

To

25-05-2015

Dr. N. Gopakumar,  
Deputy Secretary & Regional Head,  
University Grants Commission,  
South Western Regional Office,  
Gandhinagar, Bangalore – 560 009.

Sub: Final report of the Minor Research Project – Reg.  
Ref.: MRP(S)–1353/11-12/KLCA008/UGC-SWROdt. 24Sept2013.

Dear Sir,

Enclosed with please find a copy of Final Report of the Minor Research Project entitled **“Applications of diazo compounds and their metal chelates.”**. The report is for the period from 13<sup>th</sup>December 2013 to 12<sup>th</sup> June 2015 and the work is carried out at Christ College, Irinjalakuda. Kindly accept the final report. Objectives of the project was fulfilled. Kindly accept the finalreprt. I take this opportunity to thank UGC for the financial help extended to me for completing this minor research project.

Thanking you,  
Yours faithfully,

John.V.D, Department of Chemistry,  
Christ College, Irinjalakuda-680125,  
Thrissur (Dt.) Kerala, Phone : 0487-2384435  
Email: john\_vd@rediffmail.com.

Enclosed

1. Annexure III
2. Annexure III
3. Annexure III
4. Annexure III
5. Annexure III
6. Bound copy of report of the work done
7. CD(copy of final report of work done)

**PRINCIPAL**

**Christ College, Irinjalakuda**

UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI – 110 002

**STATEMENT OF EXPENDITURE IN RESPECT OF MINOR RESEARCH PROJECT**

1. Name of Principal Investigator: *Dr John V D*

2. Dept of University/College: *Christ college, Irinjalakuda*

3. UGC approval No. and Date

*Reference No. MRP(S)- 1353/11-12/KLCA008/UGC-SWRO, dated 24<sup>th</sup> September 2013.*

4. Title of the Research Project **“Applications of diazo compounds and their metal chelates”**.

5. Effective date of starting the project : 13<sup>th</sup> December 2013

6. a. Period of Expenditure: From 13<sup>th</sup> December 2013 to 12<sup>th</sup> June, 2015

6. b. Details of Expenditure

Sl no	Item	Amount Approved 1,60,000 (Rs)	Amount Sanctioned 1,10,000(Rs)	Expenditure incurred (Rs)
Non-Recurring grant				
1.	Books and Journals	10,000	10,000	10,181
2.	Equipment	50,000	50,000	51,292
Recurring grant				
3.	Contingency including special needs	20,000	10,000	23,177
4.	Chemicals and Glasswares	70,000	35,000	71338
5.	Field work and travel	10,000	5,000	10,986
<b>Total</b>		<b>160,000</b>	<b>1,10,000</b>	<b>1,66,974</b>

1. It is certified that the appointment(s) have been made in accordance with the terms and conditions laid down by the Commission.

2. It as a result of check or audit objective, some irregularly is noticed, later date, action will be taken to refund, adjust or regularize the objected amounts.

3. Payment @ revised rates shall be made with arrears on the availability of additional funds.

4. It is certified that the grant of Rs. 1,10,000 (Rupees Eighty Thousand only) received from the University Grants Commission under the scheme of support for Minor Research Project entitled “**Applications of diazo compounds and their metal chelates**”. vide UGC letter No. *MRP(S)-1353/11-12/KLCA008/UGC-SWRO*, dated 24<sup>th</sup> September 2013 has been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission.

Principal,  
Christ College Irinjalakuda

Signature of principal investigator

Dr. John V. D.

