Krishna Mahavidyalaya, Rethare Bk

IQAC 2021-2022

ACTIVITY REPORT PHYSICS DEPARTMENT

IQAC ACTIVITY No:

DATE		ermal route for supercapacitor applics	COURDIN			
DATE	FACULTY	DEPARTMENT/COMMITTEE	NAME			
Through-out the academic year	Science	Physics	Dr. Dhanaji S. Dalavi			
TIME	VENUE	NUMBER OF PARTICIPANTS	NATURE: Outdoor/Indoor			
12.30 to 2: 00 pm	Physics laboratory	04 students+01 teacher	Indoor			
SUPPORT/ASSISTANCE:	CE: Nil					

BRIEF INFORMATION ABOUT THE ACTIVITY (CRITERION NO. -):

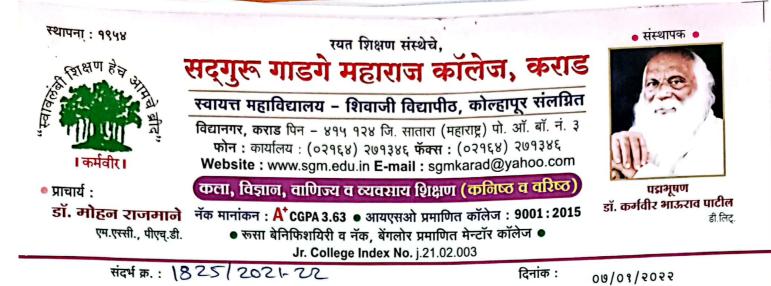
TOPIC/SUBJECT OF THE ACTIVITY	Synthesis and Characterization of Co3O4 thin film by hydrothermal route for supercapacitor application
OBJECTIVES	to analyse a scientific occurrence with an investigation or to solve a problem with an invention.
METHODOLOGY	Experimental work
OUTCOMES	students become active, engaged learners. It also helped students to develop independent critical thinking skills,

PROOFS & DOCUMENTS ATTACHED (Tick mark the proofs attached):

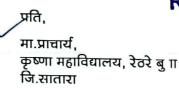
1.	Notice & Letters	2.	Student list of	3.	Activity report	4.	Photos	5.	Feedback form
6.	Feedback analysis	7.	News clip with details	8.	Certificate	9.	Any other	10	•

IQAC CELL ACTIVITY NUMBER:

- pa	Jule Dalan.
Den	IQAC, Coordinator, KRISHNA MAHAVIDYALAYA Rethare Bk; Shivnagar - 41510
	Man S. Dalavi PHYSICS Krishna Mahavi YALAYA, Tal Karad



REGISTERED AD



विषय : अग्रणी महाविद्यालय योजनेअंतर्गत रिसर्च प्रोजेक्टसाठी द्यावयाच्या ॲडव्हान्सबाबत.

महोदय,

शिवाजी विद्यापीठाच्या Research Promotion Activity for students of the affiliated Colleges या योजने अंतर्गत आपल्या विद्यार्थ्यांच्या "Synsthesis and Characterization of CO3O4 this film by hydrothermal route for supercapacitor application" या रिसर्च प्रपोजलसाठी विद्यापीठाने रू. १००००/– मंजूर केले असून सदर ॲडव्हान्स रकमेचा चेक नं. 🕉 🗲 ज्य दि. ९८/०१/२०२२ ने सोबत पाठविला आहे. कृपया सदर रकमेची पोहोच पावती त्वरीत पाठवून द्यावी.

वरील रिसर्च प्रोजेक्टसाठी मंजूर रक्कम रू.१०,०००/- खर्च करून त्याचा हिशोब व प्रोजेक्ट रिपोर्ट या अग्रणी महाविद्यालयाकडे ३१/०३/२०२२ पूर्वी सादर करावा.

कळावे, ही विनंती.

आपला विश्वार

पाना

अग्रणी महाविद्यालय सद्गुरू गाडगे महाराज कॉलेज, कराड भ

सोबत : वरीलप्रमाणे.





संहती कार्यसाधिका । शिलं परं भूषणम् Shetkari Shikshan Prasarak Mandal's

KRISHNA MAHAVIDYALAYA, RETHARE BK.

Shivnagar, Tal. Karad, Dist. Satara, 415 108 (M.S.). Phone : 02164-266346, Fax : 02164-266347 Email : kmr_sspm@yahoo.co.in Website : www.krishnamahavidyalaya.com NAAC 'B+' Grade (CGPA 2.65)

Founder : Hon. Jaywantrao Bhosale

President : Dr. Suresh Jaywantrao Bhosale Principal : Dr. Salunkhe C. B. M.Sc., Ph. D.

Ref. No. : KMR/ 1046/2021-22 प्रति

> मा . समन्वयक अग्रणी महाविद्यालय सदगुरू गाडगे महाराज कॉलेज कराड जि . सातारा .

Date: 19-09-2022.

विपय :- अग्रणी महाविद्यालया अंतर्गत राबवलेल्या रिसर्च प्रोजेक्टच्या खर्चाचा अहवाल.

महोदय.

शिवाजी विद्यापीठाच्या Research Promotion activity for students of the affiliated collages या योजनेअंतर्गत या महाविद्यालयातील विद्यार्थ्यांच्या "Synthesis and Characterization of Co3O4 thin film by hydrothermal route for supercapacitor application" या रिसर्च प्रोपोसलं साठी विद्यापीठाने रु. १००००/- मंजूर केले होते. सदर रिसर्च प्रोपोसलं साठी आपणाकडून रु. १००००/- चा ऍडव्हान्स रकमेचा चेक नं. 138027 दि. 17/०1/२०22 रोजी मिळाला असून त्यानुसार वरील रिसर्च प्रोजेक्टसाठी मंजूर रकमेच्या खर्चाचा तपशील व अहवाल आपणाकडे पाठवीत आहे.

