

**ELECTRICAL TRANSPORT STUDIES ON
MULTIFERROIC METAL ORGANIC FRAMEWORK**

VIKAS MALIK



**DEPARTMENT OF PHYSICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI**

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by

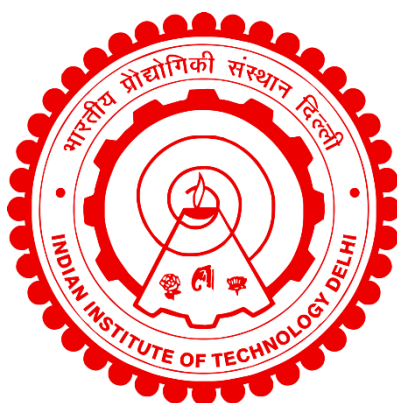
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Submitted

in fulfilment of the requirements for the degree of Doctor of philosophy

to the



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CERTIFICATE

This is to certify that the thesis entitled “**Electrical Transport Studies on Multiferroic Metal Organic Framework**” being submitted by **Mr. Vikas Malik** to Indian Institute of Technology Delhi for the award of the degree of "**Doctor of Philosophy**", is a record of the research work carried out by him under my guidance and supervision. The work or any part of the thesis have not been submitted in part or full to any other University or Institute for award of any degree or diploma.



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ABSTRACT

Organic materials are area of interest for most research projects in recent times because of their potential advantages like roll to roll production, intrinsically soft, high speed operation, and low production cost etc. over inorganic compounds. In this class, Metal Organic Framework abbreviated as MOF are the hybrid compounds in which metal ions are connected through organic ligand chain to produce a 3D structure. These MOFs club all the advantages of organic compounds with potential technicalities of inorganic materials like ferroelectricity and magnetism. The multiferroic MOFs exhibiting ferroelectric and magnetic ordering in single phase of compound have enormous application in memory and logic devices. The multiferroic MOFs displaying electronic phenomenon like negative differential resistance (NDR) can be a decent contenders for multifunctional electronic device in order to cop up the requirement of today's high technology electronic world.

This thesis discusses the detailed magnetic and electrical studies of two specific MOF systems namely Dimethyl-ammonium manganese Formate (DMnF) with chemical formula $(\text{CH}_3)_2\text{NH}_2\text{Mn}[\text{HCOO}]_3$ and Dimethyl-ammonium Iron Formate (DFeF) with formula $(\text{CH}_3)_2\text{NH}_2\text{Fe}[\text{HCOO}]_3$.

The multiferroic behavior of both DMnF and DFeF are very well established in literature and also confirmed in our work through magnetic and dielectric property measurements. The electronic conduction mechanism for this class of materials is still to be explored. This gap in literature need to be filled.

We explores the scope of an electronic phase transition alongwith the dielectric phase transition by studying current-voltage charateritics for DMnF compound (chapter 3). NDR

behaviour is noticed first time in this type of hybrid compounds with multiferroic nature having a peak to valley ratio (PVR) of 10.

Higher PVR value is estimated for DFeF MOF compound by comparing its electronic configuration with DMnF and relating it with onsite coulomb intrations in Hubbard model.

NDR characteristics are observed for DFeF with a increased PVR value of 47 (chapter 4).

Finally we investigate DFeF crystals magnetically in detail as the presence of blocking temperature T_B points toward the possibility of metastable phase existing near magnetic phase transition temperature and requires a direct magnetic study to specify the magnetic interactions for this MOF (chapter 5).

The research project has been divided into six sections with the formation given below:

Chapter 1 (Introduction) includes the outline of ongoing research progress in this work field, choice of material and the comprehensive goal of this thesis. **Chapter 2** consists of precise idea of synthesis methods used to prepare MOFs samples and different characterization techniques utilized for the present work.