Associate Professor

Christ College Irinjalakuda

**UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI – 110 002  
STATEMENT OF EXPENDITURE INCURRED ON FIELD WORK**

Name of the Place visited	Duration of the Visit		Mode of Journey	Expenditure Incurred (Rs.)
	From	To		
Calicut University, Library	23 July, 2013	25 July, 2013	Special conveyance	Rs 1645.00
STIC, Cochin University	12 December, 2013	13 December, 2013	Special conveyance	Rs 1091.00
Calicut University, Chemistry Dept	2 January, 2014	3 January, 2014	Special conveyance	Rs 1645.00
NIIST, Thiruvannthapuram	9 February, 2014	11 February, 2014	Special conveyance	Rs 2550.00
Calicut University, Library	11 March, 2014	13 March, 2014	Special conveyance	Rs 1645.00
M G University, School of Chemical Sciences	23 June, 2014	24 June, 2014	Special conveyance	Rs 1205.00
M G University, School of Chemical Sciences	30 September, 2014	1 October, 2014	Special conveyance	Rs 1205.00
<b>Total Expenditure</b>				<b>Rs 10,986.00</b>

Certified that the above expenditure is in accordance with the UGC norms for Minor Research Projects.

**PRINCIPAL**

**SIGNATURE OF PRINCIPAL INVESTIGATOR**

Dr. John V D

(Seal)

**UNIVERSITY GRANTS COMMISSION**  
**BAHADUR SHAH ZAFAR MARG**  
**NEW DELHI –110 002**

Certified that the grant of Rs.1,60,000(Rupees One lakh sixty thousand only) approved by the University Grants Commission (out of which Rs.1,10,000/- one lakh ten thousand only has been received from UGC) under the scheme for Minor Research Project to Dr JOHN V D, Associate Professor, Christ College, Irinjalakuda entitled “**Applications of diazo compounds and their metal chelates**” vide UGC letter No.MRP (S)-1353/11-12/KLCA008/UGC-SWRO, dated 24-09-2013 has been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission.

Signature of the Principal Investigator

Principal

Statutory Auditor

ANNEXURE VI  
UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI-110002.

Final Report of the work done on the Minor Research Project.

(Report to be submitted within 6 weeks after completion of each year)

1. Project report No.      Final.
2. UGC Reference No.    MRP(S) 1353/11-12/KLCA008/UGC.SWRO
3. Period of report from : 13/12/2013 to 12/06/2015.
4. Title of research project, *Applications of diazo compounds and their metal chelates.*
5. (a) Name of the Principal Investigator: Dr JOHN.V.D.  
(b) Dept and University/College where work has progressed: Christ College, Irinjalakuda.
6. Effective date of starting of the project: 13/12/2013.
7. Grant approved and expenditure incurred during the period of the report:
  - a). Total amount approved: Rs.1,60,000/-
  - b). Total expenditure: Rs.1,60,000/-
  - c). Report of the work done: (Please attach a separate sheet): Attached separate sheet.
8. a) Brief objective of the project:
  - 1) To make a scientific study about the curcuminoid analogues, the metal chelates and their utility as antitumour, antibacterial and antioxidant agents..
  - 2) To characterize the curcuminoid analogues and metal chelates by elemental analysis, magnetic measurements, IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, TG and Mass Spectral analysis.
  - 3) To synthesize phenyl azo derivatives by reacting curcuminoid analogues with benzene diazonium chloride and converting the phenylazo derivatives into metal complexes.
  - 4) To carry out antitumour studies and antibacterial studies with these compounds.
  - 5) To find out heavy metals present in the effluent water by complexing with phenyl azo derivatives and curcuminoid analogues.

b). Work done so far and results achieved and publications, if any, resulting from the work (Give details of the papers and names of the journals in which it has been published or accepted for publication).