तरी उर्वरित रु.109/- या महाविद्यालयास पाठविण्यात यावेत हि नम विनंती

खर्चाचा तपशील खालीलप्रमाणे.

अ ∙ नं	तपशील	साहित्य	खर्च
१	Shri. Samarth Trading Company	Chemicals	8644.00
२	Anushka Stationary and General Stores	Stationary	440.00
¥	Shivshakti Light House	Pencil Cells and wire cutter	220.00
	Rajan Rubber stamp and office stationers	Stationary	450.00
8	Uma Stores and Xerox	Stationery, printing and binding	355.00
		Total	10,109=00

Principal Krishna Mahavidyalaya, Rethare Bk.

समन्वयक

FORMAT FOR STATEMENT OF EXPENDITURE

- 1. SGM file No: SGM/1825/2021-2022 dated 07/01/2022.
- 2. Title of the Lead college Project: "Synthesis and Characterization of Co₃O₄ thin film by hydrothermal route for supercapacitor application".
- 3. Name of the Project Advisor: Dr. Dhanaji S. Dalavi, Physics.
- 4. Number of students participated in the project: 04
- 5. Duration of the Activity: July 2021 to May 2022.

Sanction No. and	Grant	Details of expenditure	e incurred	Amount
date	sanctioned	item wise		
SGM/1825/2021-	10,000/-	Shri. Samarth	Chemicals	8644.00
2022 dated		Trading Company		
07/01/2022		Anushka Stationers	Stationary	440.00
Advance cheque		Shivshakti Light	Pencil	220.00
No. 138027 dated		House	Cells and	
17/01/2022.			wire cutter	
		Rajan Rubber stamp	Stationary	450.00
		and office stationers		
		Uma Xerox	Stationery,	355.00
			printing	
			and	
			binding	
i gA_grain		Total	Expenditure	10, 109.00
		Gra	ant Received	10,000.00

It is certified that the sanctioned amount of Rs. 10,000/- (Rupees Ten thousand only) out of the total grant of Rs. 10,000/- (Rupees Ten Thousand) has been received for the project work to the Department of Physics, Krishna Mahavidyalaya, Rethare (Bk) by the Sadguru Gadage Maharaj College, Karad, its Letter No. SGM/1825/2021-2022, cheque No. No. 138027 dated 17/01/2022 has been utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the Shivaji University, Kolhapur.

Project Advisor

C

Dr. Dhanaji S. Dalavi

Dr. Dhanaji S. Dalavi Assistant Professor (Physics) Krishna Mahavidyalaya, Rethare (Bk.), Tal. Karad

1

Krishna Mahavidyalaya, Rethare Bk. Tel. Karad : 415 108 (MS)

UTILIZATION CERTIFICATE

Certified that the Rs.10,000/- (Ten Thousand Only) has been sanctioned by the Shivaji University, Kolhapur to couduct the lead college Project entitled **"Synthesis and Characterization of Co₃O₄ thin film by hydrothermal route for supercapacitor application**". on 2021-2022 has been incurred by the observing scrupulously all the rules and as per rates prescribed by the Shivaji University, Kolhapur.

The unspent balance of Rs. **Nil** is refunded to the Shivaji University, Kolhapur wide challan dated------ in the bank ------ in A/C No-----receipt No.-----dated------

The excess expenditure of Rs. Nil over the advance is receivable from Shivaji University, Kolhapur.

The expenditure incurred of **Rs. 10, 109** out of which **Rs. 10,000/-** has been received wide cheque No. No. 138027 dated 17/01/2022and remaining Rs.**109/-** is receivable from Shivaji University, Kolhapur.

The penal interest of Rs. **Nil** is credited to the University, vide receipt No.-----dated------ under the budget head A.4.R.2.

Certified that the original vouchers, bills and stamped receipt for the above mentioned of A/C are retained in this office and will be made available as and when required.

Place: Shivnagar

6

Date: 30/03/2022

, Krishna Mahavidyalaya, Rethare Bk, Tal. Karad : 415 108 (MS)

Date: 20/01 / 2022___

The Principal KRISHNA MAHAVIDYALYA, RETHARE BK.

Subject : Request to pass the bills for payment.

Sir,

Please find herewith the following bills for the payment while passing the bills I have checked the price of each item and is according to the price quoted by the party.

Sr. No.	Name of the Firm	Bill No. / Date	Amount	Deductions if any	Net Paid
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		10/02/2022			
	company	1-1-1			
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		0 1			8-64-4
	Thanking You,	with	Yo	ours faithfully,	·
Lił	o / Lab / Assistant	N		Head	Department
		ΜΑΗΑνίργαι ν	A, RETHARE I		Department
			A, NEI NAKE I	JR.	

ľò,

Dr. Dhanaji S. Dalavi, Assistant Professor Department of Physics, Krishna Mahavidyalaya, Rethare (Bk). Date: 20/0**2**/2022.

To, The Principal, Krishna Mahavidyalaya, Rethare (Bk) Shivnagar-415108

Subject: Submission of Bills toward purchase of chemicals under lead college Activity research project.

Respected Sir,

With reference to above mentioned subject, we have purchased chemicals and glassware through Research Promotion activity for students of the affiliated college initiated by Shivaji University, Kolhapur for the research project entitled "Synthesis and Characterization of Co₃O₄ thin film by hydrothermal route for supercapacitor application".

