

FINAL REPORT

Submitted
To



ज्ञान-विज्ञान विमुक्तये

UNIVERSITY GRANT COMMISSION
Financial Assistance
For
Minor Research Project

File No. 47-1114/14 (General/105/WRO) XII plan,
dated 30 March 2017

By

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Affiliated To Savitribai Phule Pune University, Pune

(2016-2018)



UNIVERSITY GRANTS COMMISSION

BAHADUR SHAH ZAFAR MARG

NEW DELHI – 110 002

**FINAL REPORT OF THE WORK DONE ON THE
PROJECT**

1. **TITLE OF THE PROJECT:** Synthesis of polymer supported iron and copper oxide nano-composites and their applications as catalyst in the synthesis of biologically active organic compounds containing nitrogen and sulphur
2. **NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR:** Mrs. Manisha A. Bora
B 303, Camellia, Jambhulkar Chowk, Wanowrie, Pune -410040
3. **NAME AND ADDRESS OF THE INSTITUTION:**
Bhartiya Jain Sanghtana's arts, science and Commerce College, Bakori phata,
Nagar highway, Wagholi, Pune- 412 207
4. **UGC APPROVAL LETTER NO. AND DATE:**
File No. 47-1114/14 (General/105/WRO) XII plan, dated 30 March 2017
5. **DATE OF IMPLEMENTATION:** 01/05/2016
6. **TENURE OF THE PROJECT:** 2 Years

7. **TOTAL GRANT ALLOCATED:** Rs.4, 20,000.00

8. **TOTAL GRANT RECEIVED:** Rs.3, 22,500.00

9. **FINAL EXPENDITURE:** Rs. 3, 29,921.00

10. **TITLE OF THE PROJECT:** Synthesis of polymer supported iron and copper oxide nano-composites and their applications as catalyst in the synthesis of biologically active organic compounds containing nitrogen and sulphur

11. **OBJECTIVES OF THE PROJECT:**

- To develop green ecofriendly route of synthesis of iron oxide, copper and copper oxide nanoparticles
- Synthesis of polymer nanocomposites of these nanoparticles
- Utilization of nanocomposite systems as heterogeneous catalyst in organic synthesis.
- To check the biological activity of these synthesized molecules.

12. **WHETHER OBJECTIVES WERE ACHIEVED:**

The progress has been according to the original plan of work and the objectives have by and large been achieved.

13. ACHIEVEMENTS FROM THE PROJECT:

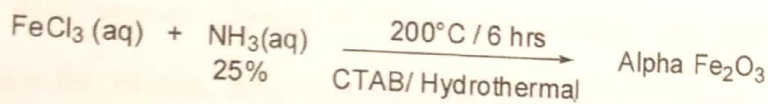
- Received Dr. Homi Bhaba Best oral presentation award at the international conference-IACR 2017 organized by S.M.Joshi college, Hadapsar.
- Received first prize at zonal level Avishkar competition under pure science teacher category organized by University Research Cell, S.P. Pune University.
- Participated and made oral presentation at 8th International Conference AMDP-2017 organized by Physics Department, S.P.Pune University.
- Participated and presented poster in the National Conference on Advanced Perspectives in Chemistry (APC-2018) organized by Chemistry Department, SPPU, Pune on 24-25 Feb. 2018.

14. SUMMARY OF THE FINDINGS:

The proposed work involves the synthesis of iron oxide and copper oxide nanoparticles, their polyaniline nanocomposites using hydrothermal methods and by using these systems as catalyst organic synthesis was carried out. The synthesized organic molecules were tested for their antioxidant activity.

Hydrothermal Synthesis of α -Fe₂O₃ nano particles:

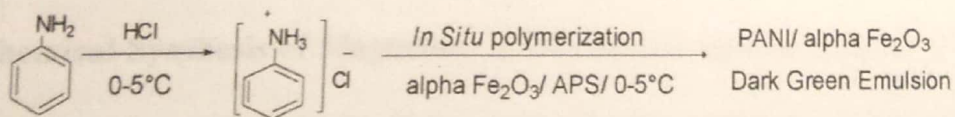
Aqueous solution of 0.01 gram of Cetyl trimethyl ammonium bromide surfactant (10ml) was taken in a beaker and to that 0.1 M solution of ferric chloride hexa hydrates (50ml) was added slowly with constant stirring. The solution was further stirred for 30 min. followed by drop wise addition of aqueous ammonia (25%) solution till the pH becomes 10. The brown red slurry obtained was heated hydrothermally at 200°C for 6 hrs.



Scheme1. Hydrothermal Synthesis of α -Fe₂O₃

Synthesis of PANI/ α -Fe₂O₃ nano composite:

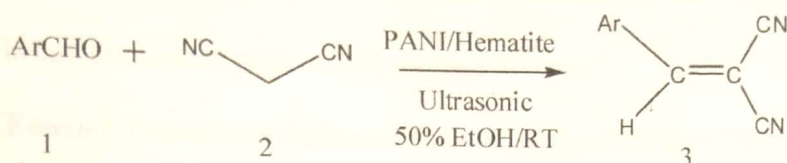
Aniline and hydrochloric acid aqueous solutions (1:1 molar ratio) were mixed and cooled in an ice bath (0-5°C) and stirred for 30 min. To this solution; α -Fe₂O₃ nanoparticles (20% W/w) were dispersed uniformly via sonication and then stirred continuously for 30 min. at 0-5°C. To this mixture 1.6 mmol of aqueous solution of APS was added slowly with constant stirring and cooling.