1. **John V D**, Seena Thomachan and Sindhu S, “Antitumour and antimicrobial activities of chloro derivatives of Synthetic Curcumin and their metal chelates”, *International Journal of Pharmaceutical chemistry*, 05(02), 2015,45-51.
2. **John V D**, Seena Thomachan and Sindhu S, Cytotoxic and antimicrobial studies of curcuminoid analogues and their metal chelates, *Amala Research Bulletin*, 34, 2014, 158-162.
3. **John V D**, Seena Thomachan and Sindhu S, “ Biochemical activities of main group metal chelates of curcuminoid analogues.” *International Journal of Biomedical and Advanced Research*.6(04),2015,355-362

c). Has the progress been according to original plan of work and towards achieving the objective if not, state reasons:

Yes

d). Please indicate the difficulties,if any,experienced in implementing the project:

Nil

e) If project has not been completed, please indicate the approximate time by which it is likely to be completed. A summary of the work done for the period (Annual basis) may please be sent to the Commission on a separate sheet

Project completed.

f) If the project has been completed, please enclose a summary of the findings of the study. Two bound copies of the final report of work done may also be sent to the Commission :

Summary in separate sheet and two bound copies of the project are attached.

g) Any other information which would help in evaluation of work done on the project. At the completion of the project, the first report should indicate the output, such as (a) Manpower trained (b) Ph. D. awarded (c) Publication of results (d) other impact, if any

One student (Sindhu S) has utilized the project contents for part of her PhD programme.

**Signature of Principal Investigator**

**Principal, Christ College, IJK**

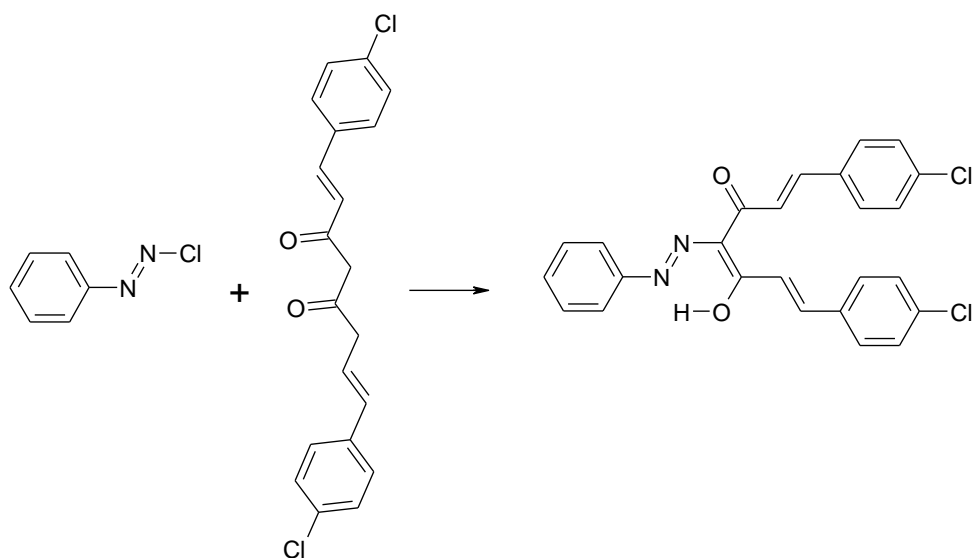
## 10. Report of the work done

### First year

For preliminary studies and literature survey utilized 3 months. In the next six months 7 new curcuminoid analogues ( 1,7-diarylheptanoids ) were synthesized using seven different aldehydes ( p-chloro benzaldehyde, o-chloro benzaldehyde, 3,4-dichloro benzaldehyde, thiophene-2-carboxaldehyde, 3-methyl thiophene-2-carboxaldehyde, 2,4-dihydroxy benzaldehyde, 2,5-dimethyl benzaldehyde). Aldehydes were condensed with acetyl acetone-boron complex. Using column chromatography using silica gel (60-120 mesh) the curcuminoids were separated and purified. In the next 3 months characterization of curcuminoid analogues were carried out. Elemental analysis, UV spectra, IR spectra, H NMR, <sup>13</sup>C NMR , TG, and mass spectral analysis were carried out for characterizing the synthesized compounds.

