cl2)CoCl]	7	CyO	0.80	120	21.0

a) Reaction conditions: solvent free; ratio of cat./co-cat (tetrabutylammonium bromide)/styrene oxide = 1/1/2556 (0.02 g); CO_2: 6 bars; temperature: 120ºC; time (6 hrs); *TON*: mole product/mole-cat.; TOF: mole product/mole cat. H. SO: styrene oxide; BuO: 1-butene oxide; CyO: cyclohexene oxide.

The catalyst system **7** was also screened for the coupling of carbon dioxide with 1-butene oxide and cyclohexene oxide (Table 1). The highest TON was observed for the coupling of 1-butene oxide with carbon dioxide. Low activity was observed for this system toward the coupling of carbon dioxide with cyclohexene oxide.

Conclusions

Iron(III) and cobalt(III) complexes with *ortho*-phenylenebis(salicylideneimine) based quadridentate Schiff base ligands (**5-8**) incorporating electron withdrawing (EWG)/ releasing substituents (ERG) on both the backbone and the axial positions were prepared. The complexes were utilized successfully as pre-catalysts for the coupling reactions of styrene oxide with carbon dioxide in the presence of Bu_4NBr under solvent-free conditions. The catalyst system described in this paper is highly selective for the production of cyclic carbonates. Preliminary experiments showed that the cobalt(III) based complexes are more active than the corresponding iron(III) complexes. Furthermore, the presence of methyl groups in the backbone of the salen ligand affect positively the activity of catalyst system. The cobalt complex, **7**, also proved to be active toward the coupling of 1-butene oxide with CO_2. DFT calculation revealed that changing the metal center largely affect the structural parameters and charge distribution.

Acknowledgments

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References

[1] Inoue S, Koinuma H, Tsuruta T. *Copolymerization of carbon dioxide and epoxide with organometallic compounds*. *Die Makromolekulare Chemie: Macromol. Chem. and Phys***.** **1969**, **130**, 210-220.

[2] Darensbourg D J, Matthew M H. *Catalysts for the reactions of epoxides and carbon dioxide*. *Coord Chem Rev* 1996, **153**: 155-174.

[3] Beckman E J. *Making Polymers from Carbon Dioxide*. *Science*, **1999**, **283**, 946-947.

[4] Klaus S, Lehenmeier M W, Anderson C E, Rieger B. *Recent advances in CO2/epoxide copolymerization: New strategies and cooperative mechanisms. Coord Chem Rev* 2011, **255**: 1460-1479.

[5] Chen F, Liu N, Dai B. *Iron(II) Bis-CNN Pincer Complex-Catalyzed Cyclic Carbonate Synthesis at Room Temperature. ACS Sustain Chem Eng* 2017, **5**: 9065-9075.

[6] Alves M, Grignard B, Mereau R, Jerome C, Tassaing T, Detrembleur C. *Organocatalyzed coupling of carbon dioxide with epoxides for the synthesis of cyclic carbonates: catalyst design and mechanistic studies*. *Catal. Sci. Technol*. 2017, **7**: 2651-2684.

[7] Li P, Cao Z. *Organometallics. Catalytic Preparation of Cyclic Carbonates from CO_2 and Epoxides by Metal–Porphyrin and −Corrole Complexes: Insight into Effects of Cocatalyst and meso-Substitution*. 2018, **37**: 406-414.

[8] Bu Z, Wang Z, Yang L, Cao S. *Synthesis of propylene carbonate from carbon dioxide using trans-dichlorotetrapyridineruthenium(II) as catalyst. Appl. Organomet. Chem*.2011, **24**: 813-816.

[9] Jijun X, Fei C, Min L, Ning L. *[ONSN]-type chromium complexes catalyzed coupling of CO_2 with epoxides. J. CO_2 Util* 2022, **62**: 102100.

[10] Decortes A, Belmonte M M, Benet-Buchholz J, Kleij, A W. *Efficient carbonate synthesis under mild conditions through cycloaddition of carbon dioxide to oxiranes using a Zn(salphen) catalyst. Chem Commun* 2010, **46**: 4580-4582.

[11] Suleman S, Younus H A, Ahmad N, Khattak Z A K, Ullah H, Park J, Han T, Yu B, Verpoort F. *Triazole based cobalt catalyst for CO_2 insertion into epoxide at ambient pressure*. *Appl Catal A: General* 2020, **591**: 117384.

[12] Fujita S I, Nishiura M, Arai M. *Ionic Liquids: The Synergistic Catalytic Effect in the Synthesis of Cyclic Carbonates. Catal. Letters* 2010, **135**: 263-268.

[13] North M, Pasquale R. *Mechanism of cyclic carbonate synthesis from epoxides and CO_2, Angewandte Chemie International Edition*, 2009, **48**: 2946-2948.

[14] Al-Qaisi F, Qaroush A K , Okashah I K, Eftaiha, A F, Vasko P, Alsoubani F, Repo T. *The Use of Sustainable Transition Metals for the Cycloaddition of Epoxides and CO_2 under Mild Reaction Conditions. Eur J Inorg Chem* 2023, **26**: e202200357.

[15] Sibaouih A, Ryan P, Axenov K V, Sundberg M R, Leskela M. Repo T *Efficient coupling of CO2 and epoxides with bis(phenoxyiminato) cobalt(III)/Lewis base catalysts*. *J Mol Catal A: Chem* 2009, **312**: 87-91.

[16] Kember M R, White A J P, Williams C K. *Highly Active Di- and Trimetallic Cobalt Catalysts for the Copolymerization of CHO and CO_2 at Atmospheric Pressure. Macromolecules* 2010, **43**: 2291-2298.

[17] Darensbourg D J, Mackiewicz R M. *Role of the Cocatalyst in the Copolymerization of CO_2 and Cyclohexene Oxide Utilizing Chromium Salen Complexes*. *J. Am. Chem. Soc.* 2005, **127**: 14026-14038.

[18] Cohen CT, Chu T, Coates GW. *Cobalt Catalysts for the Alternating Copolymerization of Propylene Oxide and Carbon Dioxide: Combining High Activity and Selectivity. J Am Chem Soc.* 2005, **127**: 10869-10878.

[19] Castro-Ruiz A, Grefe L, Mejía E, Suman S G. *Cobalt complexes with α-amino acid ligands catalyze the incorporation of CO_2 into cyclic carbonates*. *Dalton Trans*. 2023, **52**:4186-4199.

[20] Dengler JE, Lehenmeier MW, Klaus S, Anderson CE, Herdtweck E, Rieger B. *A One-Component Iron Catalyst for Cyclic Propylene Carbonate Synthesis. Eur J Inorg Chem*. 2011, **3**: 336-343.

[21] Al-Qaisi F, Genjang N, Nieger M, Repo T. *Synthesis, structure and catalytic activity of bis (phenoxyiminato) iron (III) complexes in coupling reaction of CO_2 and epoxides*. *Inorg Chim Acta*. 2016, **442**: 81-85.

[22] Abu-Surrah AS, Qaroush AK. *Benzothiophene-based complexes mediated formation of cyclic carbonates by cycloaddition of carbon dioxide to epoxides under mild solvent-free conditions. Transit. Met. Chem*, 2020, **45**:41-46.

[23] Abu-Surrah AS, Abdel-Halim HA, N. Abu-Shehab HAN, Al-Ramahi E. *Iron and cobalt salicylaldimine complexes as catalysts for epoxide and carbon dioxide coupling: effects of substituents on catalytic activity. Transit Met Chem*. 2017, **42**: 117-122.

[24] Lu X-B, Shi L, Wang Y-M, Zhang R, Zhang Y-J, Peng X-J, Zhan Z-C, Li B. *Design of Highly Active Binary Catalyst Systems for CO_2/Epoxide Copolymerization: Polymer Selectivity, Enantioselectivity, and Stereochemistry Control. J Am Chem Soc.* 2006, **128**:1664-1674.

AN OVERVIEW ON chemical profile of *salvia officinalis* population from llogara mountain area, south albania

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Abstract

In this study were analysed essential oil of *Salvia officinalis* samples from Llogara Mountain, located in Vlora area, at South Albania. Wild population of *Salvia officinalis* are widespread almost in all Albanian territory, which is one of the main exporter countries in Europe for this plant. Sage plants has a long history of medicinal and culinary use, and as an ornamental garden plant. Areal parts of sage herbs are known to have disease preventing, and health promoting properties because contain many chemical compounds (minerals, vitamins, terpenes, flavones, etc). Salvia herbs were collected in eight different stations of Llogara Mountain in the end of the June, for a five years period (2020-2024). The air dried plant samples were subjected to European Pharmacopoeia apparatus (Clevenger type) for 4 hours to obtain *Salvia officinalis* essential oil. The chemical composition of the essential oils was analysed using GC/FID technique. VF-1ms capillary column were used for separation of compounds.

Alpha- and beta-Thujone were identified as main constituents and all analysed *Salvia officinalis* samples from Llogara Mountain. Their total concentrations ranged between 31.3 and 37.2 %. The differences between stations and among the study period can be related with precipitation, sunny hours, air temperatures, changes in soil composition, etc. Even though it was not observed significant changes in the chemical profile of sage plants for analysed samples, climate changes and human activity can impact directly on the physiological processes of plants as well as at their chemical profile. Sage samples from Llogara Mountain had similar profile with other population from Albania as well as from Balkan area.

Key Words: *Llogara Mountain, Salvia officinalis, Essential oils, α-and β-Thujone, GC/FID*

1. Introduction

Albania is part of Balkan Peninsula as well as it is part of the Mediterranean area. For its favorable geographical position, our country is rich in vegetation, with about 3250 different species of plants. This development of flora is favored by the variation of territory from fields to hilly, and mountainous relief, as well as by the climate with vertical and horizontal changes. About 350 species are aromatic and medicinal plants, which constitute a good, important natural economic resource for the country but it is still not fully exploited [1]. The medicinal plants of our country are distinguished by their active ingredients and essential oils. These plants are known and mentioned since ancient times. They have been widely used in traditional medicine and in cooking [1, 2, 3, 4]. Albanian is one of the main exporter countries in Europe for sage plants and its essential oil. The activity of collecting aromatic-medicinal plants constitutes one of the main sources of income for the livelihood of poor families in rural areas. This is the main activity from which income is generated for the livelihood of these families. Many farmers want to plant their low-fertility lands with medicinal, essential oil and tannin plants. This would be a very good solution, especially in abandoned agricultural areas that are impossible to treat with agricultural machinery and irrigation water [5, 6, 7].

Salvia officinalis is a member of the family Lamiaceae and is native to the Mediterranean region, though it has naturalized in many places throughout the world. Wild population of *Salvia officinalis* are widespread in all Albanian territory. *S. officinalis* plant has been used since ancient times for snakebites, increasing women's fertility, and more. The plant had a high reputation throughout the middle-ages, with many sayings referring to its healing properties and value. It was sometimes called *S. salvatrix* (sage the savior). Sage is recommended as a diuretic, hemostatic, and tonic. Sage herb parts have many notable plant-derived chemical compounds, essential oils, minerals, vitamins that are known to have disease preventing, and health promoting properties. The prime biologically active component of common sage appears to be its essential oil, which mainly contains ketones; α-Thujone, and β-Thujone. In addition, sage leaf ect. Altogether, these compounds are known to have counter-irritant, anti-inflammatory, anti-allergic, anti-fungal and anti-septic properties. Sage leaves are a good source of antioxidant vitamin, vitamin-C which are at adequate levels in the body help maintain the integrity of blood vessels, skin, organs, and bones. It has wide uses in Albania, mainly as tea or as a food ingredient. It is often used to combat colds, sore throats, chest pains, menstrual cycle problems, etc [4 – 10]. The Llogara Mountain area is known for the growth and collection of many medicinal herbs such as *Salvia officinalis, Sideritis raeseri, Oregano vulgaris, Hypericum perforatum, Melisa officinalis,* etc. The suitable climate favors the growth of these herbs which are also known for their quality in our country [6, 8].

2. MATERIAL AND METHODS

2.1 Sampling of Salvia officinalis samples from Llogara Mountain

Essential oil of wild populations of Salvia officinalis samples from Llogara Mountain, located in Vlora area (South Albania) were analyzed in this study. Salvia herbs were sampled at the end of June, for a five years period (2020-2024). Sage samples were collected each year in 8 different stations of Llogara Mountain. The sage sampling stations were at different altitudes of 1000 – 1500 m altitude from both sides of Llogara Mountain starting from Dukat to Dhermi stations. The areal parts of the sage plants were air dried in a dark room for conserving their morphological characteristics [4 – 11].

2.2 Isolation of Salvia officinalis essential oil

Dried plant material (50 g of *Salvia Officinalis*) was subjected to hydro-distillation for 4 hours, using a modified Clevenger-type apparatus to produce essential oil. 2 ml Toluene was added to the balloon for isolation of *Salvia officinalis* essential oils. The oil was dried by anhydrous sodium-sulphate (Na_2SO_4) and kept sealed in dark glass vial at +8°C until use [6 – 8].