Chapter 3 (Temperature dependent negative differential resistance behavior in multiferroic metal organic framework $(\text{CH}_3)_2\text{NH}_2 \text{Mn} (\text{HCOO})_3$ crystals): We confirm the multiferroic nature of DMnF MOF system through temperature based magnetic and dielectric characterizations. The electrical transport properties are investigated through current-voltage I(V) correspondance. A NDR behaviour is observed in the low bias region (between -4 V to +4 V) for the temperature ranging from 100 K to 184 K with highest peak to valley ratio (PVR) of 10 at 176 K.

*A part of this research work is published in **Organic Electronics 56 (2018) 5–10.***

Chapter 4 (Negative differential resistance behaviour with high PVR value in $(\text{CH}_3)_2\text{NH}_2\text{Fe}(\text{HCOO})_3$ – a multiferroic MOF): We present the NDR characteristic behaviour for multiferroic MOF DFeF crystals in the low bias region from 0 to 3V. The multiferroic character of DFeF MOF has been confirmed through a para-antiferromagnetic transition observed at 19 K and a para-ferroelectric transition at 171 K. The largest PVR value of 47 is observed at 140 K for DFeF where in it is 10 for DMnF MOF. This increase in PVR value is explained on the basis of onsite coulomb interactions in 1D Hubbard model.

- *A part of this research work is published in **Material Science & Engineering B 294 (2023) 116534**.*

Chapter 5 (Study of the nature of magnetic phase transition in multiferroic metal organic framework $[(\text{CH}_3)_2\text{NH}_2] \text{Fe} (\text{HCOO})_3$): We investigate nature of magnetic phase transition in DFeF MOF system using intense magnetic characteristic measurements. Here we confirms the presence of metastable state using FCC-FCW magnetization measurements at different applied field, Time based magnetic characterization at stable temperature and magnetic field, KWW exponential equation fitting and by employing different cooling rates for M-T curves at a constant magnetic field.

Chapter 6 summarizes the principal findings of the present study and also scope of further works in this field.

सार

अकार्बनिक यौगिकों पर रोल टू रोल उत्पादन, आंतरिक रूप से नरम, उच्च गति संचालन और कम उत्पादन लागत आदि जैसे संभावित लाभों के कारण हाल के दिनों में अधिकांश अनुसंधान परियोजनाओं के लिए कार्बनिक सामग्री रुचि का क्षेत्र है। इस वर्ग में, मेटल ऑर्गेनिक फ्रेमवर्क को एमओएफ के रूप में संक्षिप्त किया गया है, जो हाइब्रिड यौगिक हैं जिसमें धातु आयनों को 3 डी संरचना का निर्माण करने के लिए कार्बनिक लिगेंड श्रृंखला के माध्यम से जोड़ा जाता है। ये एमओएफ कार्बनिक यौगिकों के सभी लाभों को फेरोइलेक्ट्रिसिटी और चुंबकत्व जैसे अकार्बनिक सामग्रियों की संभावित तकनीकीताओं के साथ जोड़ते हैं। किसी कंपाउंड के सिंगल फेज में फेरोइलेक्ट्रिक और मैग्नेटिक ऑर्डरिंग को प्रदर्शित करने वाले मल्टीफेरोइक एमओएफ की मेमोरी और लॉजिक डिवाइसेस में भारी उपयोग है। आज की उच्च प्रौद्योगिकी इलेक्ट्रॉनिक दुनिया की आवश्यकता को पूरा करने के लिए नकारात्मक अंतर प्रतिरोध (एनडीआर) जैसी इलेक्ट्रॉनिक घटना को प्रदर्शित करने वाले मल्टीफ़ेरोइक एमओएफ बहुक्रियाशील इलेक्ट्रॉनिक उपकरण के लिए एक अच्छे दावेदार हो सकते हैं।

यह शोध-ग्रंथ दो विशिष्ट एमओएफ प्रणालियों के विस्तृत चुंबकीय और विद्युत अध्ययन पर चर्चा करती है, रासायनिक सूत्र $(\text{CH}_3)_2\text{NH}_2\text{Mn}[\text{HCOO}]_3$ के साथ डाइमिथाइल-अमोनियम मैंगनीज फॉर्मेट (DMnF) और डाइमिथाइल-अमोनियम आयरन फॉर्मेट (DFeF), $(\text{CH}_3)_2\text{NH}_2\text{Fe}[\text{HCOO}]_3$ सूत्र के साथ।