### Second year

Antitumour and antibacterial studies were conducted with the curcuminoids for the next 3 months. Preparation of phenylazo derivatives were carried out in the next 3 months. The characterized 1,7-diarylheptanoids were converted into phenyl azo derivatives by reaction with benzene diazonium chloride . For characterizing phenyl azo derivatives 3months were utilised. Preparation of metal complexes of lead and cadmium as well as their characterization needed 6months.



Colour changes produced during complex formation was utilized for detecting heavy metal ions in waste water released from KLF Oil mills, Irinjalakuda. By using these types of ligands heavy metals present in the effluent water can be removed. Complexes of Pb and Cd were also detected. A suspension of the azo compound was prepared in ethanol and to this suspension drops of metal salts were added. The colour change indicates the complex formation.



**UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFARMARG  
NEW DELHI – 110 002**

**PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF SENDING  
THE FINAL REPORT OF THE WORK DONE ON THE PROJECT**

1. Name and address of the Principal Investigator:  
JOHN. V.D.,  
ASSOCIATE PROFESSOR  
DEPARTMENT OF CHEMISTRY  
CHRIST COLLEGE, IRINJALAKUDA  
KERALA-680125
2. Name and address of the institution: CHRIST COLLEGE, IRINJALAKUDA.
3. UGC APPROVAL NO. AND DATE: MRP(S)1353/11-12/KLCA008/UGC.SWRO  
dated 24<sup>th</sup> September, 2013
4. DATE OF IMPLEMENTATION : 13-12-2013
5. TENURE OF THE PROJECT : 18 Months (13-12-2013 TO 12-06-2015)
6. TOTAL GRANT ALLOCATED : 1,60,000 (Rupees one lakh and sixty thousand)
7. TOTAL GRANT RECEIVED : 1,10,000 (Rupees one lakh and ten thousand)
8. FINAL EXPENDITURE : 1,66,974 (Rupees one lakh , sixty six thousand  
nine hundred and seventy four)
9. TITLE OF THE PROJECT : **“Applications of diazo compounds and their  
metal chelates”**
10. OBJECTIVES OF THE PROJECT
- 1) To make a scientific study about the curcuminoid analogues and the metal chelates.
  - 2) To characterize the curcuminoid analogues and metal chelates by elemental analysis, magnetic measurements, IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, TG and Mass Spectral analysis.
  - 3) To synthesize phenylazo derivatives by reacting curcuminoid analogues with benzene diazonium chloride and converting the phenylazo derivatives into metal complexes.

4) To carry out antitumour studies and antibacterial studies with curcuminoid analogues and metal complexes. Also complexation of these azo compounds with metals Cd, Pb, were carried out. The colour change produced during complexation was utilized for identifying the metal ions present in the effluent water produced in the KLF Oil mills, Irinjalakuda.

11. WETHER OBJECTIVES WERE ACHIEVED : .....

(GIVE DETAILS)

We could successfully complete the project, by synthesizing seven new curcuminoid analogues (1,7-diarylheptanoids by condensing acetylacetone with seven different aldehydes (p-chloro benzaldehyde, o-chloro benzaldehyde, 3,4-dichloro benzaldehyde, thiophene-2-carboxaldehyde, 3-methyl thiophene-2-carboxaldehyde, 2,4-dihydroxy benzaldehyde, 2,5-dimethyl benzaldehyde. These compounds were characterized by C,H,N analysis, UV, IR, Mass and other spectral techniques. By reacting these compounds with benzene diazonium chloride phenylazo derivatives were prepared, they were characterized with various spectral techniques. Metal complexes of Pb, Cd, were prepared. Optical density of coloured metal complexes were determined after dilution. Effluent water was collected from KLF Oil mills, Irinjalakuda. The collected water was treated with the curcuminoid analogues and phenyl azo derivatives for complexation. From the colour developed and measurement of optical density presence metal ions were predicted. Curcuminoid analogues and metal complexes were scanned for antimicrobial, *invitro* cytotoxic studies, *invivo* antitumour studies and antioxidant studies. Thus all the objectives were achieved.