2.3 Apparatus and chromatography

Gas chromatographic analyses of *Salvia officinalis* essential oil were realized with a Varian 450 GC instrument equipped with a flame ionization detector and split/splitless injector. The temperature of injector was 280°C. 1 ul of *Salvia officinalis* essential oil diluted in Toluene was injected in split mode (1:50). A temperature for FID was held at 300°C. Nitrogen was used as carrier (1 ml/min) and make-up gas (25 ml/min). Hydrogen and air were flame detector gases with 30 ml/min and 300 ml/min, respectively. VF-1ms capillary column (30 m x 0.33 mm x 0.25 µm) was used to isolate compounds of *Salvia officinalis* essential oil. The oven temperature was programmed as follows: 40°C (held for 2 minutes) to 150°C (with 4°C/min), after that to 280°C with 10°C/min and held for 7 minutes. The identification of the compounds was based on comparison of their Kovats indices (KI), their retention times (RT) and the literature [6, 8, 10 17]. A mixture of n-alkanes from n-octane (C8) to eicosanes (C20) was used for calculation of KI.

RESULTS AND DISCUSSION

In this study, *Salvia officinalis* samples were taken from Llogara Mountain, for a five-year period (2020-2024). These sage samples were taken at the same stations (8 stations) for each year in the end of June. The stations were selected from Dukat to Dhermi on both sides of Llogara Mountain at an altitude of 1000 to 1500 m. This area is known for the growth and collection of sage and other medicinal plants. Essential oil of Salvia officinalis samples were analyzed using GC/FID technique. Table 1 gives the average percentages of the analyzed components for sage for this study period. The data have been averaged for each year to observe the changes over time in the sage plant from this area. In the chromatograms analyzed from these samples, 110-125 compounds were identified. In this study, the 20 main compounds were taken, which comprised 95.4% for the year 2024 to 97.9% for the year 2029. The data shown percentages for the total of identify peaks except for the peak of Toluene that was the solvent used for dilution of essential oil. The peaks lower than 0.05% were not present in this study. Some of the main compounds identified for all sage samples were: alpha-Thujone, beta-Thujone, Camphor, Cineole, Camphene, beta-Cariophyllene and alfa-Humulene. The main components in the essential oil of sage samples were monoterpenes. The total monoterpenes in the analyzed samples ranged from 88.9% for 2022 to 90.2% for 2023. The profile for the main groups of terpenes was: oxygenated monoterpenes (70.3 – 76.4%) > bicyclic monoterpenes (10.9 – 14.0%) > sesquiterpenes (5.7 – 7.7%) > monocyclic monoterpenes (0.9 – 4.3%) > aliphatic monoterpenes (1.1 – 1.7%) > aromatic monoterpenes (0.04 – 0.7%). It is noted that there is a slight difference in terms of the total and main groups of terpenes. This should be mainly related to external atmospheric factors such as humidity, amount of precipitation, amount of sunshine hours, soil composition, etc. However, their similar profile is related to the same origin of the plant. Alpha and beta-Thujones were the main constituents of sage for all samples. Their total ranged from 37.8% (2022) to 39.8% (2020). Note that, southern sage had a lower percentage of Thujones compared to other areas of Albania [6, 8]. Camphor was one of the main compounds found in higher percentages in southern sage with 18.2 – 23.4%. Cineole was another compound found in higher percentage compared to other terpenes. Its percentages were in the range between 6.9% (2024) to 10.3% (2021). Their presence (Camphor and Cineole) are important for the use of sage tea and its essential oil. These compounds (Thujones, Camphour and Cineole) belong to the oxygenated monoterpenes together with Borneol, Bornyl acetate, Linalool, Terpinen-4-ol and alpha-Terpineol. Oxygenated monoterpenes constitute the main group of terpenes. Their lowest level was in 2022 with 70.3% while the maximum was in 2023 with 76.4%. In general, it can be said that there is a preservation of the level of these compounds despite climatic changes from year to year (humidity, hours of sunshine, air temperatures, etc.). This should be mainly related to the genotype of the plant that grows in these areas. Another important group of compounds found in sage were the bicyclic monoterpenes (alpha+beta-Pinene, Camphene and cis-Sabinene hydrate) the maximum for which was in the years 2024 (14.0%) and the minimum in the year 2023 (10.9%). These compounds are more volatile than the other groups (they are the first to appear in the chromatogram of essential oil for sage samples) and therefore their fluctuation for different years should be related to the average air temperature for these years as well as to the drying temperature and the temperature at which the sage plants were stored before they were analyzed. This is also supported by the fact that for the same years when there is a decrease in the percentage of bicyclic monoterpenes, higher levels of sesquiterpenes (compounds with higher boiling points because of their molecular mass) were found. Thus, the maximum of sesquiterpenes (beta-Caryophyllene and alpha-Humulene) was found for the year 2023 (7.7%) while the minimum for the year 2024 (5.7%). Air humidity, amount of precipitation and hours of sunshine also have an impact on the changes observed for bicyclic monoterpenes and sesquiterpenes in the analyzed samples. For monocyclic terpenes (alpha+gamma-Terpinene and Limonene), it is noted that their maximum was for the years 2021 (3.2%) while their minimum was found for 2023 with 0.9%. Although the changes of this group are very large from year to year, we can say that they are mainly influenced by the relative percentages of other compounds. Also, atmospheric factors (humidity, amount of precipitation, etc.) or the method of storage/treatment of the samples before their analysis are not excluded. Aliphatic monoterpene (Myrcene) and aromatic monoterpene (para-Cymene) were found in non-significant percentages ($\leq$ 1%) to have an impact on the overall chemical profile of salvia samples from Llogara Mountain. The data obtained from this work showed that the chemical composition of Salvia officinalis from Southern Albania was almost the same chemical profile as other works reported from the other areas of Albania and Balkan countries as well as Mediterranean areas [5 – 8, 11, 13 – 17].

Table 1. Average data for chemical composition of Salvia officinalis from Llogara Mountain, South Albania

	Year 2020	**Year 2021**	**Year 2022**	**Year 2023**	**Year 2024**
alfa-Pinene	3.25 ± 0.82	3.41 ± 0.95	3.86 ± 1.35	3.76 ± 1.08	3.14 ± 1.15
Kamfen	7.85 ± 2.28	5.98 ± 1.25	6.19 ± 1.88	5.52 ± 1.38	8.34 ± 1.98
beta-Pinene	1.52 ± 0.56	3.14 ± 0.96	2.54 ± 0.86	1.62 ± 0.54	2.49 ± 0.56
Miricen	1.12 ± 0.48	1.71 ± 0.57	1.12 ± 0.52	1.32 ± 0.49	1.09 ± 0.35
Limonen	0.75 ± 0.24	0.25 ± 0.08	0.51 ± 0.15	0.08 ± 0.02	0.30 ± 0.07
alfa-Terpinen	1.94 ± 0.62	0.43 ± 0.14	3.60 ± 0.92	0.30 ± 0.07	1.94 ± 0.53
Cineole	8.52 ± 1.95	10.25 ± 2.65	9.63 ± 2.31	8.16 ± 2.08	6.93 ± 1.66
para-Cimen	0.11 ± 0.05	0.08 ± 0.02	0.58 ± 0.15	0.67 ± 0.17	0.04 ± 0.01
gama-Terpinen	0.52 ± 0.15	0.71 ± 0.26	0.20 ± 0.07	0.51 ± 0.19	0.42 ± 0.21
Cis-Sabinenhidrat	0.26 ± 0.06	0.11 ± 0.05	0.10 ± 0.04	0.03 ± 0.01	0.25 ± 0.06
Linaleol	0.28 ± 0.08	0.39 ± 0.15	0.11 ± 0.02	0.30 ± 0.08	0.34 ± 0.10
Alfa-Tujone	32.51 ± 4.28	31.05 ± 8.65	32.95 ± 9.05	35.18 ± 6.63	31.52 ± 7.41
Beta-Tujone	7.28 ± 1.24	7.64 ± 2.38	4.89 ± 2.36	4.26 ± 1.47	6.36 ± 1.69
Kamfur	18.24 ± 3.28	19.32 ± 4.18	18.57 ± 4.28	23.44 ± 5.24	21.25 ± 5.21
Borneol	2.74 ± 0.38	2.38 ± 0.85	2.23 ± 0.62	2.56 ± 0.98	3.36 ± 0.85
Terpinen-4-ol	0.62 ± 0.12	0.46 ± 0.16	0.49 ± 0.21	0.51 ± 0.15	1.11 ± 0.32
alfa-Terpineol	0.26 ± 0.08	0.35 ± 0.12	0.22 ± 0.08	0.22 ± 0.06	0.04 ± 0.01
Acetat Bornili	1.85 ± 0.42	2.45 ± 0.69	1.13 ± 0.55	1.77 ± 0.51	0.79 ± 0.26
beta-Karafilen	4.21 ± 0.65	3.68 ± 0.65	3.47 ± 0.91	4.05 ± 1.21	3.28 ± 0.82
Humulen	3.08 ± 0.61	3.85 ± 0.87	3.26 ± 0.86	3.68 ± 1.06	2.40 ± 0.55
Total	**96.91**	**97.63**	**95.62**	**97.91**	**95.39**
Oxygenated Monoterpene's	72.55	74.36	70.31	76.40	71.95
Bicyclic Monoterpene's	12.62	12.53	12.58	10.90	13.97
Monocyclic Monoterpene's	3.21	1.39	4.31	0.89	2.66
Aliphatic Monoterpene's	1.12	1.71	1.12	1.32	1.09
Aromatic Monoterpene's	0.11	0.08	0.58	0.67	0.04
Total Monoterpene's	89.61	90.07	88.89	90.18	89.71
Seskuiterpenes	7.29	7.53	6.73	7.73	5.68

CONCLUSIONS

Essential oil from wild population of *Salvia officinalis* from Llogara Mountain, South Albania were analyzed by using hydro-distillation (Clevengar apparatus) followed by GC/FID technique. Samples were taken in eight stations starting from Dukat to Dhermi in both sides of this mountain in an altitude of 1000 – 1500 m. Averages of results of a five years period (2020-2024) were presented in this study. The data were based on the 20 main compounds that were found for analyzed *Salvia officinalis* samples. The main compounds identified for all sage samples were: alpha-Thujone, beta-Thujone, Camphor, Cineole, Camphene, beta-Cariophyllene and alfa-Humulene. The main group of terpenes found in the analyzed samples was oxygenated monoterpenes. The highest level of them was found for sage samples taken in 2023. After them, bicyclic monoterpenes and sesquiterpenes were found. Monocyclic monoterpenes, aliphatic monoterpenes and aromatic monoterpenes were found in non-significant percentage. The differences found between the years are related to atmospheric factors (humidity, amount of precipitation, etc.) or the way the samples were stored/treated before their analysis. The chemical composition found for sage plants from Llogara Mountain has noticeable differences from northen areas of our country. The main difference is related to the lower percentages for alpha+beta-Thujone for essential oil of *Salvia officinalis* samples from South Albania while for Cineole and Camphor it is the opposite, these are found in abundance compared to northen areas. This difference can be related to climatic factors (humidity, amount of precipitation, altitude, hours of sunshine, etc.), geological factors (chemical content of the soils where the plants grow, slope, etc.) and biological factors (type of plants, genotype). Chemical profile of *Salvia officinalis* samples from South Albania area was almost the same with other reported studies from Balkan and Mediterranean areas.

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References

[1] Kathe W., Honnef S. & Heym A. (2003) Medicinal and Aromatic Plants in Albania, Bosnia-Herzegovina, Bulgaria, Croatia and Romania. A study of the collection of and trade in medicinal and aromatic plants (MAPs), relevant legislation and the potential of MAP use for financing nature conservation and protected areas” WWF Deutschland / TRAFFIC Europe-Germany.

[2] Bernotiene G., Nivinskiene O., Butkiene R., Mockute D. (2007), Essential oil composition variability in sage (Salvia officinalis L.), Chemija, 18, pp. 38–43.

[3] Burnie D. (1995) Wild Flowers of the Mediterranean. ISBN 0-7513-2761-1

[4] Oniga I., Oprean R., Toiu A., Benedec D., (2010) Chemical composition of the essential oil of *Salvia officinalis* L. from Romania. Reviste of Medico-Chirurgical Societatii de Medici si Naturalsi din Iasi; Nr. 114(2):593-599

[5] Perry N.B., Anderson R.E., Brennan N.J., Douglas M.H., Heaney A.J., McGimpsey J.A., Smallfield B.M. (1999) Essential Oils from Dalmatian Sage (Salvia officinalis L.): Variations among Individuals, Plant Parts, Seasons, and Sites; Journal of Agricultural Food and Chemistry, Vol47/5, pp.2048-2054

[6] Nuro A., Shëngjergji D., Napuçe A., Dama A., Salihila J., Dervishi A., Peci Dh., (2017) "Karakterizimi Kimik me Teknikën GC/FID për Vajin Esencial të Popullatave të Salvia Officinalius që Rriten në Shqipëri" Reviste shkencore e Albanian University "Optime". Nr 1 Fq 29-39 (2017) ISSN 2220 – 461X

[7] Miguel G., Cruz C., Faleiro M., Simões M., Figueiredo A., Barroso J., Pedro L. (2011), Salvia officinalis L. essential oils: effect of hydrodistillation time on the chemical composition, antioxidant and antimicrobial activities, Natural Product Research: Formerly Natural Product Letters, 25, pp. 526–541.

[8] Papa, S. Bacu, A. & Nuro, A. (2017). "Variation of monoterpene content and specific intergenic region of CP-DNA group natural populations of *Salvia Officinalis* L. of Northern Albania in a similar order". *European Journal of Biotechnology and Genetic Engineering, Nr. 4 (1)*, pp.1-10, ISSN 2397 - 2076

[9] Council of Europe (1997). European Pharmacopoeia, 3rd edn. Strasbourg: Council of Europe.