Herewith I kindly request you to issue check of Rs. 8,644/- in favour of **Shri**. **Samarth Trading Company, Islampur** toward the purchase of chemicals.

Thanking You

Yours Faithfully

Project Advisor Research Promotion Activity

Dr. Dhanaji S. Dalavi Assistant Professor (Physics) Krlshna Mahavidyalaya, Rethare (Bk.), Tal. Karad

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Invoice Reverse	e No.:- CO/0147/2021-22 (Date:- 10 / 02 / 2022 e Charge (Y/N):- Maharashtra	Physics PO No Date :	o.:-	ot.)		Code	27	Transpo Vehicle n	ort Mo umbei upply	ode:-		priginal Buye 2	er's Copy
	:- The Principal, Krishna Mah s :- Rethare BK	o Party avidyal	aya,	Shivı	nagar			Name:- Address:-		Ship	to Pa	rty	
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5. No.	Product Description	Make	GST	Qty	Rate	Amount	Discount	Taxable Value	St. Jacobi	CGST		SGST	Totai
									Rate	Amount	Rate	Amount	
1221	drogen Peroxide (30% W/W) ution 500 ml.	Loba	18%	1	400.00	400.00	40%	240.00	9	21.60	9	21.60	283.20
	Foil (Nickel Foil) 250 gm.	Loba	18%	1	4300.00	4300.00	40%	2580.00	9	232.20	9	232.20	3044.40
3 Cot	balt Nitrate (Co (No3) 2)	Loba	18%	1	1000.00	1000.00	40%	600.00	9	54.00	9	54.00	708.0
He	xahydrate 100 gm.	LODA								14.04	9	14.04	184.08
	monia Solution 500 ml.	Loba	18%		260.00	260.00	40%	156.00		18.90	9	18.90	247.80
1.07	dium Nitrate (NaNO3) 500 gm.	Loba	18%		350.00	350.00	40%	210.00		12.96	9	12.96	169.9
7 Sod	assium Chloride (KCL) 500 gm. lium Sulphate (Na2SO4) Anhydrous	Loba Loba	18% 18%	_1 _1	240.00	240.00 230.00	40%	144.00 138.00		12.42	9	12.42	162.84
8 Cob) gm. balt (II) Chloride Hexahydrate (Loba	18%	1	1100.00	1100.00	40%	660.00	9	59.40	9	59.40	778.80
•	L26H2O) 100 gm. lic Acid 500 gm.	Loba	18%	1	390.00	390.00	40%	234.00	- 9	21.06	9	21.06	276.12
	line (C6H5NH2) 500 ml.	Loba	18%	1	750.00	750.00	40%	450.00	9	40.50	9	40.50	531.00
	olene (C7H5FO) 500 ml.	Qualigen	18%	1	339.00	339.00	40%	203.40	9	18.31	9	18.31	240.02
	ble Monomer 500 ml.		18%	1	1425.00	1425.00	40%	855.00	9	76.95	9	76.95	1008.90
	ble Monomer 25 gm.		18%	1	1425.00	1425.00	40%	855.00	9	76.95	9	76.95	1008.9
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Shri Samarth Trading Company	
Peth-Sangli Road, Opp. Ganesh Servicing Center Shripadnagar, ISLAMPUR. Pin 415 409, Tal. Walwa, Dist. Sangli. Tel (02342) 225394, 225773 Mob. 9822256373 E-mail : samarth.trading@yahoo.com	ET CA
GSTIN : 27AMVPP2791E1ZG	
Date : / / 20 Receipt No. : 233	0.0
Received with thanks from M/s. The principal,	
krishna mahavidyalaya. Rethare BK	,
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By Cash / D.D. / Cheque No Date :	/ /20
In Part / Full / Advance Payment of Our Bill No. CD/0147/2021-22 Date: 10	10212022
For Shri Samarth Trac	ling Company
Rs. 8644/-	
Subject to realisation of cheque.	r
V NORA WINDA	

Dr. Dhanaji S. Dalavi, Assistant Professor Department of Physics, Krishna Mahavidyalaya, Rethare (Bk) Date: **\$6**/01/2022.

To, Principal Krishna Mahavidyalaya, Rethare BK

Subject: Order for purchase of chemicals under SUK lead college activity proposal. Reference: Quotation No.S.S.T.C/0263/2021-2022 dated 03/01/2022.

Respected Sir,

With reference to above mentioned subject, we would like to purchase chemicals of Rs. 8700/- through Research Promotion activity for students of the affiliated college initiated by Shivaji University, Kolhapur for the research project entitled **"Synthesis and Characterization of Co₃O₄ thin film by hydrothermal route for supercapacitor application".**

Herewith I kindly request you to allow us to place order as per the lowest quotation. List of chemicals and glassware with prize is attached for your ready reference.

Thanking You

DII 121

Yours Faithfully

Project Advisor Research Promotion Activity Dr. Dhanaji S. Dalavi Assistant Professor (Physics) Krishna Mahavidyalaya, Rethare (Bk.), Tal. Karad

Dr. Dhanaji S. Dalavi, Assistant Professor Department of Physics, Krishna Mahavidyalaya, Rethare (Bk) Date: **QS**/01/2022

To, Shri. Samarth Trading Company, Islampur.

Subject: Order for purchase of chemicals under SUK lead college activity proposal. Reference: With reference to your Quotation No.S.S.T.C/0263/2021-2022 dated 03/01/2022.