DMnF और DFeF दोनों के मल्टीफेरोइक व्यवहार को साहित्य में बहुत अच्छी तरह से स्थापित किया गया है और चुंबकीय और परावद्युत माप के माध्यम से हमारे काम में भी इसकी पुष्टि की गई

है। सामग्री के इस वर्ग के लिए इलेक्ट्रॉनिक चालन तंत्र का पता लगाना अभी बाकी है। साहित्य में इस कमी को भरने की जरूरत है।

हम DMnF कंपाउंड (अध्याय 3) के लिए विद्युत प्रवाह-वोल्टेज विशेषताओं का अध्ययन करके परावद्युत अवस्था परिवर्तन के साथ-साथ एक इलेक्ट्रॉनिक अवस्था परिवर्तन के दायरे की पड़ताल करते हैं। एनडीआर व्यवहार इस प्रकार के हाइब्रिड यौगिकों में पहली बार देखा गया है, जिसमें 10 के शिखर से घाटी अनुपात (पीवीआर) की बहुलौह प्रकृति होती है।

डीएफईएफ एमओएफ कंपाउंड के लिए डीएमएनएफ के साथ इलेक्ट्रॉनिक विन्यास की तुलना करके और हबर्ड मॉडल में ऑनसाइट कॉलम इंटरेंशंस के साथ उच्च पीवीआर मूल्य का अनुमान लगाया गया है। 47 (अध्याय 4) के बढ़े हुए पीवीआर मूल्य के साथ डीएफईएफ के लिए एनडीआर विशेषताएँ देखी जाती हैं।

अंत में हम DFeF क्रिस्टल की चुंबकीय रूप से विस्तार से जांच करते हैं क्योंकि अवरुद्ध तापमान T_B की उपस्थिति चुंबकीय अवस्था परिवर्तन तापमान के पास मौजूद मेटास्टेबल चरण की संभावना की ओर इशारा करती है और इस यौगिक (अध्याय 5) के लिए चुंबकीय बातचीत को समझने के लिए प्रत्यक्ष चुंबकीय अध्ययन की आवश्यकता होती है।

इस शोध-ग्रंथ के कार्य को नीचे दिए गए विवरण के साथ सात खंडों में विभाजित किया गया है:

अध्याय 1 (परिचय) में इस कार्य क्षेत्र में चल रही शोध प्रगति की रूपरेखा, सामग्री का चुनाव और इस थीसिस का व्यापक लक्ष्य शामिल है। **अध्याय 2** में एमओएफ के नमूने तैयार करने के लिए प्रयुक्त संश्लेषण विधियों के सटीक विचार और वर्तमान कार्य के लिए उपयोग की जाने वाली विभिन्न लक्षण वर्णन तकनीक शामिल हैं।

अध्याय 3 (मल्टीफेरोइक मेटल ऑर्गेनिक फ्रेमवर्क $(\text{CH}_3)_2\text{NH}_2 \text{Mn} (\text{HCOO})_3$ क्रिस्टल में तापमान पर निर्भर नकारात्मक अंतर प्रतिरोध व्यवहार): हम तापमान पर निर्भर चुंबकीय और परावद्युत माप के माध्यम से DMnF एमओएफ प्रणाली की बहुपरत प्रकृति की पुष्टि करते हैं। विद्युत परिवहन गुणों की जांच विद्युत प्रवाह-वोल्टेज $I(V)$ विशेषता अध्ययन के माध्यम से की गई है। 100 K से 184 K तक के तापमान के लिए कम विद्युत बायस क्षेत्र (-4 V से +4 V के बीच) में एक एनडीआर व्यवहार देखा गया है, जिसमें 176 K पर 10 का उच्चतम शिखर से घाटी अनुपात (PVR) है।