[10] David F., Scanlan F., Sandra P., Szelewski M. (2010), Analysis of essential oil compounds using retention time locked methods and retention time databases, Application, Agilent Technologies, 5988-6530EN.

[11] Khedher M.R.B., Khedher S.B., Ikbal Chaieb, Slim Tounsi S., Hammami M., (2017) Chemical composition and biological activities of *Salvia officinalis* essential oil from Tunisia, Experimental and clinical science journal; Nr. 16: 160–173; doi: 10.17179/excli2016-832

[12] Adams RP. (1995), Identification of essential oil components by gas chromatography/mass spectroscopy, Allured Publishing Cor-poration, Carol Stream: Illinois, USA.

[13] Bozin B., Mimica-Dukic N., Simin N., Anac-kov G. (2006), Characterization of the volatile composition of essential oils of some Lamiaceae spices and the antimicrobial and antioxidant activities of the entire oils, J. Agric. Food Chem.,54, 1822-1828.

[14] Daferera D.J., Ziogas B.N., Polissiou M.G. (2000), GC–MS analysis of essential oils from some greek aromatic plants and their fungitoxicity on Penicillium digitatum, Jour-nal of Agricultural Food Chemistry, 48, 2576–2581.

[15] Damyanova S., Mollova S., Stoyanova A., Gubenia O. (2016) Chemical composition of Salvia officinalis l. essential oil from Bulgaria; Ukrainian Food Journal. 2016. Volume 5. Issue 4, pp. 695-700

[16] Awen B.Z., Unnithan C.R., Ravi S., Kermagy A., Prabhu V., Hemlal H. (2011) Chemical Composition of Salvia officinalis Essential oil of Libya, Journal of Essential Oil Bearing Plants, Vol. 14/1. Pp.89-94

[17] Konig W.A., Bulow N., Saritas Y. (1999), Identification of sesquiterpene hydrocarbons by gas phase analytical methods, Flavour Fragr. J., 14, 367-378.

Assessment of the Effects of Iron (III) Oxide Nanoparticles and Iron (III) Chloride on Glutathione Reductase Activities

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Abstract

Iron (III) oxide nanoparticles (Fe_2O_3 NPs) and Iron (III) chloride ($FeCl_3$) are used in water treatment and many fields. In this study, we studied effect of Fe_2O_3 NPs and $FeCl_3$ on antioxidant enzyme glutathione reductase (GR) activity. In this work, 0, 25, 50, 100, 250 and 500 mg/L (ppm) concentrations of Fe_2O_3 NPs and $FeCl_3$ were applied to glutathione reductase (GR) from baker's yeast *(Saccharomyces cerevisiae) in vitro*. Percentage changes in GR enzyme activities with applying of 25, 50, 100, 250 and 500 mg/L Fe_2O_3 NPs were calculated as -2.42 ; -1.04 ; -5.65 ; -3.81 and -4.84, respectively. Percentage changes in GR enzyme activities with applying of 25, 50, 100, 250 and 500 mg/L $FeCl_3$ were calculated as +0.38 ; -0.25 ; -0.63 ; -4.76 and -14.64, respectively. As a result, it was determined in the study that when the Fe_2O_3 NPs concentration increased, there were slight statistically significant decreases in the GR activities at high concentrations (100, 250, 500 mg/L) compared to the control (N=3, $P<0.05$). When $FeCl_3$ concentration increased, a statistically significant decrease was determined in GR activity only at 500 mg/L $FeCl_3$ concentration compared to the control (N=3, $P<0.05$). $FeCl_3$ inhibited the GR enzyme more than Fe_2O_3 NPs *in vitro*.

Keywords: Iron (III) oxide nanoparticles; iron (III) chloride; glutathione reductase; nanoparticles

1. Introduction

Iron (III) oxide nanoparticles (Fe_2O_3 NPs) are used in magnetic hyperthermia as a cancer treatment method, drug delivery as carriers, magnetic resonance imaging, tissue engineering, cell separation, protein purification, enzyme immobilization, and biosensing [1,2]. Fe_2O_3 NPs are also used in environmental remediation and water treatment due to low cost technology [3].

Iron (III) chloride ($FeCl_3$) is also used in the drinking water production, the treatment of municipal and industrial wastewater and the processing of sludge as a coagulant and flocculant. Because of its reactive properties, iron (III) chloride is also used in the electronic industry as an etching agent [4].

Glutathione reductase (EC 1.8.1.7) (GR) is important antioxidant that produces reduced glutathione (GSH) in the presence of β-nicotinamide adenine dinucleotide 2'-phosphate reduced (NADPH) and oxidized glutathione (GSSG) [5].

Nanoparticle and chloride salts of iron are used in water treatment and other fields, and they affect living organisms and enzyme activities by being released into the environment. Iron compounds may inhibit GR activity. When GR is inhibited, the amount of GSH in the cell decreases and oxidizing agents harm the cells. Therefore, we decided to examine the effects of Fe_2O_3 NPs and $FeCl_3$ on the GR enzyme.

2. Materials and Methods

Iron (III) oxide (Fe_2O_3), crystalline, nanopowder, <50 nm particle size by BET (Brunauer–Emmett–Teller) analysis; iron (III) chloride ($FeCl_3$), sublimed grade, ≥99.9 % trace metals basis; baker's yeast *(Saccharomyces cerevisiae)* glutathione reductase were purchased from Sigma-Aldrich. L-glutathione oxidized ≥98 % was purchased from Serva. β-Nicotinamide adenine dinucleotide 2′-phosphate reduced with tetrasodium ≥95 % was obtained from Roth.

Fe_2O_3 NPs were sized at <50 nm by BET (Brunauer–Emmett–Teller) analysis with Sigma-Aldrich. However, the size of Fe_2O_3 NPs was found to be 31±11 nm as a result of TEM (Transmission Electron Microscope) (Hitachi High Tech HT 7700) analysis at East Anatolia High Technology Application and Research Center of Ataturk University (DAYTAM), Erzurum, Turkiye (Figure 1 and Table 1). With Malvern Zeta Sizer Nano ZSP at DAYTAM, potential, conductivity, polydispersity index, zeta size and mobility analysis were performed (Table 1).

Actual Fe amount was 31.875 ± 0.901 mg/L Fe by ICP-MS (inductively coupled plasma-mass spectrometry) (Perkin Elmer NexION 350X) at Central Research Laboratory Application and Research Center of Adiyaman University (ADYUMLAB), which corresponded to 91.143 ± 2.576 mg/L Fe_2O_3 of nominal 100 mg/L

Fe_2O_3 NPs. Limit of detection (LOD) and limit of quantitation (LOQ) were 2.703 and 9.010 mg/L Fe, respectively.

Fe amount by ICP-MS at ADYÜMLAB was 13.690 ± 0.289 mg/L Fe, corresponding to 39.763 ± 0.839 mg/L $FeCl_3$ of nominal 100 mg/L $FeCl_3$. LOD and LOQ were 0.867 and 2.890 mg/L Fe, respectively.

The recovery for internal standard yttrium-89 (Y89) after 18 measurements was 120.47 ± 9.99%.

Table 1: Characterization of Fe_2O_3 NPs

Particle properties of Fe_2O_3 NPs	**Method**	**Datas**
Size (nm)	BET (Brunauer–Emmett–Teller)	<50[a]
Particle Size Distribution (nm)	TEM	31±11[b]
Particle Size Distribution (nm)	Zeta Sizer	1408±165[b]
Polydispersity Index (PDI)	Zeta Sizer	0.476±0.060[b]
Zeta Potential (mV)	Zeta Sizer	6.590±0.253[b]
Mobility (µmcm/Vs)	Zeta Sizer	0.5163±0.0201[b]
Conductivity (mS/cm)	Zeta Sizer	0.0239± 0.0056[b]

Datas were mean ± standard deviation, [a]Knowledge from Sigma-Aldrich, [b]Knowledge from DAYTAM, Erzurum, Turkiye. For this, a dilute solution of Fe_2O_3 NPs in distilled water was prepared by vortexing, incubating in ultrasonic bath at 25 °C.for analysis at DAYTAM.

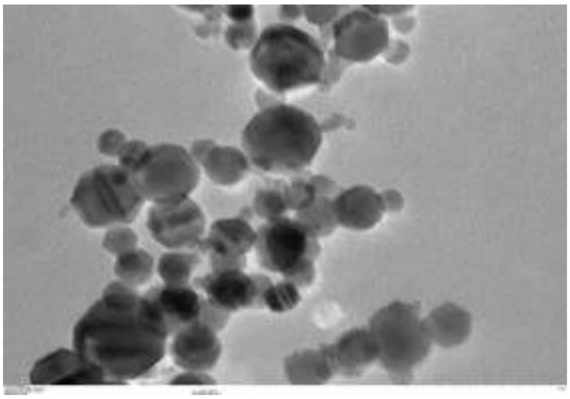

Figure 1. Image of Fe_2O_3 NPs taken by TEM

GR enzyme concentration was determined spectrophotometrically by method of Lowry et al. (1951) at 750 nm [6]. The detailed method is explained by Karadag (2021) [7].

GR activities were measured at 340 nm and at 37°C by UV-1800 UV-VIS incubated spectrophotometer (Shimadzu Scientific Instruments). The oxidation of NADPH to $NADP^+$ through GSSG was determined by reading the absorbances (Carlberg and Mannervik 1975) [8]. GR was expressed in U/mg protein. The detailed method is explained by Karadag (2021) [7].

Interaction of enzyme and iron compounds was performed as belows. 5000 mg/L Fe_2O_3 NPs and $FeCl_3$ stock solutions were each prepared in 2 mL distilled water. The solutions were placed in a 50 kHz, 60 W ultrasonic bath (ISOLAB) at 25°C, for 20 minutes for preventing aggregates. Then, concentrations of from 25 to 500 mg/L Fe_2O_3 NPs and $FeCl_3$ were adjusted by adding distilled water and 700 µL of GR solution prepared in potassium phosphate buffer pH 7.6. Mixture volume of GR, distilled water, iron compounds was 1000 µL. The mixtures were incubated for 10 minutes and GR activities were determined at room temperature. These *in vitro* adjustments were made to determine the effect of substances on enzyme activity (Karadag 2021) [7].

Statistical analysis

Datas were given as arithmetic mean ± standard deviation. We used one-way analysis of variance, Student–Newman–Keul's test at SPSS 22.

3. Results

When applying 0 to 500 mg/L solutions of Fe_2O_3 NPs to the GR, we determined slight statistically significant decreases in the GR activities at high concentrations (100, 250, 500 mg/L) according to the control (N=3, $P<0.05$) (Table 2). We calculated percentage changes in the GR activity with applying of 25, 50, 100, 250 and 500 mg/L of Fe_2O_3 NPs as -2.42 ; -1.04 ; -5.65 ; -3.81 and -4.84, respectively.

When applying 0 to 500 mg/L solutions of $FeCl_3$ to the GR, we determined a statistically significant decrease in the GR activity only at 500 mg/L $FeCl_3$ concentration according to the control (N=3, $P < 0.05$) (Table 2). We calculated percentage changes in the GR activitiy with applying of 25, 50, 100, 250 and 500 mg/L of $FeCl_3$ as +0.38 ; -0.25; -0.63 ; -4.76 and -14.64, respectively.

Table 2: Effects of iron (III) oxide nanoparticles and iron (III) chloride on glutathione reductase activities

Concentration (mg/L)	GR activities (U/mg) of Fe_2O_3 NPs	GR activities (U/mg) of $FeCl_3$
0	867 ± 12^{a}	799 ± 8^{a}
25	846 ± 12^{abc}	802 ± 15^{a}
50	858 ± 18^{ab}	797 ± 7^{a}
100	818 ± 14^{c}	794 ± 24^{a}
250	834 ± 15^{bc}	761 ± 7^{a}
500	825 ± 7^{c}	682 ± 27^{b}

The letters “a, b, c” belonging to Fe_2O_3 NPs in the Table are used to show the distinction between the groups. Different letters indicate statistical difference between activity levels (N=3, $P < 0.05$). The letter "b" belonging to $FeCl_3$ indicates a statistical difference between the activity levels (N=3, $P < 0.05$).

4. Discussion

In some works, it has been determined that iron oxide nanoparticles increase, decrease or do not change enzyme activity. For example; Reddy et al. (2017) [9] oral treated 30, 300 and 1000 mg/kg bw Fe_2O_3 NPs to rats for 28 days. They found an increase in antioxidant enzyme activities such as glutathione reductase, glutathione peroxidase, glutathione S transferase, and catalase. Gaharwar et al. (2017) [10] exposed 100, 200, 400 and 800 µg/mL Fe_2O_3 NPs for 24, 48 and 72 h to rats’ lymphocytes. They reported a decrease at amount of GSH (reduced glutathione) and a decrease at SOD (superoxide dismutase) and CAT (catalase) activities. Özgür et al. (2018) [11] evaluated the effects of various doses (50, 100, 200, 400 and 800 mg/L) of Fe_3O_4 NPs on rainbow trout (*Oncorhynchus mykiss*) spermatozoon at 4 °C for 24 h *in vitro*. They found a decrease at catalase (CAT) and superoxide dismutase (SOD) activitiy after 100 mg/L exposure of Fe_3O_4 NPs. Jarahian et al. (2018) [12] treated dose of 100 mg/kg body weight Fe_3O_4 NPs to male Wistar rats at presence of a constant magnetic field. They reported an unchanging activity at GPX (glutathione peroxidase) and SOD according to the control group at rat liver.