12. ACHIEVEMENTS FROM THE PROJECT

We could synthesize seven new curcuminoid analogues and their metal chelates. These curcuminoid analogues play vital roles as antitumour agents, as antibacterial agents and as antioxidants. The phenylazo derivatives form coloured metal chelates with heavy metal ions. Thus they can be utilized as heavy metal scavengers from effluent water from industries. Three papers were published.

1. **John V D**, Seena Thomachan and Sindhu S, “Antitumour and antimicrobial activities of chloro derivatives of Synthetic Curcumin and their metal chelates”, *International Journal of Pharmaceutical chemistry*, 05(02), 2015,45-51.
2. **John V D**, Seena Thomachan and Sindhu S, Cytotoxic and antimicrobial studies of curcuminoid analogues and their metal chelates, *Amala Research Bulletin*, 34, 2014, 158-162.
3. **John V D**, Seena Thomachan and Sindhu S, “ Biochemical activities of main group metal chelates of curcuminoid analogues.” *International Journal of Biomedical and Advanced Research*.6 (04),2015,355-362

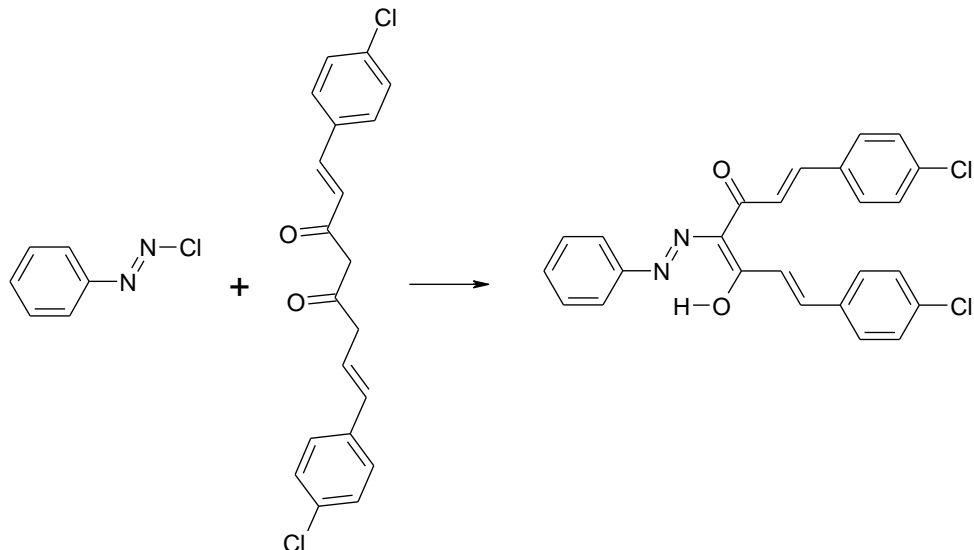
The research work carried out in the project is utilized in the PhD programme of one student S. Sindhu. We could develop antitumour drugs after conducting further studies. The project also helps one to find out the heavy metals present in effluent water and remove them.

### 13. SUMMARY OF THE FINDINGS .....

#### *SUMMARY*

In recent years research in the field of coordination chemistry of biologically important materials has gained considerable momentum. This is because metal complexes play numerous vital roles in the chemistry of living matter. Similarly it has been revealed that the biological significance, especially medicinal importance, of many plant chemicals are associated with their ability to form complexes, with various inorganic species such as metal ions. However structural and biochemical aspects of these types of complexes has not received as much attention as they deserve. Thus chemical and biochemical examination of active plant products have considerable importance. The present investigation has been so designed as to provide some insight on various chemical and biochemical aspects of metal complexes of certain synthetic analogues of the active chemical components, present in the traditional Indian medicinal plant turmeric namely curcuminoids. Structurally curcuminoids are 1,7-diarylhepta-1,6-diene-3,5-diones and a group of naturally occurring 1,3-diketones in which the diketo function is directly linked to olefinic groups.