इस शोध कार्य का एक अंश Organic Electronics 56 (2018) 5–10 में प्रकाशित हुआ है।

अध्याय 4 ($(\text{CH}_3)_2\text{NH}_2 \text{Fe} (\text{HCOO})_3$ - एक मल्टीफेरोइक एमओएफ में उच्च पीवीआर मान के साथ नकारात्मक अंतर प्रतिरोध व्यवहार): हम 0 से 3V तक निम्न बायस क्षेत्र में मल्टीफेरोइक एमओएफ DFeF क्रिस्टल के लिए एनडीआर विशेषता व्यवहार प्रस्तुत करते हैं। DFeF एमओएफ के मल्टीफेरोइक चरित्र की पुष्टि 19 K पर देखे गए अनुचुंबकीय - प्रति-लौहचुंबकीय संक्रमण और 171 K पर पैराइलेक्ट्रिक-फेरोइलेक्ट्रिक संक्रमण के माध्यम से की गई है। 47 का सबसे बड़ा पीवीआर मूल्य DFeF के लिए 140 K पर मिला है जहां DMnF एमओएफ के लिए यह 10 है। पीवीआर वैल्यू में इस वृद्धि को 1डी हबर्ड मॉडल में ऑनसाइट कूलम्ब इंटरैक्शन के आधार पर समझाया गया है।

- *इस शोध कार्य का एक अंश Material Science & Engineering B 294 (2023) 116534*

में प्रकाशित हुआ है।

अध्याय 5 (मल्टीफेरोइक धातु कार्बनिक ढांचे $(\text{CH}_3)_2\text{NH}_2 \text{Fe} (\text{HCOO})_3$ s में चुंबकीय चरण संक्रमण की प्रकृति का अध्ययन): हम तीव्र चुंबकीय विशेषता माप का उपयोग करके DFeF

एमओएफ प्रणाली में चुंबकीय चरण संक्रमण की प्रकृति की जांच करते हैं। यहां हम अलग-अलग लागू क्षेत्र में एफसीसी- एफसीडब्ल्यू चुंबकीयकरण मापन, स्थिर तापमान और चुंबकीय क्षेत्र पर समय आधारित चुंबकीय लक्षण वर्णन, केडब्ल्यूडब्ल्यू घातीय समीकरण फिटिंग और एक निरंतर चुंबकीय क्षेत्र में एम-टी घटता के लिए विभिन्न शीतलन दरों को नियोजित करके मेटास्टेबल स्थिति की उपस्थिति की पुष्टि करते हैं।

अध्याय 6 वर्तमान अध्ययन के प्रमुख निष्कर्षों का सार प्रस्तुत करता है और इस क्षेत्र में आगे के कार्यों का दायरा भी बताता है।

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LIST OF ABBREVIATIONS

NDR	: Negative Differential Resistance
PDR	: Positive Differential Resistance
PVR	: Peak to Valley Ratio
RTD	: Resonant Tunneling Diode
MOF	: Metal Organic Framework
DMA	: Dimethylammonium
DMnF	: Dimethylammonium Manganese Formate
DFeF	: Dimethylammonium Iron Formate
FN	: Fowler Nordheim
DMF	: Dimethylformamide
ZFC	: Zero Field Cooled
FC	: Field Cooled
FCC	: Field Cooled cooling
FCW	: Field Cooled Warming
TMI	: Thermo-magnetic Irreversibility
XRD	: X-Ray Diffraction
SEM	: Scanning Electron Microscopy
SQUID	: Superconducting Quantum Interference Device
AFM	: Antiferromagnetic
FE	: Ferroelectric
PE	: Paraelectric
PM	: Paramagnetic

- I-V** : **Current vs. Voltage Graph**
- M-T** : **Magnetization vs. Temperature Plot**
- M-H** : **Magnetization vs. Magnetic Field Hysteresis Loop**
- P-E** : **Polarization vs. Electric Field Hysteresis Loop**