In some studies, it has been determined that iron compounds reduce glutathione reductase enzyme activity. For example; Kireçci (2018) [13] exposed 10 mg/L Fe to *Saccharomyces cerevisiae* culture medium. It was observed that glutathione reductase avtvity decreased compared to the control. Garcia-Alfonso et al. (1995) [14] treated 5mM iron (III) chloride to Vero cells cultured in microtitre plates. They found 56% decreased glutathione reductase activity according control activity in cells. Temel et al. (2018) [15] exposed different heavy metal ions (Co^{2+}, Zn^{2+}, Pb^{2+}, Fe^{2+}, Cd^{2+}, Al^{3+} and Fe^{3+}) (0.01–3 mM) to GR from japanese quail (*Coturnix coturnix japonica*) liver *in vitro*. They found non competitive inhibition of glutathione reductase at Fe^{3+} exposure. Swiergosz-Kowalewska et al. (2006) [16] collected voles (*Clethrionomys glareolus*) from three sites located 4, 8, and 30 km from the zinc-lead smelter. They found a negative relationship between iron and GR activity in the kidney.

A study on nanoparticles, consistent with the results we found, was conducted by Fırat et al. (2022) [17]. Fırat et al. exposed to 0.05 mg/L $CuSO_4$, CuO NPs, and $CuSO_4$+CuO NPs to freshwater fish, *Oreochromis niloticus* for 4 and 21 days. Especially at 21 days, $CuSO_4$ and CuO NPs, alone and combined, decreased tissue glutathione reductase, superoxide dismutase, catalase, glutathione-S-transferase avtivities and decreased amount of plasma total protein and glutathione.

5. Conclusion

Although iron compounds or nanoparticles increase, decrease or do not change enzyme activities, we observed slight statistically significant decreases regarding the effects of Fe_2O_3 NPs on GR activities. When applying 0 to 500 mg/L solutions of Fe_2O_3 NPs to the GR, the maximum inhibition was 5.65% and no major inhibition took place. This means that Fe_2O_3 NPs, which are used in many areas such as magnetic hyperthermia as a cancer treatment method, cause some inhibition of GR enzyme activities *in vitro*. In the case of $FeCl_3$, we observed a decrease in GR activities at high concentrations such as 500 mg/L $FeCl_3$. At 500 mg/L solutions of $FeCl_3$, the maximum inhibition was 14.64%. This means that $FeCl_3$, which is used in many areas such as drinking water production, domestic and industrial wastewater treatment, causes inhibition of GR enzyme activity at high concentrations *in vitro*. $FeCl_3$ inhibited the GR enzyme more than Fe_2O_3 NPs *in vitro*.

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References

[1] Mahmoudi, M., Hofmann, H., Rothen-Rutishauser, B., Petri-Fink, A. Assessing the in vitro and in vivo toxicity of superparamagnetic iron oxide nanoparticles. *Chem Rev* 2012; **112**: 2323–2338. https://doi.org/10.1021/cr2002596

[2] Rosen, J.E., Chan, L., Shieh, D.B., Gu, F.X. Iron oxide nanoparticles for targeted cancer imaging and diagnostics. *Nanomed-Nanotechnol* 2012; **8**: 275–290. https://doi.org/10.1016/j.nano.2011.08.017

[3] Baumann, J., Köser, J., Arndt, D., Filser, J. The coating makes the difference: acute effects of iron oxide nanoparticles on Daphnia magna. *Sci Total Environ* 2014; **484**: 176–184. https://doi.org/10.1016/j.scitotenv.2014.03.023

[4] Akzo Nobel Base Chemicals, *Water treatment chemicals* (2007, October 26). Retrieved from http://www.akzonobel.com/ic/system/images/AkzoNobel_WTCBrochureENG_tcm18-9982.pdf.

[5] Halliwell, B., Gutteridge, J.M.C. *Free radicals in biology and medicine.* 3rd edn. New York: Oxford University Press, 1999, p 143-144.

[6] Lowry, O.H., Rosebrough, N.J., Farr, A.L., Randall, R.J. Protein measurement with the folin phenol reagent. *J Biol Chem* 1951; **193**: 265–275. https://doi.org/10.1016/S0021-9258(19)52451-6

[7] Karadag, H. Inhibition of glutathione reductase activity from baker's yeast (Saccharomyces cerevisiae) by copper(II) oxide nanoparticles and copper(II) chloride. *B Environ Contam Tox* 2021; **106 (4)**: 647–651. https://doi.org/10.1007/s00128-021-03136-4

[8] Carlberg, I., Mannervik, B. Purification and characterization of the flavoenzyme glutathione reductase from rat liver. *J Biol Chem* 1975; **250(14)**: 5475–5480.

[9] Reddy, U.A., Prabhakar, P.V., Mahboob, M. Biomarkers of oxidative stress for in vivo assessment of toxicological effects of iron oxide nanoparticles. *Saudi J Biol Sci* 2017; **24(6)**: 1172–1180. https://doi.org/10.1016/j.sjbs.2015.09.029

[10] Gaharwar, U.S., Meena, R., and Rajamani, P. Iron oxide nanoparticles induced cytotoxicity, oxidative stress and DNA damage in lymphocytes. *J Appl Toxicol* 2017; **37(10)**: 1232–1244. https://doi.org/10.1002/jat.3485

[11] Özgür, M.E., Ulu, A., Balcıoğlu, S., Özcan, İ., Köytepe, S., and Ateş, B. The toxicity assessment of iron oxide (Fe_3O_4) nanoparticles on physical and biochemical quality of rainbow trout spermatozoon. *Toxics* 2018; **6(4)**: 62. https://doi.org/10.3390/toxics6040062

[12] Jarahian, A., Fatahian, S., Shahanipour, K. Investigation the effect of Fe_3O_4 nanoparticles on liver and stress oxidative parameters at the presence of magnetic field in rat. *Nanomed J* 2018; **5(2)**: 96-101. https://doi.org/10.22038/nmj.2018.005.006

[13] Kireççi, O.A. The effects of Mn, Mg, Cd, Fe Metals on the avtivitiy of antioxidant enzymes in Saccharomyces cerevisiae. *KSU J Agric Nat* 2018; **21(4)**: 520-528. https://doi.org/10.18016/ksudobil.359165

[14] Garcia-Alfonso, C., Sanz, P., Repetto, G., Repetto, M., and Lopez-Barea, J. Direct determination of glutathione reductase in cells cultured in microtitre plates as a biomarker for oxidative stress. *Atla-Altern Lab Anim* 1995; **23(4)**: 531-538. https://doi.org/10.1177/026119299502300417

[15] Temel, Y., Taher, S.S.M., Hamza, M.A., Shafeeq, I.H., Koçyiğit, U.M., Çiftci, M. Investigation of the inhibition effects of some metal ions on glutathione reductase enzyme from japanese quail (Coturnix coturnix japonica) liver. *Cumhuriyet Sci J* 2018; **39(3)**: 679-687. https://doi.org/10.17776/csj.414680

[16] Swiergosz-Kowalewska, R., Bednarska, A., Kafel, A. Glutathione levels and enzyme activity in the tissues of bank vole Clethrionomys glareolus chronically exposed to a mixture of metal contaminants. *Chemosphere* 2006; **65**: 963–974. https://doi.org/10.1016/j.chemosphere.2006.03.040

[17] Fırat, Ö., Erol, R., and Fırat, Ö. Effects of individual and co-exposure of copper oxide nanoparticles and copper sulphate on Nile Tilapia Oreochromis niloticus: nanoparticles enhance pesticide biochemical toxicity. *Acta Chim Slov* 2022; **69**: 81–90. http://dx.doi.org/10.17344/acsi.2021.6995

PESTICIDES IN ALBANIAN VEGETABLE FARMING: CHALLENGES AND STRATEGIES FOR SUSTAINABLE AGRICULTURE

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Abstract

The widespread use of pesticides in vegetable farming raises concerns about food safety, environmental contamination, and human health risks. This study aims to assess pesticide use practices among Albanian farmers, evaluate their knowledge of pesticide application and safety measures, and identify key challenges to promoting sustainable agriculture.

A structured survey was conducted with vegetable farmers across various regions of Albania to collect data on pesticide selection, application practices, adherence to safety measures, and awareness of associated health risks. Face-to-face interviews were used to gather responses, and statistical analyses were performed to identify trends and correlations between farmer knowledge, risk perception, and pesticide use behavior.

Findings indicate that while many farmers recognize the risks associated with pesticide use, knowledge gaps persist due to limited training and access to information. A significant portion of respondents were unaware of restrictions on certain pesticides and had not received formal training on their proper application. Although farmers acknowledged the negative effects of pesticides on food safety, health, and the environment, adherence to protective measures, particularly the use of personal protective equipment (PPE), remained insufficient. The study also highlights challenges such as weak regulatory enforcement, economic constraints, and the need for improved farmer education programs.

Addressing pesticide-related risks in Albanian vegetable farming requires a multifaceted approach, including continuous training for farmers, increased access to safer alternatives, and stronger regulatory enforcement. Enhancing farmers' knowledge and providing adequate resources are essential steps toward ensuring food safety, protecting public health, and promoting sustainable agricultural practices in Albania.

***Key Words**: Pesticide residues, Food safety, Sustainable agriculture, Vegetable farming, Risk perception*

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1. Introduction

The responsible use of pesticides is essential for food security and environmental protection. Pesticides should be used sustainably, minimizing risks to human health and ecosystems which relies on plants health (FAO, 2022).

In order to mitigate the risks linked to pesticide application, the European Union has enacted a comprehensive legislative framework, which defines rules for the approval of active substances, their uses in plant protection products and their permissible residues in food [1]. Plant protection products must be utilized appropriately, ensuring adherence to best practices in plant protection. Proper use encompasses the application of good plant protection principles and strict compliance with the conditions outlined in Article 31 of Regulation (EC) No 1107/2009, as well as the specifications provided on the product labeling [2]. Directive 2009/128/EC mandates that farmers receive proper training and certification on the safe and sustainable use of pesticides while adhering to Integrated Pest Management (IPM) principles, prioritizing non-chemical alternatives. It also requires regular inspection of pesticide application equipment, record-keeping of pesticide use, and the implementation of measures to protect water bodies, human health, and sensitive areas from contamination [3]. At the international level, organizations such as the FAO and WHO regulate pesticide use by providing guidelines on responsible application, trade, and disposal, while promoting risk reduction and sustainable alternatives.

For the management and use of plant protection products (PPPs), Albania has established a comprehensive regulatory framework aligning its legislation with European Union standards, including the “Action Plan for the Sustainable Use of Plant Protection Products, 2022–2032” aiming to enhance agricultural productivity while safeguarding human health and the environment. This plan has been drafted in accordance with the European Union Directives on the sustainable use of pesticides [4]. Another aspect of Albanian legislation is the registration of plant protection products (PPP), which permits the registration of only those products authorized in one of the European Union member states. Studies have shown that pesticides are widespread across Albania, affecting surface waters, soil, sediments, fruits and vegetables [5-7]. According to FAOSTAT data, pesticide use in Albania for the period 1990-2022 has increased from 121 t to 295.61 tons per year (FAOSTAT 2025).

Enhancing food safety, increasing the value of primary products, and improving coordination throughout the food value chain are essential steps to boost efficiency. Addressing these gaps will help meet consumer expectations for safe, nutritious, and sustainable food while ensuring alignment with EU food safety standards.

This study aims to collect and analyse data on pesticide use among Albanian farmers across various regions, offering insights into their knowledge of proper application techniques and their awareness of the potential risks associated with pesticide use.

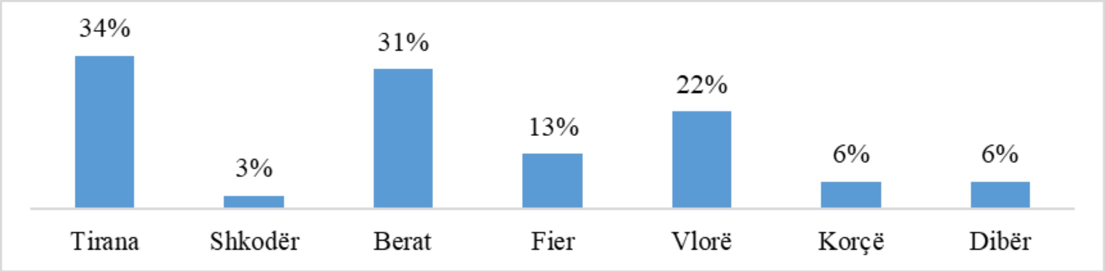
2. Methodology

A structured survey was carried out among vegetable farmers across different regions of Albania. The survey explored various aspects, including pesticide knowledge, selection, application practices, compliance with safety measures, awareness of health risks, and access to training. Only farmers engaged in commercial vegetable production were included, excluding those growing crops for personal use.

This study focused on cultivation practices for specific vegetable crops, namely tomatoes, cucumbers, onions, potatoes, lettuce, spinach, arugula, parsley, and dill. The surveyed farms encompassed both open-field cultivation and greenhouse production. Farmers from various age groups were selected to analyze differences in approaches between younger and more experienced farmers.

