Synthesis and characterization of transition metal nanoparticles in electronic and catalysis applications

Final report of

MINOR RESEARCH PROJECT

File No. 47-1994/11 (WRO) dated 16th February 2012

Submitted to

UNIVERSITY GRANTS COMMISSION,

WESTERN REGIONAL OFFICE, PUNE

Submitted by

Mr. S. P. Deshmukh

Principal Investigator

Department of Chemistry

D.B.F. Dayanad College of Arts & Science,

Solapur-413002 (Maharashtra)

Annexure – VIII

UNIVERSITY GRANTS COMMISSION
BAHADUR SHAH ZAFAR MARG
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:**

Mr. Deshmukh S. P., Assistant Prof., Chemistry Department, D.B.F. Dayanand College of Arts & Science, Solapur.

2.**NAME AND ADDRESS OF THE INSTITUTION**:

Dayanand Institutes,Solapur,
D.B.F. Dayanand College of Arts & Science, Solapur- 413 002.

3.**UGC APPROVAL NO. AND DATE**: 47-1994/11 (WRO) dated 16th February 2012

4**. DATE OF IMPLEMENTATION:** September 2012

5. TENURE OF THE PROJECT: September 2012 to September 2014 (Two years)

6. **TOTAL GRANT ALLOCATED:** Rs. 1,50,000/-

7. **TOTAL GRANT RECEIVED:** Rs. 1,20,000/-

8. **FINAL EXPENDITURE:** Rs. 1,27,219.80

9. **TITLE OF THE PROJECT:** Synthesis and Characterization of transition metal nanoparticles in electronic and catalysis applications

10. **OBJECTIVES OF THE PROJECT:** Separate sheet is attached

11. **WHETHER OBJECTIVES WERE ACHIEVED:** Yes
(GIVE DETAILS)

12. **ACHIEVEMENTS FROM THE PROJECT**: Separate sheet is attached

13. **SUMMARY OF THE FINDINGS:** Separate sheet is attached
(IN 500 WORDS)

14. **CONTRIBUTION TO THE SOCIETY:** Separate sheet is attached
(GIVE DETAILS)

15. WHETHER ANY PH.D. ENROLLED/PRODUCED: No
OUT OF THE PROJECT

16. NO. OF PUBLICATIONS OUT OF THE PROJECT: 01
(PLEASE ATTACH RE-PRINTS)

OBJECTIVES OF THE PROJECT:

The main objectives of the proposed research work were

1. To synthesize metal nanoparticles using chemical reduction method with selective capping as well as reducing agent to get uniform and better morphology.
2. Synthesized metal nanoparticlesto study its optical and electronic property
3. To characterize these supported metal nanoparticles by using UV-Vis, FT-IR spectroscopy and XRD, FE-SEM, EDAX, TEM analytical techniques.
4. These characterizations techniques use to study its particle size, surface area and along with optical properties for the better catalytic purpose.
5. Then supported metal nanoparticles with high surface area with smaller particles size is use to study catalysis application.

ACHIEVEMENTS FROM THE PROJECT:

1. Titania supported silver nanoparticles were synthesized via *in situ* sol–gel followed by reduction method with dextrose as reductant and sodium dodecyl sulfate as stabilizer.
2. The methodology used in this process wasimproved which confirm by the different instrumental techniques.
3. The synthesized nanoparticles were characterized by X–ray diffraction, transmission electron microscopy, Fourier transform Infra-Red spectroscopy and UV–visible measurements.
4. These characterizations confirm that silver nanoparticles incorporated with titania surfaces in which silver is more crystalline than that of titania. While particle size of silver 18-23 nm along with 7-11 nm for titania was observed by the TEM analysis. While EDAX pattern confirm chemical homogeneity of titania supported silver nanoparticles. FT-IR confirms SDS capping of silver metal to attain stability. While UV-Vis spectrophotometer showed that optical properties of titaniasupported silver nanoparticles.
5. This titania supported silver nanoparticles were used for the catalysis. The functional group conversion of 4-nitrophenol to 4-aminophenol was efficiently carried out by this heterogeneous catalyst. Kinetic studies reveal that this catalyst is selective and efficiently reusable in this chemical reaction.

SUMMERY OF THE FINDINGS

The catalyst play significant role in various chemical reactions for the functional group conversions. The silver nanoparticles can play important role in the catalytic reaction because of large surface area, better morphology. Supported silver nanoparticles were synthesized via *in situ* sol–gel followed by reduction method with dextrose as reductant and sodium dodecyl sulfate as stabilizer. This *in situ* sol gel method is more efficient for the catalyst preparation which was confirms by the characterization.