Respected Sir,

With reference to above mentioned subject, herewith I am sending order for the purchase of purchase chemicals of Rs. 8700/- through Research Promotion activity for students of the affiliated college initiated by Shivaji University, Kolhapur for the research project entitled **"Synthesis and Characterization of Co**₃**O**₄ **thin film by hydrothermal route for supercapacitor application".**

Herewith I kindly request you to allow provide the chemicals and glassware as per given in the quotation within 15 days from this order. List of chemicals and glassware with prize is attached for your ready reference.

Thanking You

Project Advisor Salan Research Promotion Activity. Dr. Dhanaji S. Dalavi Assistant Professor (Physics) Krishna Mahavidyalaya, Pethare (Bk.), Tal. Karad

Principal Principal Krishna Mahavidyalaya, Rethare Bk, Tal. Karad : 415 108 (MS)

Shetkari Shikshan Prasarak Mandal's Krishna Mahavidyalaya, Rethare (Bk)

Order for the purchase of Chemicals and glassware

List of Chemicals

Sr. No.	Description	Make	Unit	Rate	Req. Qty.	Total
1	Hydrogen Peroxide (30% W/W) Solution	Loba	500ml	400.00	1	400.00
2	Ni Foil (Nickel Foil)	Loba	250gm	4,300.00	1	4,300.00
3	Cobalt Nitrate (Co (No3) 2)	Loba	100gm	1,000.00	1	1,000.00
	Hexahydrate Ammonia Solution	Loba	500ml	260.00	1	260.00
4	Sodium Nitrate (NaNO3)	Loba	500gm	350.00	1	350.00
5	Potassium Chloride (KCL)	Loba	500gm	240.00	1	240.00
6 7	Sodium Sulphate (Na2SO4) Anhydrous	Loba	500gm	230.00	1	230.00
8	Cobalt (II) Chloride Hexahydrate (Loba	100gm	1,100.00	1	1,100.00
	CoCL26H2O) Oxalic Acid	Loba	500gm	390.00	1	390.00
9	Aniline (C6H5NH2)	Loba	500ml	750.00	1	750.00
10	Labolene (C7H5FO)	Qualigen	500ml	339.00	1	339.00
11		40	500ml	1,425.00	1	1,425.00
12	Pyrolle Monomer		25gm	1,425.00	1	1,425.00
13	Indole Monomer			Total A	mount =	12,209.00
				Discount	@ 40% =	4,883.60
				Discount Less	s Total =	7,325.40
				GST	@ 18% =	1,318.57
	<u>1</u>			Grand	TOTAL =	8,644.00

Dr. Dhanaji S. Dalavi, Assistant Professor Department of Physics, Krishna Mahavidyalaya, Rethare (Bk). Date: 28/03/2022.

To, The Principal, Krishna Mahavidyalaya, Rethare (Bk) Shivnagar-415108

Subject: Submission of Bills toward purchase of contingency under lead college Activity research project.

Respected Sir,

1

With reference to above mentioned subject, we have purchased contingent items of Rs.1,465/- through Research Promotion activity for students of the affiliated college initiated by Shivaji University, Kolhapur for the research project entitled **"Synthesis and Characterization of Co**₃**O**₄ **thin film by hydrothermal route for supercapacitor application".** For the purchase of said items I have paid Rs. 1,465/-

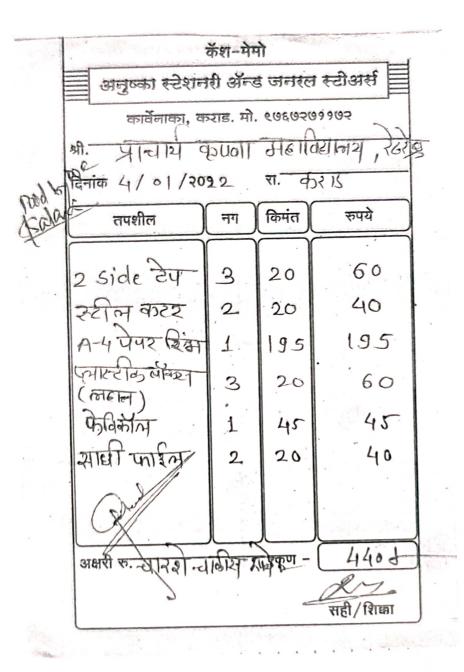
Herewith I kindly request you to issue check of Rs. 1,465/- in favour of **Dr**. **Dhanaji Suresh Dalavi** toward the purchase of contingent items.

Thanking You

Yours Faithfully

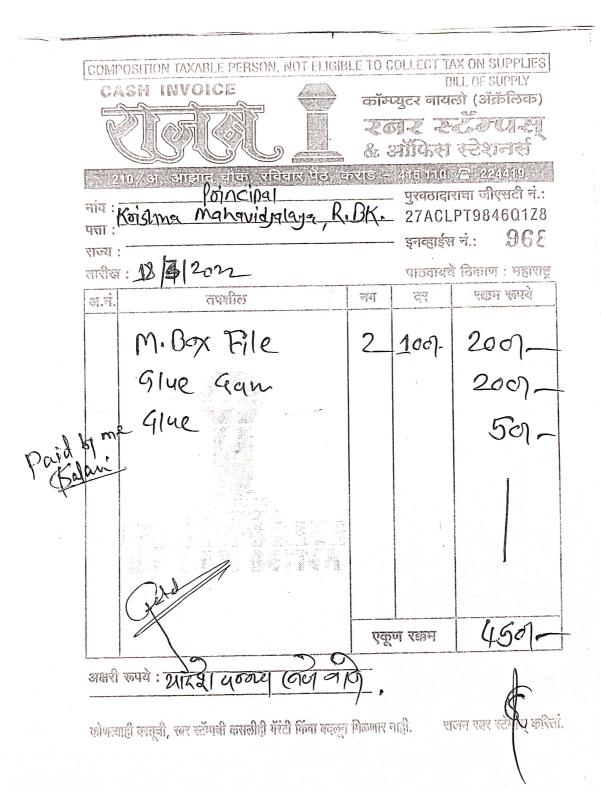
Project Advisor Research Promotion Activity