A total of 37 farmers participated in face-to-face interviews. The number of surveyed farms in each region was determined based on INSTAT data, prioritizing areas with the highest vegetable production. Consequently, the regions with the largest number of interviewed farmers also corresponded to those with the highest vegetable output. While Albania consists of 12 counties, an effort was made to include farms from as many of them as possible to gain a broader understanding of pesticide application practices across diverse environmental and cultural conditions. The table below presents the distribution of interviewed farmers by county.

Figure 1. Geographical distribution of surveyed farmers across Albanian counties

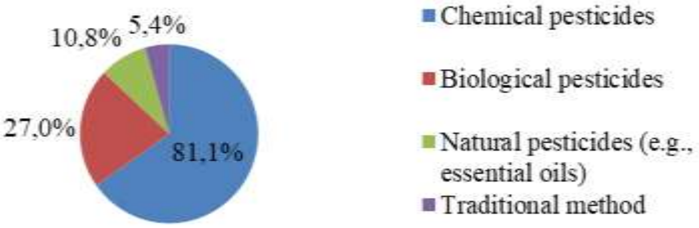


Data were collected through structured interviews and field observations involving farmers. To ensure consistency and reliability, a structured questionnaire was developed and used as a guide during the interviews. The collected responses were analyzed using Microsoft Excel, applying graphical methods for data visualization. Basic statistical analyses were conducted to identify trends and correlations between farmers’ knowledge, their perception of risks, and their behavior in pesticide use.

3. Results and discussion

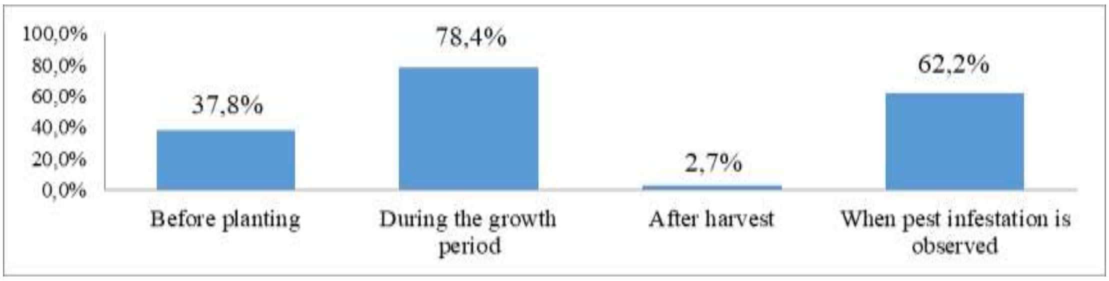
The survey results provided valuable insights into key challenges related to pesticide use, including product selection, application practices, compliance with safety measures, awareness of health risks, and access to training. All respondents reported being familiar with pesticides and confirmed their regular use for crop protection in vegetable farming. The types of pesticides most commonly used by farmers are illustrated in the Figure 2. As shown, 81.1% of farmers use chemical pesticides, and 67% of them do not follow a standardized treatment protocol. Instead, their pesticide selection is primarily based on recommendations from agronomists.

Figure 2. The distribution of pesticide application for the treatment of vegetables in Albania



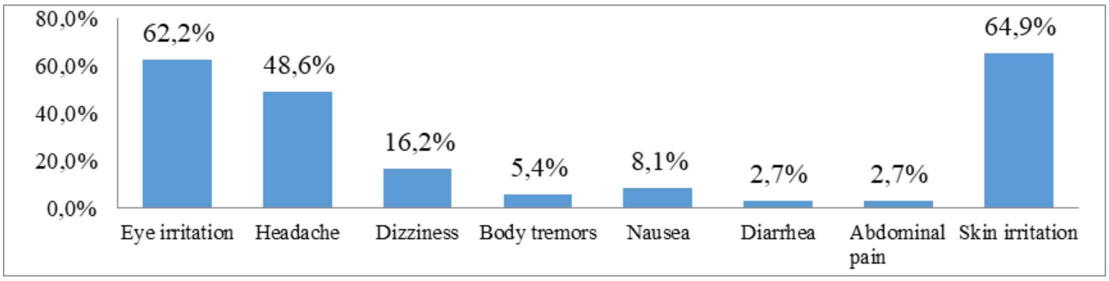
Among the respondents, only 32 % of farmers reported using complementary methods alongside pesticides, such as essential oils, adhesive traps for insects, tobacco, and poultry waste to enrich the soil. When it comes to dosage, 45.9 % of farmers rely on the product label, 56.8% follow the recommendations of agricultural pharmacists, and 40.5% base their practices on the advice of agronomists. This is although only half of the farmers claim to be familiar with official standards and regulations regarding pesticide use. Moreover, 90% of the interviewed farmers stated that they follow the label instructions concerning dosage, application timing, and the use of personal protective equipment.

Figure 3. Plant Protection Products application timing



For pest monitoring, 94.59 % of the farmers reported relying on visual observation, while only two stated that they perform analyses or use traps. Although most respondents are aware of the potential impact of pesticides on food quality and safety, they admitted that they do not carry out product analyses before placing their produce on the market. The majority of farmers acknowledged being at risk of adverse health effects due to pesticide exposure. Their awareness stems from information found on product labels, media sources, personal experience, and advice from specialists at the Directorate of Agriculture. Most of the farmers reported having experienced symptoms of acute pesticide poisoning, as illustrated in Figure 4. Additionally, only two farmers mentioned suffering from long-term health issues, particularly related to liver function.

Figure 4 Symptoms experienced when applying pesticide



76 % of the interviewed farmers reported receiving a visit from a representative of the government's agricultural service experts for training on the proper use of pesticides. Despite these visits, the data collected from this survey indicate a low-risk perception regarding health risks and limited knowledge about pesticide use. This limited outreach indicates that current training efforts are insufficient to adequately cover the broader agricultural community’s needs and ensure widespread knowledge on the safe use of pesticides.

4. Conclusion

Ensuring the sustainable management of pesticide use risks in Albania requires an integrated approach that includes regulatory enforcement, resilient crops, greenhouse improvements, safer farming methods, efficient irrigation policies, and farmer training. Stricter regulatory enforcement is essential, as farmers feel insecure about the quality of pesticides sold at sales points. Around 70% of the respondents reported encountering pesticide inefficacy in disease control, prompting them to repeat spraying with different products.

The use of disease-resistant planting materials can help reduce the reliance on pesticides. Healthier seeds and seedlings contribute to more sustainable farming practices. Encouraging the adoption of safer agricultural practices is crucial. Only 27% of farmers currently practice Integrated Pest Management (IPM), despite acknowledging its benefits. However, the high cost of these alternatives remains a barrier. Farmers should be guided to implement approved standards such as Good Agricultural Practices (GAP).

Improving greenhouse infrastructure can help control insect intrusion and contribute to a reduction in pesticide use. The farmers emphasize the need for governmental subsidies or financial aid for greenhouse investments, as the costs are high and unaffordable for many.

Irrigation water management is another critical area. All interviewed farmers in Lushnja, one of Albania’s largest vegetable-producing regions, reported that insufficient irrigation water negatively affects crop health and yield.

Training for farmers is essential, as the findings reveal a lack of adequate understanding regarding pesticide use. This is a key issue in preventing unsafe pesticide practices in the future. The over-reliance on pesticides, coupled with a lack of knowledge about proper handling and limited access to pesticide training, presents a significant risk for farmers and consumers, particularly in terms of pesticide exposure and residues on crops. Enhancing farmers' knowledge and providing adequate resources are vital steps toward ensuring food safety, protecting public health, and promoting sustainable agricultural practices in Albania.

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References

[1] Carrasco Cabrera. L, Di Piazza. G, Dujardin. B, Marchese. E, & Medina Pastor. P. (2024). The 2022 European Union report on pesticide residues in food. EFSA Journal,22(4), e8753. https://doi.org/10.2903/j.efsa.2024.8753

[2] Regulation (EC) No 1107/2009 of the European Parliament and of the Council of 21 October 2009 concerning the placing of plant protection products on the market.

[3] Directive 2009/128/EC of the European Parliament and of the Council of 21 October 2009 establishing a framework for Community action to achieve the sustainable use of pesticides.

[4] FAO 2022, Action plan for the sustainable use of plant protection products 2022-2032. https://qbz.gov.al

[5] Nuro.A, Marku.E, Myrtaj.B. 2018 “Organochlorine pesticides and their residues in surface waters of Albania”. DOI: 10.5937/zasmat1804475N

[6] Mai. S, Ninga.E, Mukaj.M, Cara.M. 2019 “The Presence of Pesticide Residues in Select Commodities in Albanian Market”.

[7] Likaj. M, Marku.E. 2024 “Determination of some pesticide residues in vegetables in Albanian market”

EVALUATION of DIFFERENT FILLER MATERIALS on GLASS FIBER REINFORCED POLYMER COMPOSITES for the AUTOMOTIVE INDUSTRY

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Abstract

Composite materials were developed through intensive research in materials science as a result of the failure of pure materials to meet the performances demanded in sectors such as automotive, aerospace and defense over the years. In line with the developments in the composite industry, studies are implemented to improve performance with fillers and reinforcements. Considering both cost and accessibility, research on the reuse of waste materials is rapidly increasing and producing sustainable solutions. [1,2] Disposal of organic waste produces greenhouse gases that will affect climate conditions. Instead of a disposal process, it is possible to convert it into a carbon-based product called Biocarbon which can be used as a filler in polymer composites. [3]

In this study, the effect of conventional and bio-based flame-retardant fillers on glass fiber reinforced polyester composites was investigated. Biocarbon was used as a bio-based and environmentally friendly additive, which can replace with aluminum trihydrate that is one of the conventionally preferred flame-retardant chemicals. Composite production in various compositions was carried out open mold lay-up method. Apart from the control sample, 2 different compositions were determined and glass fiber reinforced composite was produced. Synergistic effects were observed when conventional and bio-based additive materials were used together in the structure. Glass fiber discontinuous mat and plain-woven glass fiber were used as reinforcement materials. At the end of production, 3 composite plates were obtained. Tensile, three-point bending and impact tests were performed to investigate the mechanical properties of the produced samples. The flame retardant characteristics were evaluated by horizontal burning test (UL 94 HB) to compare the effects of filler materials. The results of the tests applied to composites containing aluminum trihydrate, biocarbon and hybrid structures were compared. Results showed that the biocarbon-added samples showed higher tensile, bending, impact resistance and flame-retardant properties than the unfilled composite. As a result, it was observed that biocarbon-added composites can compete with the functionality provided by conventional additive materials and show sufficient performance to be used in the automotive industry.

Key Words: *Aluminum Trihydrate, Biocarbon, Filler, Glass fiber, Polymer composites*

References

[1] İlhan, R., & Feyzullahoğlu, E. (2019). Cam elyaf takviyeli polyester (ctp) kompozit malzemelerde kullanılan doğal elyaflar ve dolgu maddeleri. *El-Cezeri*, *6*(2), 355-381.
[2] Rajendran, S., Palani, G., Arunprasath, K., Shanmugam, V., Marimuthu, U., & Veerasimman, A. (2024). Eco-friendly sugarcane biochar filler for enhanced mechanical

properties in S-glass/polyester hybrid composites. *Cleaner Engineering and Technology*, *20*, 100759.
[3] Sundarakannan, R., Arumugaprabu, V., Manikandan, V., & Vigneshwaran, S. (2020). Mechanical property analysis of biochar derived from cashew nut shell waste reinforced polymer matrix. *Materials Research Express*, *6*(12), 125349.

Utilization of Inductively Coupled Plasma (ICP) in Monitoring and Analysis of Some Heavy Metals in Jordanian Dams.

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Abstract

This study involved the collection of water and sediment samples from four Jordanian dams, which are: Kafrain, King Talal, Shuaib, and Mujib dams during both summer and winter seasons. The concentration levels of Cd, Co, Hg, Fe, Ni, Zn, and Pb ions were measured using ICP-OES. During the summer, Kafrain Dam exhibited the highest concentrations of metal ions in sediment samples, with levels exceeding WHO limits for all metal ions except Zn. Conversely, Mujib Dam showed the highest concentrations of metal ions in water samples, particularly Fe, Co, and Cd, surpassing WHO and JSMO permissible limits. In winter, no metal ions were detected in water samples across all dams, while Mujib Dam had the highest metal ion concentrations in sediment samples, with Pb, Co, Cd, Ni, and Fe exceeding WHO and JSMO guidelines. The study suggests using nanotechnology-treated filters to mitigate heavy metal contamination and recommends future research on alternative sewage plant locations and binding compounds to prevent metal accumulation.

Key Words: *Jordan; Water; ICP; Sediment; Dams*

1. Introduction

Jordan, located in the Middle East and bordered by Saudi Arabia, Iraq, Syria, and the Palestinian Territories, faces significant water scarcity. This issue, intensified by climate change, population growth, and the overuse of water resources, has prompted the country to invest in dam construction. These dams now play a crucial role in supplying water and energy to meet the needs of Jordan's population [3].

The significance of dams in Jordan is paramount due to the country's extreme water scarcity. Jordan, one of the most water-scarce nations globally, experiences less than 200 mm of annual rainfall over 91% of its land area. With a semi-arid Mediterranean climate, characterized by low precipitation and high evaporation (approximately 92.2% of annual precipitation), water is a critical and limited resource. The construction of dams has been essential in ensuring a reliable water supply for Jordan's population. Additionally, the increasing frequency of droughts and climate-related disasters underscores the importance of these dams in mitigating the impacts of climate change [3].