Seven new curcuminoid analogues ( 1,7-diarylheptanoids ) were synthesized using seven different aldehydes ( p-chloro benzaldehyde, o-chloro benzaldehyde, 3,4-dichloro benzaldehyde, thiophene-2-carboxaldehyde, 3-methylthiophene-2-carboxaldehyde, 2,4-dihydroxy benzaldehyde, 2,5-dimethyl benzaldehyde). Aldehydes were condensed with acetyl acetone-boron complex. By column chromatography using silica gel (60-120 mesh) the curcuminoids were separated and purified. Characterization of curcuminoid analogues were carried out by Elemental analysis, UV spectra, IR spectra, H NMR, <sup>13</sup> C NMR , TG, and mass spectral analysis. Antitumour and antibacterial studies were conducted with the curcuminoids. The characterized 1,7-diarylheptanoids were converted into phenyl azo derivatives by reaction with benzene diazonium chloride . Elemental analysis, UV spectra, IR spectra, H NMR, <sup>13</sup> C NMR , TG, and mass spectral analysis were carried out for characterizing the phenyl azo derivatives. Preparation of metal complexes of lead and cadmium as well as their characterization were done.



Colour changes produced during complex formation was utilized for detecting heavy metal ions in waste water released from KLF Oil mills, Irinjalakuda. By using these types of ligands heavy metals present in the effluent water can be removed. Complexes of Hg and Sn were also detected. A suspension of the azo compound was prepared in ethanol and to this suspension drops of metal salts were added. The colour change indicates the complex formation.

#### 14.CONTRIBUTION OF THE SOCIETY..... (GIVE DETAILS)

The synthesis of new curcuminoid analogues were carried out in such a way that they are more advantageous to be used as antitumour, antioxidant and antibacterial agents. Aldehydes were selected according to the above concept. Curcuminoid analogues were characterized using CHN analysis, UV, IR, NMR and Mass spectral analysis. Antitumour, antibacterial and antioxidant studies were also conducted. Among the synthesized compounds the highly active compounds were selected for further studies. This will be useful to the society. Three research papers were published out of this.

**1. John V D**, Seena Thomachan and Sindhu S, “Antitumour and antimicrobial activities of chloro derivatives of Synthetic Curcumin and their metal chelates”, *International Journal of Pharmaceuticalchemistry*, 05(02), 2015,45-51.

2. **John V D**, Seena Thomachan and Sindhu S, Cytotoxic and antimicrobial studies of curcuminoid analogues and their metal chelates, *Amala Research Bulletin*, 34, 2014,158-162.

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Detection and removal of heavy metal ions from effluent water is also advantageous to Society. Polluted water can be detected and heavy metal ion can be removed from it by using phenylazo compounds, which easily form coloured products.

15. WHETHER ANY Ph.D. ENROLLED/PRODUCED AS A PART OF THIS PROJECT:

One student, Sindhu S has enrolled for PhD programme.

16. NO. OF PUBLICATIONS OUT OF THE PROEJCT : Three

1. **John V D**, Seena Thomachan and Sindhu S, “Antitumour and antimicrobial activities of chloro derivatives of Synthetic Curcumin and their metal chelates”, *International Journal of Pharmaceuticalchemistry*, 05(02), 2015,45-51.

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PRINCIPAL INVESTIGATOR

REGISTRAR / PRINCIPAL

### **ACKNOWLEDGE MENT**

I gratefully acknowledge the financial support of University of Grants Commission for completing this minor research project successfully.

I am much obliged to the Principal, Christ College, Irinjalakuda and the Head of Department of Chemistry for providing me the adequate facilities for the experimental studies.

I would like to acknowledge my gratitude to my colleagues of Christ College, Irinjalakuda for their valuable advice and timely help in completing the project.

**Dr. John V D**

Associate Professr,

Dept.of Chemistry

Christ College, Irinjalakuda-680125