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SHIVAJI UNIVERSITY, KOLHAPUR RESEARCH SENSITIZATION SCHEME FOR COLLEGE STUDENTS

UNDER LEAD COLLEGE, ACTIVITY

SUBMISSION OF PROJECT REPORT

Title Of The Project: SYNTHESIS AND CHARACTERIZATION OF CO₃O₄ THIN FILM BY HYDROTHERMAL ROUTE FOR SUPERCAPACITOR APPLICATION.

BY

MR. PARDESHI SIDDHARTH RAHUL, MISS. PATOLE PRAJAKTA BALASO, MISS. PATOLE KOMAL POPAT, MR. ZELE RITESH DATTATRAYA

UNDER THE GUIDANCE OF DR. DHANAJI S. DALAVI ASSISTANT PROFESSOR, KRISHNA MAHAVIDYALAYA, RETHARE, BK

ACADEMIC YEAR: 2021-2022

DECLARATION

We hereby declare that the project report entitled **"Synthesis and Characterization of Co₃O₄ thin film by hydrothermal route for supercapacitor application"** submitted by us has been completed and written by us, has not previously formed and published in any other University in India or any other country or examining body to the best of our knowledge.

Place: Shivnagar

Date:

Name of Project Students

MR. PARDESHI SIDDHARTH RAHUL, Definition
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CERTIFICATE

This is to certify that thesis entitled **"Synthesis and Characterization of Co**₃**O**₄ **thin film by hydrothermal route for supercapacitor application"** which is being submitted herewith is the result of original research work completed by Mr. Pardeshi Siddharth Rahul, Miss. Patole Prajakta Balaso, Miss. Patole Komal Popat and Zele Ritesh Dattatraya under my supervision and guidance and to the best of my knowledge and belief, the work embodied in this project work has not formed earlier.

Place: Shivnagar

Date: 20/04/2022

Project Advisor

(Dr. Dhanaji S. Dalavi)

1. Introduction:

Over the last few decades, to fulfil the growing energy demands and to reduce environmental pollution, energy harvesting using renewable energy sources, their proper conversion, and advanced storage techniques have become a leading area of research and are being extensively studied. Global warming is receiving worldwide attention, and means to improve its harmful consequences are of the greatest urgency. Major changes in energy technology will be necessary, which will impact the global economy. Renewable energy sources such as wind, solar, etc are irregular, and their availability depends on various situations like time, weather, and geographical location [1]. To avail the energy as per user's demand even though the sources are absent, it's become more vital to convert and store energy effectively. Therefore, over the last few decades, to fulfill the growing energy demands and to reduce environmental pollution, energy harvesting using renewable energy sources, their proper conversion, and advanced storage techniques have become a leading area of research and are being extensively studied [2] [3].

Various energy storage devices like fuel cells, batteries, capacitors, and electrochemical capacitors (ECs) or supercapacitors (SCs) are widely used to store energy. Supercapacitors with high specific power (>10 kW/kg) and low internal resistance are superior to a battery as being able to store and deliver energy at relatively high rates. They are widely used in many devices such as mobile phones, digital cameras, and solar cell power storage due to their features like long cycling stability (>10⁵ cycles), fast charge-discharge rate, and easy fabrication with low maintenance [4]. All of the above features of supercapacitors enable them to provide high power in areas where current battery technology fails. But low energy density, despite all these features, greatly affects its commercial use. Transition metal oxides (TMOs), their hydroxides, conducting polymers (CPs), carbon, and their derivatives have been used as active materials for energy storage applications.

Depending upon the charge storage mechanism, supercapacitors are classified into three types electrochemical double-layer capacitors (EDLC), pseudocapacitors, hybrid capacitors/lithium-ion capacitors. In EDLC charge is stored and electrostatically, in which accumulation and separation of charge take place at the electrode and electrolyte interface (also known as Helmholtz's double layer). Carbon and its derivatives like graphene, activated carbon etc have been widely used as EDLC electrodes. In pseudocapacitor, the charge is stored with faradaic processes in the electrode, with reversible adsorption, redox reactions (in TMOs), and reversible electrochemical doping-dedoping process (in CPs) in the electrodes as per chosen electrode material. Transition-metal oxides (TMOs) like WO₃, Co₃O₄, NiO, V₂O₅, IrO₂, RuO₂, Fe₃O₄, MnO₂ etc, transition metal sulphides and conducting polymers (CPs) such as Polyaniline (PANI), Polypyrrole (PPy), poly (3,4-ethylenedioxythiophene) (PEDOT), polyacetylene, poly (4-styrene sulfonate) (PSS), poly-phenylene-vinylene (PPV), polythiophene, polyvinyl alcohol (PVA) etc. are widely used in pseudocapacitor.