Dams are substantial structures designed to regulate river flows and store water for future use. In Jordan, key dams such as King Talal, Kafrain, Mujib, and Shuaib are vital for providing water for domestic, agricultural, and industrial purposes, as well as generating hydroelectric power. These dams support agriculture by enabling crop irrigation and increasing yields, and they contribute to industrial growth by supplying a consistent source of energy through hydroelectricity. Jordan is home to several major dams that play a crucial role in water storage and management. Among them, King Talal Dam stands as one of the largest dams in Jordan, with a capacity of 85 million cubic meters (MCM), and was completed in 1977 [1]. Kafrain Dam, constructed between 1986 and 1997, has a storage capacity of 8.5 MCM [12], while Shuaib Dam, built in 1969, holds up to 1.4 MCM of water [12]. Mujib Dam, the most recent addition, was completed in 2003 and has a total storage capacity of 29.8 MCM [12]. Fig. 1 shows the distribution of the studied dams in Jordan. These dams contribute significantly to Jordan's water resources, supporting agriculture, drinking water supply, and overall water security.

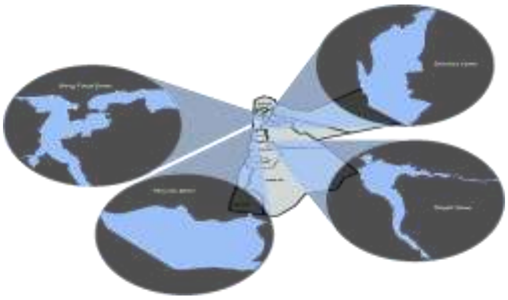

Fig. 1. Map showing the distribution of the studied dams in Jordan.

Heavy metals naturally occur in the Earth's crust, but their danger stems from their tendency to accumulate in living tissues, disrupting essential biological functions. Their presence can impair organ function, interfere with cellular processes, and ultimately contribute to severe health complications [12]. This research included the study of a group of heavy elements such as: (Cd, Pb, Fe, Hg, Ni, Co and Zn). The allowed percentages of some heavy metal ions, according to the World Health Organization (WHO) and the Jordanian Standards and Metrology Organization (JSMO), are shown below in Table 1: [1-5].

Table 1. The concentrations of some heavy metals, according to the World Health Organization and the Jordanian Standards and Metrology Organization.

Components (mg/L)	**WHO guidelines for drinking (WHO, 2011)**	**Jordanian standards for irrigation (JSMO, 2006)**	**Jordanian standards for drinking (JSMO, 2015)**
Fe	0.3	5.0	1.0
Hg	0.006	0.002	0.006
Cd	0.003	0.01	0.003
Zn	5.0	5.0	4.0
Co	0.05	-	-
Pb	0.01	-	-
Ni	0.07	-	-

In 2005, Ghrefat & Yusuf studied sediment samples from 35 different sites of Wadi Al Arab Dam using atomic absorption spectroscopy (AAS). The mean concentrations of Mn, Fe, Cu, Zn and Cd ions were (30, 11.27, 0.06, 0.34 and 0.009) respectively [9].

During the dry season (June–September 2006) and winter season (November–February 2007), Manasreh *et al.* have collected sediment samples from sixty-five sites in the Mujib Dam and analyzed for heavy elements, including Fe, Mn, Cd, Zn, Ni, and Pb. The analysis was performed using an Atomic Absorption/Flame Emission Spectrophotometer (AAS/FES). The results indicated that Fe had the highest concentration, while Cd had the lowest [11].

During the summer of 2009, Fandi *et al.* have studied eleven heavy metals such as (Cu, Zn, Cr, Mn, Fe, Co, Mo, Pb, Ni, Hg and Sn) in five water samples were collected from three different locations inside and the outlet of the King Talal Dam by Inductively Coupled Plasma / Mass Spectrometry (ICP-MS). The results indicated that Sn has the highest level of concentrations while Cu has the lowest [7].

Our goal is to measure the total levels of inorganic pollutants such as (Zn, Pb, Co, Cd, Ni, Hg, and Fe) in sediment and water samples of some Jordanian dams during winter and summer seasons.

2. Experimental

Ten sediment samples and ten water samples total of (20 samples) were collected from reachable places from each of the studied four Jordanian dams: Al-Kafrain Dam, King Talal Dam, Mujib Dam, and Shuaib Dam, during the summer (September 2022) and winter (March 2023) seasons (see Fig. 2). Water samples were taken from the surface reservoir from every site with 100 m separating each sample from. Sediment samples were taken from the reservoir sides at the depth (20 cm) from every site with 100 m separating each sample. The distance was measured using a distance measuring device. The collected samples were kept in 50-mL polyethylene (PE) bottles. They were well preserved and transported to the laboratory.

Fig. 2 represents the reachable locations in each dam.

2.1. *Preparation of samples.*

Sediment samples were transported to the laboratory in (PE) bottles and dried in the oven at 55°C for 24 hours. Then, the samples were manually digested; 5 grams of each dry sediment sample were digested in 25 mL of nitric acid and heated at 70°C until all the added nitric acid was evaporated. Next, 5 mL of hydrochloric acid was added and heated until all the acid was removed. Finally, 10 mL of water was added and left to sit for 30 minutes. This solution was transferred to a 25 mL volumetric flask, which was then precisely filled with water to the mark. The final extracts were filtered using 0.45-µm filters [2]. Water samples were filtered using filter papers then passed through 0.45-µm filters prior to measurement.

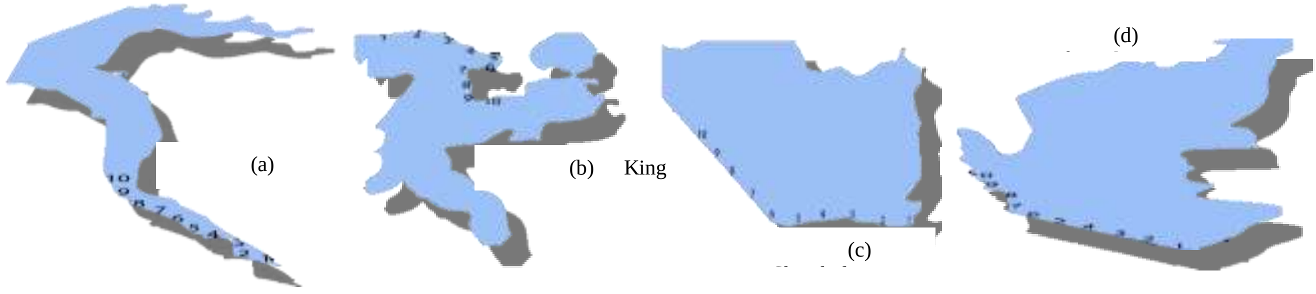

Fig. 2. Maps of the reachable locations in the studied dams.

2.2. *Chemical and instrument*

All chemical reagents that were utilized for measurements using the ICP instrument (AR grade) were bought from Sigma-Aldrich and were used in preparation of solutions. Standards (1000 ppm) of Zn, Ni, Co, Cd, Hg, and Pb from Thermo-Scientific were used for ICP measurements.

Stock solutions were prepared as the following: 10 mL solutions of 1000 ppm of each studied metal ions (Ni, Co, Cd, Zn, Pb, and Hg) were diluted to 100 ppm by 5% of nitric acid. Standard solutions were then prepared by diluting appropriate volumes of stock solution using nitric acid as a solvent to get 0.05, 0.1, 0.3, 0.5, and 0.7 ppm concentrations. Fe ions were prepared by dissolving 0.290 g of iron chloride ($FeCl_3$) in nitric acid.

Inductively Coupled Plasma Optical Emission Spectroscopy (*ICP-OES*) model iCAP 7000 SERIES (Germany) was used.

3. Results and discussion

Calibration curves were constructed using the prepared calibration solutions. Standard addition method was employed to remove matrix effects. Least-square equations and the correlation coefficients (R^2) were estimated by linear regression of the calibration data using a spreadsheet (Excel®) software. The calibration data ($R^2 > 0.997$) were obtained and employed to calculate the concentrations of the studied metal ions.

3.1. Summer season

Table 2 shows the average concentrations in (ppm) of each metal ion in all dams in the summer season for water and sediment samples. The samples were diluted by a suitable dilution factor for each metal ion. Each result is based on the average of triplicate measurements after multiplying by the metal's dilution factor, for the 10 samples from 10 collection points for each of the water and sediment samples.

3.2. Winter season

The average concentrations of each metal ion in the water and sediment samples from all dams in the winter season are shown in Table 3 in (ppm). For each metal ion, the appropriate dilution factor was used to dilute the samples. Each result is based on the average of triplicate measurements for the 10 samples from 10 collection points for the water and sediment samples, multiplied by the metal's dilution factor. The concentrations of Hg ions were not presented in Table 3 as they were not detected. All studied metal ions were not detected in water samples.

Table 2. The average concentrations in (ppm) (as conc ± std. dev) of metal ions in water and sediment samples in all dams during the summer season**.**

DAM*	**Pb** (±0.0586)	**Cd** (±0.0849)	**Co** (±0.0992)	**Zn** (±0.1065)	**Ni** (±0.0542)	**Hg** (±0.0645)	**Fe** (±2.4736) **
AVG-Kf-S	0.1378	0.1443	0.1210	0.1983	0.1477	0.1177	0.4187
AVG-Kf-W	-	0.0807	-	0.0513	-	-	2.2164
AVG-Muj-S	0.0570	-	-	0.2918	0.0592	-	0.3370
AVG-Muj-W	-	0.0630	0.1958`	0.1573	0.0546	-	1.4163
AVG-Sh-S	0.0939	0.2416	0.2399	0.0847	0.0624	0.1317	6.6473
AVG-Sh-W	-	0.0508	0.0828	-	-	-	0.1409

*Abbreviations: AVG: Average; S: sediment; W: water; Kf: Kafrain; Muj: Mujib; Sh: Shuaib.

** Concentrations of Fe ions were studied alone due to their high concentrations compared to other concentrations.

Table 3. The average concentrations in (ppm) (as conc ± std. dev) of metal ions in sediment samples in all dams during the winter season**.**

DAM*	**Pb** (±0.0598)	**Cd** (±0.1751)	**Co** (±0.0966)	**Zn** (±0.7541)	**Ni** (±0.3470)	**Fe** (±14.9773) **
AVG-Kf-S	0.1403	0.3722	0.2620	0.6311	0.6900	32.9553
AVG-Sh-S	0.0757	0.2180	0.0841	1.1028	0.4966	19.9495
AVG-Muj-S	0.0769	0.4060	0.1374	2.1095	0.8398	32.7787
AVG-Kt-S	0.1243	0.0621	0.1376	0.3735	0.3327	20.5607

*Abbreviations: AVG: Average; S: sediment; Kf: Kafrain; Muj: Mujib; Sh: Shuaib; Kt: King Talal.

** Concentrations of Fe ions were studied alone due to their high concentrations compared to other concentrations.

4.3. a. Kafrain dam

During summer, the concentrations of ions in sediment sample were higher than those in water samples as shown in Fig. 3 and 4. This increase was attributed to the large volume of water, which led to a dilution effect, reducing ion concentrations in water. Table 1, which presents the permissible concentrations set by the WHO & JSMO, indicates that the levels of Pb, Cd, and Co ions exceeded the allowable limits in sediment samples. However, in water samples, only Cd surpassed the permissible threshold. In winter, the concentrations of metal ions in sediment samples remained higher than those in the water samples due to the abundance of water and the increased deposition of metal ions. Among the analyzed metals, only Zn remained within the permissible limits established by the WHO & JSMO, while other metal ions were not detected in the water samples.

4.3.b. Shuaib Dam

As shown in Fig. 5 during the summer season, the concentrations of heavy metal ions were higher in sediment samples than in water samples. This was primarily due to the lower water volume, which caused the sediments to become stagnant, allowing metal ions to accumulate. According to Table 1, the levels of Pb, Co, Cd, and Hg ions in sediment samples exceeded the permissible limits, while in water samples, only Co and Cd surpassed the allowable threshold. In winter, as shown in Fig. 6, the increased water level enhanced the sedimentation process, leading to a higher accumulation of metal ions in sediments while diluting their concentrations in water samples (dilution effect). As indicated in Table 1, Pb, Co, Cd, and Ni ions concentrations exceeded the permissible limits in sediment samples, whereas only Zn ion remained within the acceptable range.

4.3.c. Mujib Dam

During the summer: Mujib dam differs from the rest of the studied dams, because some water samples contain the highest concentration of heavy metal ions, and this can be attributed to the scarcity of water and the proximity of water to the sediments (due to the shallow depth). According to Table 1, it was found that the concentrations of Pb and Ni ions in sediment samples were higher than the permissible limit, and the concentrations of Cd, Ni and Pb ions were higher than the permissible limit in the water samples as depicted from Fig. 7. As shown in Fig. 8: In contrast to the metal ions in the water samples, the metal ions in sediment samples were present in higher concentrations. These results may be attributed to the location of the dam in Wadi-Mujib, which is surrounded by mountains that allow water to flow through it, as the flowing water carries pollutants and silt during this process. It was noted that the concentration of Zn ions is within the permissible level. However, the concentrations of Pb, Co and Cd ions are higher than the permissible limit, as shown in Table 1.