Scheme2. Synthesis of PANI/ α -Fe₂O₃ nanocomposite

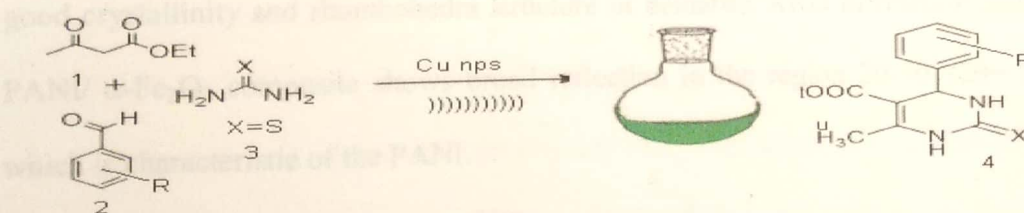
General procedure for ultrasonic synthesis of Knoevenagel condensation

A mixture of aromatic aldehyde (1mmol), malononitrile (1mmol) and PANI/ α -Fe₂O₃ (20 mg) in 50% aqueous ethanol (5 mL) was taken in a 50 mL of the reaction vessel. The reaction mixture was exposed to ultra sonication at room temperature. The completion of reaction was monitored by TLC (20% Pet ether: Ethyl acetate)



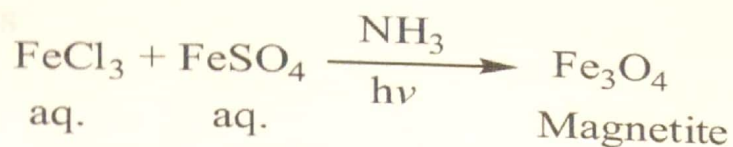
Ultrasonic Synthesis and Characterization of 3, 4-Dihydropyrimidin-2-Ones and Thi-2-Ones by using copper nanoparticles

A mixture of ethyl aceto acetate [1 mmol], Thio urea [1mmol] and methanol [5ml] and copper nanoparticles [20mg] in 25 ml conical flask. The reaction mixture kept for sonication for 30 min. After complication of 30 min add aromatic aldehyde [1mmol], then again reaction mixture kept in sonication for 30 min. The reaction complication confirmed by thin layer chromatography (TLC). The crude product obtained is recrystallized by using ethanol.



To check the antifungal activity of synthesized materials

Photochemical Synthesis of Magnetite: In this method aqueous solution of iron salts are exposed to u.v. radiations in photo reactor for the synthesis of iron oxide nanoparticles.



Structural and Morphological Analysis

UV-DRS Analysis: The diffuse reflectance (DR) UV-Vis spectrum of solid $\alpha\text{-Fe}_2\text{O}_3$ and PANI / $\alpha\text{-Fe}_2\text{O}_3$ nano composite spectrum of the $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles presents a broad absorption peak at 280 nm and 304 nm.

Fourier Transform Infrared Spectroscopy (FTIR) : The chemical composition of $\alpha\text{-Fe}_2\text{O}_3$ and PANI/ $\alpha\text{-Fe}_2\text{O}_3$ was confirmed by FTIR spectroscopy. FTIR spectra of $\alpha\text{-Fe}_2\text{O}_3$ shows characteristic peaks at 486 cm^{-1} and 585 cm^{-1} can be attributed to stretching vibration frequency of Fe-O bond in $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles. FTIR spectrums of PANI/ $\alpha\text{-Fe}_2\text{O}_3$ nanocomposite shows the characteristic bands at 1590 cm^{-1} and 1510 cm^{-1} can be attributed to quinoid and benzenoid ring stretching vibrations respectively confirming the structure of PANI

X-ray powder Diffraction (XRD)

XRD pattern of $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles shows the sharp diffraction peaks indicating good crystallinity and rhombohedra structure of hematite. XRD diffraction patterns of PANI/ $\alpha\text{-Fe}_2\text{O}_3$ composite shows broad reflection in the region $20\text{-}30^\circ$ ($2\theta = 25.33^\circ$), which is characteristic of the PANI.

Scanning Electron Microscopy (SEM):

FESEM clearly indicates spherically rippled microstructures containing numerous slightly agglomerated hematite nano spheres with uniform diameter of 30-40 nm.

16. WHETHER ANY PH.D. ENROLLED/PRODUCED OUT OF THE PROJECT:

Yes, the principal investigator has registered for PhD at Chemistry Research center, Nowrosjee Wadia College, Pune under the guidance of Dr. Vasant V. Chabukswar.

17. NO. OF PUBLICATIONS OUT OF THE PROJECT:

The paper is communicated for publication in Ultra-sonic Sonochemistry, a peer reviewed journal.

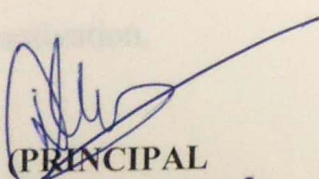
'Ultra-sonication mediated Knoevenagel synthesis by using

PANI/ α -Fe₂O₃ nano catalyst'

Manisha A. Bora^{a,b}, Vasant V. Chabukswar^b

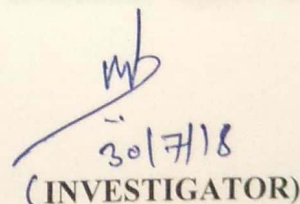
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30/7/18
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