Among TMOs, the platinum group metal (PGM) oxides like Iridium oxide (IrO₂)), rhodium oxide (RhO₂), and ruthenium oxide (RuO₂), are the best electrode materials for electrochemical capacitor application. Many researchers studied these materials, as they possess high specific energy and specific capacitance, but much costly for their commercial use. Low-cost cobalt oxide (Co_3O_4) is considered a promising material, which has been studied most intensively due to its good rate capability, excellent redox activity, environmental friendliness, high surface area, and high theoretical specific capacitance (SC) of approximately 3560 F/g (theoretical capacity 890 mAh/g). Co_3O_4 is p-type antiferromagnetic oxide semiconductor, with three polymorphs such as monoxide or cobaltous oxide (CoO), cobaltic oxide (Co_2O3), and cobaltosic oxide or cobalt cobaltite (Co_3O_4), having highest Curie temperature (Tc =1396K). Different strategies are being used to enable devices to raise their specific

power without compromising its specific energy, and as well as better rate capability, fast charging-discharging route, and high specific capacitances.

Literature survey reveals that the structure of synthesized electrode material plays a vital role in energy storage performance in order to enhance the specific energy and specific power performance of the material. In this framework, in the past few years, reports showed that morphological studies resulted in various nanostructures of Co_3O_4 such as nanoparticles [5], urchin-like nanostructure[6], nanofibers [7], needle-like [8], nanoflakes [9], quantum dots (QDs) [10], nanocubes[11] etc have been extensively investigated. However, hydrothermal method has many advantages such as it has ability to create crystalline phases of the material that are not stable at their melting point and the materials that have a high vapor pressure near their melting points can be grown by the hydrothermal method. In the hydrothermal method, the morphology of synthesized material can be controlled easily with the help of suitable pressure and temperature depending on the main ingredients of the precursor solution. The concentration of precursor and reaction time also has a great impact on the morphology.

The flower like porous arrangement of nanowires in Co_3O_4 electrode (diameter 50 nm) synthesized using hydrothermal route, assist the easy charge transport and distribution of electrolyte in bulk of electrode, resulted into SC of 493 F/g[12]. Yang et al. designed uniform ultrathin Co_3O_4 nanosheet arrays via two-step hydrothermal reaction showed a SC of 1782 F/g at current density 5 mA/cm². Additionally, it showed long-term stability by retaining capacitance greater than 90 % after 2000 cycles [13]. In 2015, Feng et al. reported large scale ultrathin Co_3O_4 nanofilms without any organic additive by facile one-step hydrothermal route with the support of cobalt-ammonia complexes nitrate. The electrode showed large SC 1400 F/g at current density of 1A/g [14]. Hierarchical hollow Co_3O_4 nanotubes by template-hydrothermal reaction with guanidine hydrochloride as precipitant and activated carbon as hard template have been reported by Yao et al in 2015. The

results show that 1-D hierarchical hollow nanotubes of Co_3O_4 has higher SC (1006 F/g at 1A/g) compared to Co_3O_4 prepared with guanidine hydrochloride without hard template (608 F/g at 1 A/g) [15].

Researchers are using hydrothermal route followed by high temperature heating to synthesize Co_3O_4 nanostructures. However, Feng and co-workers synthesized hollow Co_3O_4 microspheres at the low temperature (120 °C) hydrothermal method without subsequent heating. The electrochemical studies provided ultrahigh SC of 1227 F/g at 1A/g current density for synthesized hollow Co_3O_4 microspheres[16]. The addition of ions (such as Na⁺, Cl⁻, NO₃⁻, CO_3^{2-} , K⁺ etc) in the precursor and distinct hydrolysis agent has an influence on the morphology of the precursor. The hydrolysis, electronegativity, and ionization of additive ions has an effect on the growth of the precursor. The 3-D hierarchical flower-like Co_3O_4 assembled with an ions-assisted hydrothermal method with NaCl additive and hexamethylenetetramine (HMT) as hydrolysis agent retains 96.07 % after 10000 cycles at 5 A/g current density[17]. Recently, Paliwal and co-workers synthesized sedge like Co_3O_4 nanoarrays with SC of 2510 F/g at 4 A/g current density in 6M KOH [18].

Different strategies are being used to enable devices to raise their specific power without compromising its specific energy, and as well as better rate capability, fast charging-discharging route, and high specific capacitances. To solve the problems mentioned above, herein, we proposed Co_3O_4 based thin film by a very facile and simple double hydrothermal technique for the synthesis as the electrode material for energy storage device application.

2. Synthesis of Nanostructured Co₃O₄ thin film:

The nanostructured Co_3O_4 thin film was synthesized from a precursor solution containing $Co(NO_3)_{2.6H_2O}$ (1 mmol), NH_4F (4 mmol), $CO(NH_2)_2$ (5 mmol) in 16 ml distilled water (DW). Solution was magnetically stirred for 15 min. Ni foam was

cleaned by mixture of acetone, ethanol and DI water with assistance of ultrasonication for several minutes. Afterwards, cleaned Ni foam put against the wall of a Teflon lined stainless steel autoclave and the resulting solution is transferred into it. The autoclave was maintained at 100°C for 4 h. For effective mass loading of active material, we have adopted second hydrothermal reaction. Typically, synthesized structure was kept in a solution containing $Co(NO_3)_2.6H_2O$, $CO(NH_2)_2$ and DW in a autoclave for at 120°C for 2 (or for 6 hours). Then, the film was taken out from autoclave and washed several times with DW and acetone to remove impurities. Finally, the films were annealed at 250°C for 3 hours. Here films developed films at 2 hours and 6 hours were denoted as $Co_3O_{4@}2$ and $Co_3O_{4@}6$ respectively.