4.3.d. King Talal Dam

As for King Talal Dam, the concentrations of metal ions in the studied sediment samples were higher than the concentrations of metal ions in water samples as shown in Fig 9. These results may be explained by: 1- Rain has accelerated the sedimentation process. 2- Water sources inside the dam, such as the Zarqa River, where industrial wastewater emptied into it constitutes a source of pollution. It was found that the concentrations of Pb, Cd, Co, and Ni ions were greater than the permissible limit according to Table 1. Zn ions were within the permissible limit.

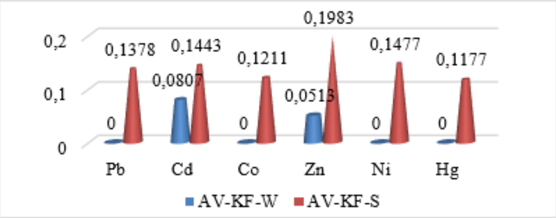

Fig 3. The average concentrations of some heavy metal ions in sediments and water samples in Kafrain Dam in the summer season.

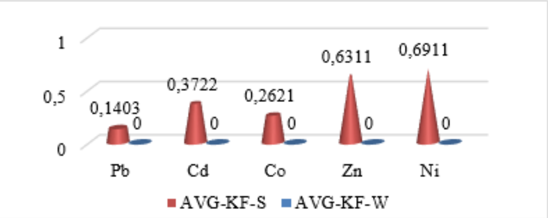

Fig 4. The average concentrations of some heavy metal ions in sediments and water samples in Kafrain Dam in the winter season.

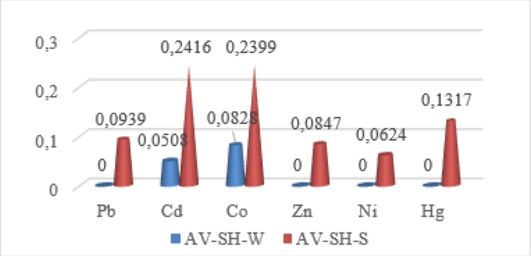

Fig5. The average concentrations of some heavy metal ions in sediments and water samples in Shuaib Dam in the summer season.

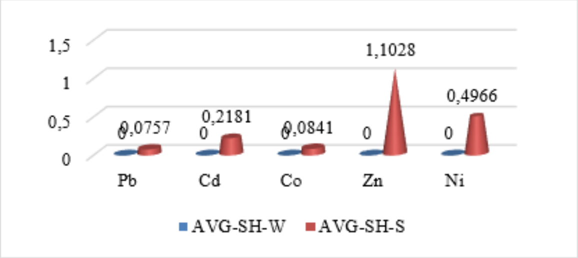

Fig. 6. The average concentrations of some heavy metal ions in sediments and water samples in Shuaib Dam in the winter season.

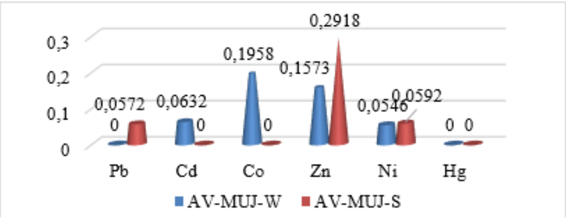

Fig. 7. The average concentrations of some heavy metal ions in sediments and water samples in Mujib Dam in the summer season.

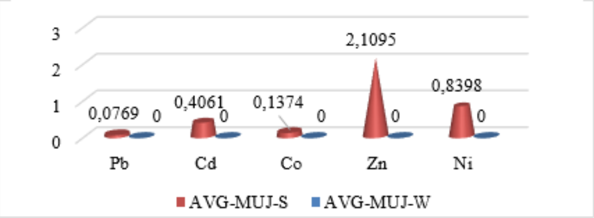

Fig. 8. The average concentrations of some heavy metal ions in sediments and water samples in Mujib Dam in the winter season.

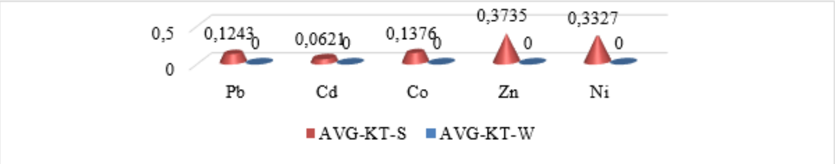

Fig. 9. The average concentration of some heavy metal ions in sediments and water samples in King Talal Dam in winter season.

4.4. Discussion

The high concentrations of the studied metal ions may be explained by:

- Cd: 1- Some human factors such as the use of pesticides, and 2- Burning fossil fuels, and 3- Sewage [4-6].
- Co: The decomposition of cyanobacteria and humic materials [6].

- Pb: 1- Combustion of fossil fuels, and 2- Deposition of organic sediments [6].
- Fe: 1- The effluent of the Salt and Fuhays treatment plant; 2- The weathering of ferruginous sandstone naturally, especially the Kurnub Sandstone group that includes the research area's site; and 3- Human sources, like pesticides and fertilizers used in agriculture [4].
- Zn: 1- Human factors, including: *a*- pesticides used in farming, *b*- fertilizers, and *c*- sewage which is most likely from the Al-Lajoun (WWTP) [9-11].
- Ni: 1- The limestone rocks present in the area, 2- Liquid waste, and 3- Pesticides used in agricultural activities [11].
- Hg: 1- High quantity of Hg is linked to sandy rocks, and 2- The levels of Hg in sediments varies depending on the type of rock and soil that makes up the dam [8].

5. Conclusion

This study offers information to assess the concentrations of certain metal ions found in water and sediment samples taken in summer and winter seasons from some Jordanian dams. The concentrations were dispersed as follows among the dams: In summer sediment samples, the highest levels of Pb and Ni ions were found in Kafrain Dam as (Ni>Pb), the highest concentrations of Zn ions were found in Mujib Dam, and the highest concentrations of Fe, Cd, Co, and Hg ions were found in Shuaib Dam as (Fe>Cd>Co>Hg). Regarding the water samples taken during the same season, the Kafrain Dam had the highest levels of Fe and Cd ions as (Fe>Cd), while the Mujib Dam had the highest levels of Co, Zn, and Ni ions as (Co>Zn>Ni), and finally Pb and Hg ions were not detected. The levels of metal ions, however, were not detected in water samples during the winter season. Regarding ion concentrations in the sediment samples, they were distributed as follows: the highest levels of Pb, Co, and Fe ions were found in the Kafrain Dam as (Fe>Co>Pb), whereas the highest levels of Cd, Zn, and Ni ions were found in the Mujib Dam as (Zn>Ni>Cd). In contrast, during the winter, Hg ions were not detected in any sample. According to the WHO and JSMO shown in Table 1, metal ions with a percentage higher than the allowable limit for drinking and agricultural were found in sediment samples taken in the summer. These ions include those found in Kafrain and Shuaib dams, such as Hg and Cd ions. Cd was the only ion found in summertime water samples from Kafrain Dam, Mujib Dam, and Shuaib Dam that had a concentration greater than allowed limit. On the other hand, during the winter, all sediment samples contained only Cd and Fe ions in concentrations higher than the permissible level for drinking and agriculture according to WHO and JSMO shown in Table 1.

In light of the study's findings, which revealed high concentrations of some metal ions in various samples obtained from some of the studied Jordanian dams, some solutions have been proposed to avoid such findings. They may include:

- Finding better locations for sewage plants away from dams.
- Discovering some harmless compounds that bind to heavy metals.
- Treating filters with nanotechnology that can remove heavy metals from water.

6. References

[1] Abualhaija, M. M., & Mohammad, A.-H. (2021). Assessing Water Quality of Kufranja Dam (Jordan) for Drinking and Irrigation: Application of the Water Quality Index. Journal of Ecological Engineering, 22(9).

[2] Alnawaiseh, A., Arar, S., Kailani, M., & Alawi, Dr. M. (2020). MONITORING OF HEAVY METAL POLLUTION IN SEDIMENT SAMPLES FROM SELECTED JORDANIAN DAMS. Fresenius Environmental Bulletin, 29, 11753–11760.

[3] Ammary, B. Y. (2007). Wastewater reuse in Jordan: Present status and future plans. Desalination, 211(1-3), 164-176.

[4] A. B., & Al-Hawari, Z. (2015). Assessment of heavy metal pollution of surface sediments in Wadi Shu'ayb, Jordan. Jordan journal of civil engineering, 9(3).Edition, F. (2011). Guidelines for drinking-water quality. WHO Chronicle, 38(4), 104–108.

[5]Edition, F. (2011). Guidelines for drinking-water quality. WHO chronicle, 38(4), 104-8.

[6]El-Radaideh, N., Al-Taani, A. A., & Al Khateeb, W. M. (2017). Status of sedimentation in King Talal Dam, case study from Jordan. Environmental Earth Sciences, 76, 1-17.

[7]Fandi, K., Qudsieh, I., Muyibi, S., & Massadeh, M. (2009). Water pollution status assessment of King Talal Dam, Jordan. Advances in Environmental Biology, 3, 92–100.

[8]García-Ordiales, E., Esbrí, J. M., Covelli, S., López-Berdonces, M. A., Higueras, P. L., & Loredo, J. (2016). Heavy metal contamination in sediments of an artificial reservoir impacted by long-term mining activity in the Almadén mercury district (Spain). Environmental Science and Pollution Research, 23, 6024-6038.

[9]Ghrefat, H., & Yusuf, N. (2006). Assessing Mn, Fe, Cu, Zn, and Cd pollution in bottom sediments of Wadi Al-Arab Dam, Jordan. Chemosphere, 65(11), 2114-2121.

[10]Hilal, A. H. A., & Alhaija, M. M. A. (2010). Nutrients in Water and Sediments of King Talal Dam-Jordan. 3(3).

[11]Manasreh, W., Hialat, I., & El-Hasan, T. (2009). Heavy metal and anionic contamination in the water and sediments in Al-Mujib reservoir, central Jordan. Environmental Earth Sciences, 60, 613–621. https://doi.org/10.1007/s12665-009-0201-7.

[12]Ministry of Water and Irrigation (MWI). (2023, August). Retrieved from http://mwi.gov.jo.

[13]Singh, R., Gautam, N., Mishra, A., & Gupta, R. (2011). Heavy metals and living systems: An overview. Indian journal of pharmacology, 43(3), 246-253.

INVESTIGATING THE PROPERTIES OF RECYCLED AND VIRGIN POLY (ETHYLENE TEREPHTHALATE) TEXTURED YARNS: EFFECT OF DIFFERENT BLENDING RATIOS

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Abstract

Nowadays, there is an increasing interest on the evaluation of recycled polyester in textile industry due to European Green Deal sustainable goals. The recycling and reuse of polyester from wastes is very desirable however it is still challenging. In this study, it is aimed to investigate the blending of recycled and virgin poly (ethylene terephthalate) to obtain textured yarns. The effect of recycled and virgin poly (ethylene terephthalate) yarns blending ratio (100:0, 0:100, 25:75, 50:50, 75:25) (% w/w) on the mechanical, thermal and color properties of textured yarns were investigated. The results showed that blending ratio affect the thermal and mechanical properties of the yarns while have no significant effect on dyeability characteristics.

Key Words: *Recycled polyester, Recycling, Sustainable textiles, Textile waste, Mechanical and thermal properties*

1. Introduction

Nowadays, plastics are a complementary part of our modern life that has been wide range of applications including packaging, construction, agriculture, and households [1]. They have some specific advantages including the low production costs, low density, corrosion resistance, potential in molding in different shapes and sizes [1-5]. Global plastic production has a 3–4% annual growth which raised from 322 Mt in 2015 to 367 Mt in 2020 [6,7]. Additionally, it is estimated that the plastic production will be tripartite in the next 30 years [8,9]. However, the rapid growth of the plastic production leads to the resource depletion and ecological destruction problems because most of the plastics generally discarded in landfills or incinerated [10,11]. Therefore, there is an urgent necessity to recycle and reclaim the plastics [12,13].

Polyethylene terephthalate (PET) is one of the most widely used plastic especially in textile fibers and food packaging (film, bottles etc.) applications. From the view of sustainable and zero waste society, PET is considered as an alternative feedstock not a waste (Fig.1). Accordingly, PET is the most widely recycled and reused non-renewable plastic globally [14,15]. Including the reduction of the energy and related carbon footprint of textile industry which is known as one of the world's biggest polluters [17]. Recently, the raise in both the number of studies on rPET fibers and the quality of the rPET fiber-based textile products also supports the significance of recycling applications and the evaluation of rPET fibers in the textile sector [19].

In this study, yarns were obtained by texturing different ratios (100:0, 0:100, 25:75, 50:50, 75:25) (% w/w) of rPET and virgin PET partially oriented yarns. The properties of final samples were investigated by using color, mechanical, thermal and structural tests.

Fig. 1. The cycle of used plastic bottles to become rPET yarn

2. Material And Method

2.1. Material

Semi-dull polyester and semi-processed semi-finished polyester chips obtained from INDORAMA Ventures PCL (Thailand) company were used as raw materials. The viscosity of the chips (internal viscosity) (IV) is 0,650 dL/g, and the amount of chips per gram is 35 chips/g.

2.2. Method

2.2.1 Production of Virgin and Recycled Polyester partially oriented yarn

Polyteks Textile Industry Research and Education Co. produced POY and rPET POY yarns with identical dtex/filament (78/72) using semi-dull polyester and rPET chips via melt spinning. Yarns were textured by friction texturing for sock knitting. Blends of rPET and virgin POY in 100:0, 0:100, 25:75, 50:50, 75:25 (% w/w) ratios were processed, resulting in five 1 kg, 300 denier bobbins labeled P1 to P5. The production speeds were 2900 m/min for virgin POY and 3000 m/min for rPET POY. These POY yarns were later textured using the company’s friction texturing machine, resulting in 334/288 (dtex/filament) textured yarns.

2.2.2 Bobbin Dyeing Process

The produced 334/288 (dtex/filament) textured yarns were sent from Taşdelen Group to Sintaş for bobbin dyeing using 1.0 g of B.TFBL dyestuff. The yarns were washed at 80°C for 20 minutes, then rinsed at 50°C for 20 minutes. Dyed bobbins were split into five equal parts at Polyteks by a transfer machine (500 m/min), winding 200 g of yarn per bobbin. Each coil was tested for strength with a STATIMAT device, and two color charts were knitted and analyzed using a spectrophotometer at Polyteks.

2.3. Characterization

2.3.1 Characterization of filament yarns

According to the TS 244 ISO 2060 standard, the weight (yarn number) of the determined length of the yarn was calculated. This process was repeated 5 times for each of the 5 yarn samples. Yarn strength tests were carried out in the STATIMAT brand test device according to the DIN EN ISO 2062 standard. [20,21]

2.3.2 Characterization of textured yarns

2.3.2.1 Differential scanning calorimetry (DSC):

Thermal analyzes of the yarns were carried out in the HITACHI 7020 DSC device according to the ISO 11357-7 standard. Analysis parameters were studied between 30-300°C temperatures with 10°C/min temperature increase. The weights of the samples vary between 6-10 mg. [22]

2.3.2.2 Characterization of dyed filament yarns:

According to the TS 244 ISO 2060 standard, yarn number of the determined length of the dyed yarn was calculated. This process was repeated 3 times for each of the 5 yarn samples. Measurements were made on the color charts in CIELAB color space with a hand-held spectrophotometer device in the laboratory unit of Polyteks. The color chart number 5 was taken as a reference (100% PET). [20,21,23] Additionally the whiteness indexes were calculated by according to the following equations as described as below Equation (1) [24,25].

$$\text{Whiteness index} = 100 - \sqrt{}(100-L^*)^2 + a^2+b^2 \qquad (1)$$

3. Results and Discussion

3.1. Properties of filament yarns

Table 1 shows the mechanical properties of POY yarns. The evenness, dtex number and tenacity of the yarns were similar. The elongation at break of rPET yarn was found to be slightly lower than the virgin PET yarns.

Table 1. Mechanical properties of POY yarn

Sample	Dtex/F[a]	Dtex	Elongation at break (%)	Tenacity (cN/dtex)	Eveness	Shrinkage
1:3	78/7	138.7	121.6	2.5	0.82	52.7
2:2	78/7	138.8	124	2.4	0.83	63.4

3.2. Thermal Properties

The thermal properties of the yarns were determined using DSC. In DSC curves (Fig.2), the endothermic peaks of fibers showed similar shapes. The melting temperature of the blended yarns were closed to virgin PET. In addition, the melting temperature of the blended yarns were decreased with the increased rPET ratio.

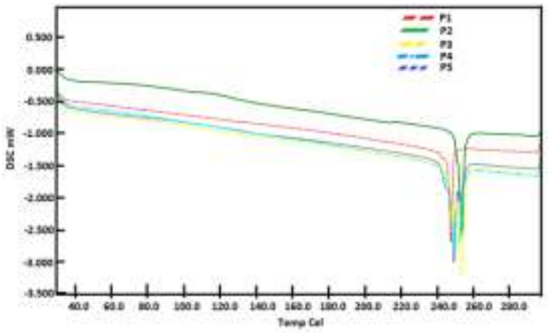

Fig.2. DSC curves with overlapping of the yarns samples. (DSC thermograms)

Table 2. Thermal properties of yarns from DSC

Sample Code	Blending Ratio (rPET: PET) % w/w	Heat storage capacity (Cp) (mJ/mg)	Tm (°C)
P1	100:0	5.33	247.9°C
P2	0:100	3.55	253.9°C
P3	25:75	6.58	253.6°C
P4	50:50	7.42	249.1°C
P5	75:25	7.95	249.6°C

3.3. Mechanical Properties of Textured Undyed and Dyed Yarns

Table 3. Comparison of mechanical testing of textured undyed and dyed yarns

Sample Code	Blending Ratio (rPET: PET) % w/w	Dtex/F	Dtex	Elongation at break (%)	Maximum force (cN)	Tenacity (cN/dtex)
P1(undyed)	100:0	334/288	360.5	17.51	1285.43	3.57
P1 (dyed)			380.4	24.35	1302.96	3.43
P2 (undyed)	0:100	334/288	360.7	23.18	1360.51	3.78
P2 (dyed)			398.3	25.34	1314.81	3.30
P3 (undyed)	25:75	334/288	360.8	23.21	1394.92	3.87
P3 (dyed)			380.6	29.15	1376.95	3.62
P4 (undyed)	50:50	334/288	360.4	18.92	1223.37	3.40
P4 (dyed)			380.5	27.96	1337.99	3.52
P5 (undyed)	75:25	334/288	360.5	17.13	1269.19	3.53
P5 (dyed)			380.3	25.90	1278.23	3.36

The dyed yarns have higher dtex value than the undyed ones. The elongation at break of both undyed and dyed yarns were reduced with the increased rPET ratio as expected. The undyed blended sample that has higher virgin PET ratio (P3) showed the highest tenacity (3.87 cN/tex). This increase was supposed due to the higher tenacity of PET fibers than rPET fibers. This increase was supposed due to the higher tenacity of PET fibers than rPET fibers [26].

3.4. Color of Dyed Textured Yarns

The effect of blending ratio on the color characteristics of the yarns is shown in Table 4. Also, the visual appearances of the yarns are shown in Fig.3. There is not any apparent variation in dyeability between different PET yarns as a function of blending ratio. The blending ratio lead to very small changes in color coordinates. When the dyeability of the yarns is compared, it is seen that the L* values are as P3>P5>P4>P1>P2, while +a* and -b* values determined to be P1>P2= P3> P5> P4 and P2=P3>P1>P4>P5, respectively.

Fig.3. Color charts of dyed yarns

Table 4. Color coordinates of blue dyed textured yarns

Sample Code	Blending Ratio (rPET: PET) % w/w	L*	a*	b*	ΔE	Whiteness Index
P1	100:0	37.65	0.98	-41.32	3.12	25.19
P2*	0:100	35.38	0.96	-41.41	0.00	23.24
P3	25:75	38.04	0.96	-41.41	3.46	25.46
P4	50:50	37.78	0.58	-40.95	2.95	25.51
P5	75:25	37.96	0.70	-40.86	2.98	25.71

4. Conclusion

Recycled PET based yarns were blended with virgin yarns to achieve circular economy goals for textile applications. The main conclusions are as follows:

The blending ratio of rPET:PET showed no apparent effect on the dyeing properties of yarns.

There was a reduction on elongation at break of both undyed and dyed yarns with the increased rPET ratio.

Moreover, the melting temperature of the blended yarns were decreased slightly with the increased rPET ratio.

This study brings important contributions to development possibility of blended polyester yarns from waste and virgin PET showing comparable characteristics with that of the virgin polyester yarns. The production of final yarns can provide environmental benefits due to evaluation of rPET.

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References

[1] Li S, Vela IC, Järvien M, Seeman M. 2021. Polyethylene terephthalate (PET) recycling via steam gasification – The effect of operating conditions on gas and tar composition. Waste Management 130, 117-126.
[2] Barnes DK, Galgani F, Thompson RC, Barlaz M. 2009. Accumulation and fragmentation of plastic debris in global environments. Philos Trans R Soc B: Biological Sciences 364(1526), 1985–1998.
[3] Jambeck JR, Geyer R, Wilcox C, Siegler TR, Perryman M, Andrady A, Narayan R, Law KL. 2015. Plastic waste inputs from land into the ocean. Science 347(6223), 768–771.
[4] da Costa JP, Santos PS, Duarte AC, Rocha-Santos T. 2016. (Nano) plastics in the environment–sources, fates and effects. Sci Total Environ 566, 15–26.
[5] De Sá LC, Oliveira M, Ribeiro F, Rocha TL, Futter MN. 2018. Studies of the effects of microplastics on aquatic organisms: what do we know and where should we focus our efforts in the future? Sci Total Environ 645, 1029–1039.
[6] 2016. Plastics—the facts 2016. An analysis of European plastics production, demand and waste data. Plastics Europe.
[7] 2021. Plastics—the facts 2021. An analysis of European plastics production, demand and waste data. Plastics Europe. Retrieved from https://plasticseurope.org/wp-content/uploads/2021/12/Plastics-the-Facts-2021-web-final.pdf
[8] Lebreton L, Andrady A. 2019. Future scenarios of global plastic waste generation and disposal. Palgrave Communications 5(1), 1–11.
[9] Tournier V, Topham C, Gilles A, David B, Folgoas C, Moya-Leclair E, Kamionka E, Desrousseaux M-L, Texier H, Gavalda S. 2020. An engineered PET depolymerase to break down and recycle plastic bottles. Nature 580(7802), 216–219.
[10] Choi YJ, Kim SH. 2015. Characterization of recycled polyethylene terephthalates and polyethylene terephthalate–nylon6 blend knitted fabrics. Textile Research Journal 85(4), 337-345.
[11] Wu DY, Wang SS, Wu CS. 2021. Textile Fabrics Containing Recycled Poly (ethylene terephthalate), Oyster Shells, and Silica Aerogels with Superior Heat Insulation, Water Resistance, and Antibacterial Properties. ACS Applied Polymer Materials 2021 online publication. [https://doi.org/10.1021/acsapm.1c00365]
[12] Li G, Jiang Z, Wang W, Chu Z, Zhang Y, Wang C. 2019. Electrospun PAN/MAPbI3 composite fibers for flexible and broadband photodetectors. Nanomaterials 9(1), 50.
[13] Li J, Chao L, Liao Q, Xu Z. 2019. Environmentally-friendly technology for rapid on-line recycling of acrylonitrile-butadiene-styrene, polystyrene and polypropylene using near-infrared spectroscopy. Journal of Cleaner Production 213, 838–844.
[14] Uyanık S. 2019. A study on the suitability of which yarn number to use for recycle polyester fiber. The Journal of The Textile Institute online publication. [https://doi.org/10.1080/00405000.2018.1550889]
[15] Wu WJ, Sun XL, Chen Q, Qian Q. 2022. Recycled Poly (Ethylene Terephthalate) from Waste Textiles with Improved Thermal and Rheological Properties by Chain Extension. Polymers online publication. [https://doi.org/10.3390/polym14030510]
[16] Guo Z, Eriksson M, Motte HDL, Adolfsson E. 2021. Circular recycling of polyester textile waste using a sustainable catalyst. Journal of Cleaner Production online publication. [https://doi.org/10.1016/j.jclepro.2020.124579]
[17] Luu TTA, Baker JR. 2021. Exploring Consumers’ Purchase Intention of rPET Bottle-Based Apparel in an Emerging Economy. Journal of Open Innovation: Technology, Market, and Complexity online publication. [https://doi.org/10.3390/joitmc7010022]
[18] Özkan İ, Gündoğdu S. 2021. Investigation on the microfiber release under controlled washings from the knitted fabrics produced by recycled and virgin polyester yarns. The Journal of The Textile Institute online publication. [https://doi.org/10.1080/00405000.2020.1741760]
[19] Telli A, Özdil N. 2013. Properties of the yarns produced from r-pet fibers and their blends. Tekstil ve Konfeksiyon 23(1), 3-10
[20] TS 244 EN ISO 2060. 1999. Textiles-Yarn from packages- Determination of linear density (mass per unit length) by the skein method.
[21] DIN EN ISO 2062. 2009. Textiles - Yarns from packages - Determination of single-end breaking force and elongation at break using constant rate of extension (CRE) tester.
[22] ISO 11357-7. 2015. Plastics - Differential scanning calorimetry (DSC) - Part 7: Determination of crystallization kinetics.

[23] [https://slidetodoc.com/renk-kavramlar-eklemeli-renk-sistemimodeli-magenta-yellow-white/]
[24] Hamedi S, Koosha M. 2019. Intelligent Chitosan/PVA nanocomposite films containing black carrot anthocyanin and bentonite nanoclays with improved mechanical, thermal and antibacterial properties. Progress in Organic Coatings online publication. [https://doi.org/10.1016/j.porgcoat.2018.11.028]
[25] Terzioğlu P, Güney F, Parın FN, Şen İ, Tuna S. 2021. Biowaste orange peel incorporated chitosan/polyvinyl alcohol composite films for food packaging applications. Food Packaging and Shelf Life 30(3), 100742.
[26] Sarıoğlu E. 2017. Ecological Approaches in Textile Sector: The Effect of r-PET Blend Ratio on Ring Spun Yarn Tenacity. Periodicals of Engineering and Natural Sciences 5(2), 176-180.