3. Characterization:

The surface morphology and stucuture of the samples were examined by scanning electron microscopy (SEM; Model JEOL-JSM-6360, Japan, operated at 20 kV) with a thin layer of gold sputter coated prior to analyses. All the electrochemical measurements were performed in an electrolyte (2 M KOH) in a conventional three-electrode arrangement comprising platinum wire as the counter electrode and Ag/AgCl serving as the reference electrode using electrochemical quartz crystal measurements (model-CHI-400A) made by CH Instruments, USA. The scan rates of CV were in the range of 10-200 mV s⁻¹. EIS was studied at 100000 Hz to 0.1 Hz at 10 mV applied potential with respect to reference electrode.

4. Results and Discussion:

4.1 Morphological Study:

Fig. 1 (a and b) shows SEM images of nanostructured Co_3O_4 thin film. Fig. 1 (a) revealed non-uniform nanoflowers having sharp spikes microstructures with an average size of 10-12 µm in diameter has resulted of as-developed nanostructures

 $Co_3O_4@6$ film. Fig. 1 (b) shows the morphology as-developed nanostructures $Co_3O_4@2$ film, revealed non-uniform sharp spikes comprising nanoflowers with thinner sharp spikes with an average size of 5 µm. The morphology have greatly influenced on electrochemical properties. The as-synthesized films display a highly porous structure that facilitates a large surface area surface which is beneficial for good electrochemical performance.

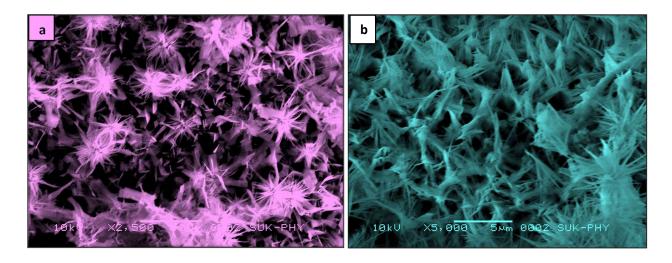


Figure.1 SEM images of as-synthesized (a) $Co_3O_4@6$ and (b) $Co_3O_4@2$.

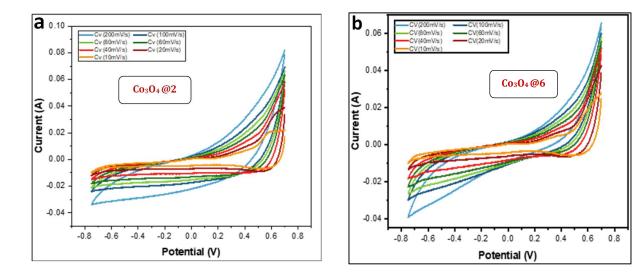
4.2 Cyclic voltammograms (CV) :

Cyclic voltammogram (CV) curves of Co_3O_4 nanoflower electrode is shown in Figure 2. CV curve of as-synthesized $Co_3O_4@2$ (figure 2a) and $Co_3O_4@6$ (figure 2b) at various scan rates 200, 100, 80, 60, 40, 20, 10 mV s⁻¹ within a potential range of 0.7 V to -0.75 V (vs Ag/AgCl) in 2 M KOH aqueous electrolyte were performed is shown in figure 2a and 2b. Figure 2c shows the CV of $Co_3O_4@6$ and $Co_3O_4@2$ at 10mV s⁻¹ scan rate. The CV profiles strongly influence with the morphology and the surface properties. The non-ideally rectangular shape of the CV curves demonstrate that the capacitive behavior is mainly characteristic of Faradic pseduocapacitance process originating from reversible redox reaction rather than electric double layer capacitance. Hence, a typical pseduocapacitance

behavior is observed in the CV curves due to Faradaic redox reaction [7]. The areal capacitance (C_a , F cm⁻²) values at different scan rates (v, mV s⁻¹) in the CV measurement are calculated using the following equation

$$C_a = \frac{\int I \, dV}{v. A. V}$$

V (in volt) is the applied potential window and A (in cm⁻²) is the area of the active electrode material. As the scan rate increase from the 10 to 200 mV s⁻¹, areal capacitance of both the as-synthesized nanostructures decrease. Both the films displayed highest areal capacitance value at 10 mV s⁻¹. The calculated areal capacitance at 10 mVs⁻¹ for Co₃O₄@6 and Co₃O₄@2 are found to be 324 and 362 mF cm⁻² respectively. The increase in areal capacitance can be explained by the more effective insertion/extraction of ions into the Co₃O₄@2 film based on larger diffusivity at low reaction temperature during second hydrothermal process and the highly porous morphology.



 $Synthesis \ and \ Characterization \ of \ Co_3O_4 \ thin \ film \ by \ hydrothermal \ route \ for \ Supercapacitor \ application$

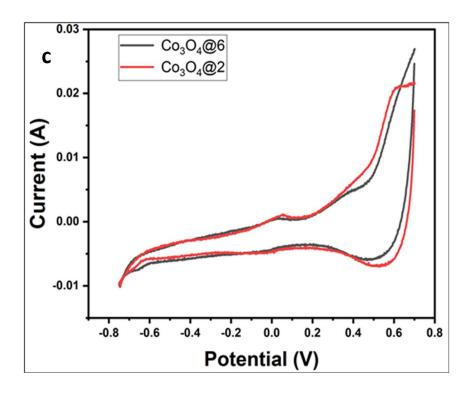


Figure 2 Cyclic voltammograms curve recorded at various scan rates within a potential range of 0.7 V to -0.7 V (vs Ag/AgCl) in 2 M KOH aqueous electrolyte of assynthesized (a) $Co_3O_4@2$ (b) $Co_3O_4@6$ (c) CV for both synthesized thin films at 10mV/s scan rate.

4.3 Galvanostatic charge-discharge (GCD) measurement :

The GCD curves of for $Co_3O_4@2$ (in figure 3a) and $Co_3O_4@6$ (in figure 3b) were studied within a potential range of 0.7 V to -0.7 V (vs Ag/AgCl) at 5, 7.5, 10, & 15 mA cm⁻² current densities, using a three-electrode cell in 2 M KOH aqueous electrolyte. Variation in capacity as a function of current density displayed the high areal capacitance at low current density. Discharge curve of $Co_3O_4@2$ and $Co_3O_4@6$ shows a significant deviation from a straight line, indicating that the capacity mainly comes from the faradaic redox reactions.

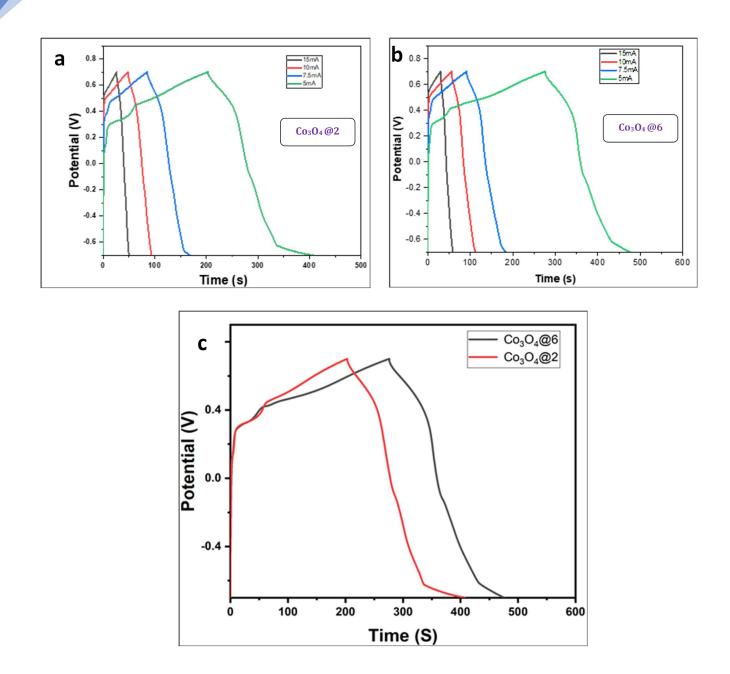


Figure 3. GCD curve recorded at within a potential range of 0.7 V to -0.7 V (vs Ag/AgCl) are performed at 5, 7.5, 10, & 15 mA cm⁻² current densities using a threeelectrode cell in 2 M KOH aqueous electrolyte of as-synthesized (a) $Co_3O_4@2$ (b) $Co_3O_4@6$ (c) GCD for both synthesized thin films at 5 mA cm⁻² current density.

4.4 ELECTROCHEMICAL IMPEDANCE MEASUREMENTS (EIS) :

EIS was studied for Co_3O_4 nanoflower electrode in the frequency range of 100000 Hz to 0.1 Hz at 10 mV applied potential with respect to reference electrode is shown in Figure 4. The diameter of the semicircle in EIS spectrum represent the electron transfer resistance (R_{ct}), which controls the electron transfer kinetics of the redox reaction at the electrode interface. Thus, both the $Co_3O_4@6$ and $Co_3O_4@2$ exhibited high electrochemical performance with low R_{ct}. The curve of $Co_3O_4@2$ showed a smaller diameter of the semicircle than that of the $Co_3O_4@6$. Low internal resistance is achieved by decreasing the time of hydrothermal process, that facilitates the electron transfer kinetics and resulted in improved areal capacity.

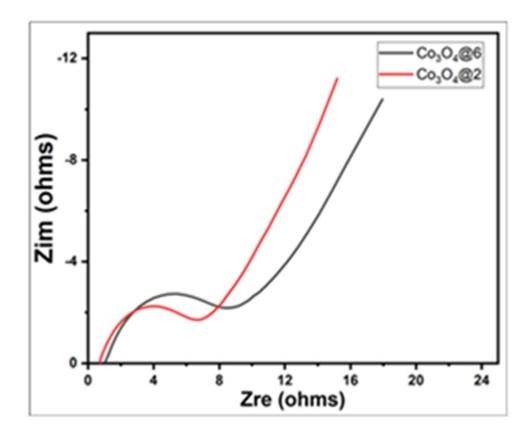


Figure 4. EIS of as synthesized Co₃O₄@2 and Co₃O₄@6 nanostructures.

Conclusions:

Nanostructured Co₃O₄ thin film has been deposited by hydrothermal method for high performance energy storage application. The effect of varying hydrothermal temperature and time on the morphology and capacitance of as-synthesized electrode was investigated. SEM study revealed non-uniform nanoflowers having sharp spikes microstructures was resulted. As the reaction time in second hydrothermal decreses, the density of spikes increases and nanoflowers spikes becomes more sharp. The obtained electrode delivered high operating voltage (about 1.4V), low resistance with a areal capacitance of 362 mF cm⁻² at a 10 mV s⁻¹ scan rate. The effect of reaction time variation in synthesis process on electrochemical performance is studied and decrease of hydrothermal time resulted in low resistance and relatively high areal capacitance of synthesized electrode. The excellent capacitance of the nanostructured Co_3O_4 is due to large surface area provided by nanostructured Co₃O₄ with porous morphology having sharp spikes. It has been evidenced that the morphology resulted in good areal capacitance which confirms that the film deposited by double hydrothermal route are well suited for high performance energy storage application.

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