Results and discussions: The titania supported silver nanoparticle was analyzed by XRD revels that silver particles are face centered cubic with no peaks of oxide were observed, while tetragonal titania has showed anatase phase only. While silver particles deposited on titania matrix confirm by TEM analysis. TEM evidence also indicated that silver NPswith average crystallite sizewas found to be within 18 – 23 nm. Then titania NPs are non–spherical in shapes and sizes ranging in between 7 – 11 nm.The EDAX patternconfirm that the samples were composed of Ag, Ti and O with molar ratio of Ag to Ti is about 0.98 %. This study agrees well with expected composition of silver with respect to titanium centre in the sample. The UV–visible spectrum of silver suspensions was found to 412 nm. The stability of silver colloids was monitor for different periods of months. It has been seen that there is no change in the maxima of surface plasmon resonance band of silver NPs and remains identical even after six months.FTIR studiesshows that SDS capping of silver metal to attain stability.A broad band in the 400 cm^{-1} – 900 cm^{-1} region with peaks at about 470 cm^{-1}, 590 $cm^{-1}$and 645 cm^{-1} confirms the anatase TiO_2.

Thermal stability of synthesized nanoparticles was studied by Thermogravimetric with differential thermal analysis (TG–DTA) data. The weight loss is observed below 200^{o}C is due to desorption of the physically adsorbed water molecules. After 200^{o}C, the sample shows slow weight loss due to the constant decomposition of the organic framework including capping and reducing agent. It is assumed that loss in sample weight is attributed just due to the burning of capping agents. After 300^{o}C, there is no change in weight loss of sample significantly. The annealing of supported silver particles is started at room temperature and ended at 500^{o}C. The loss of water and capping agent in sample is observed with broad DTA endothermic peaks in the regions 30–175^{o}C and 200–280^{o}C, respectively

Catalytic studies: The catalytic studies of supported silver NPs were conducted through conversion of 4–NP to 4–AP in water as a solvent at room temperature. This conversion was monitored by using UV–visible spectrophotometer. Addition of the catalyst into the reaction mixture caused a gradual fading of the characteristic yellow color of 4–NP and finally yellow color of the 4–NP solution was changed to white due to the formation of 4–AP. The catalytic activity of Ag/TiO_2 NPs catalyst is much higher in comparison to results available in the literature. In our experiment, the rate of reaction is higher ($k = 1.169\ min^{-1}$) at much smaller amount of Ag/TiO_2 NPs, which is effective catalytic conversion than the reported.

Effect of $NaBH_4$ concentration: Effect of $NaBH_4$ concentrationon the catalytic reduction reaction was also studied. The content of the sodium borohydride in the reactive system greatly exceeds that of 4–NP so that the rates of the reduction are assumed to be independent of the concentration of sodium borohydride and thus the kinetics of the reduction can be treated as first–order in 4–NP concentration.

Effect of catalyst amount: It is observed that the percentage of conversion increases with increasing the amount of catalyst up to 3 mg and then decreases. Therefore, 3 mg of the catalyst was used as optimum amount for further studies.

Reusability of the catalyst: Stability and recyclability is of great importance for the practical applications of catalysts.The reusability of the catalyst were studied after the first cycle, the catalyst was centrifuged, washed with water and dried at 110^{o}C for 2 h and then which was recycled for three successive cycles without significant loss of its activity.

Conclusions: These supported silver NPs were found to be an excellent catalyst towards reduction of 4–NP to 4–AP at room temperature. This suggests the commercial exploitation of this heterogeneous catalyst for the synthesis of other organic transformations.

CONTRIBUTION TO THE SOCIETY:

The catalysis play key role for the industrial processes. The number of chemical reaction carried out in many industries in which functional group conversion of chemical compound depends upon the methodology in presence of catalyst or absence of catalyst.So that using the catalyst the methodology was improved and it became economical. Therefore catalyst can reduce the cost, energy and working efficiency of the process. In our methodology titania supported silver nanoparticles work much more efficient that other reported catalyst. It was confirmed by kinetics of the reaction, reusability of catalyst and time period required for the conversion of 4-NP to 4-AP. Hence these studieswere benefited to our society as product from the industry.

Research Paper Published:

"Titania-supported silver nanoparticles: An efficient and reusable catalyst for reduction of 4-nitrophenol" S.P. Deshmukh, R. K. Dhokale, H.M. Yadav, S.D. Delekar ***Applied Surface Science 273 (2013) 676– 683 4th march 2013.***

(ISSN No.: 0169-4332 Impact Factor: 2.735 SNIP: 1.396 SJR : 913)

V.J. Bembalkar & Co.

Chartered Accountants

Flat No. 505, 5th Floor, Deshbhakt H.arinarayan
Soni City, Soni Nagar, Near Damani Nagar,
Solapur-413001

V.J. Bembalkar

Chartered Accountant

M.Com., F.C.A.,

Phone: - 2383144, 238119

Minor Research Project

UTILISATION CERTIFICATE

It is certified that the grant of Rs.1.50.000/- (One lakh fifty thousand only) sanctioned to Mr. S. P. Deshmukh by university Grants commission file letter no. 47-1994/11 (WRO) dated 16th February 2012 towards minor research project has been utilized for which it has been sanctioned and in accordance with the terms and condition laid down by the commission.

If the result of check or audit objection some irregularity is notified at latter stage action will be taken to refund or regularize the objected amount.

Total actual expenditure incurred for this project is Rs. 127219.80 (Rupees one lack twenty seven thousand two hundred nineteen and Ps. eighty only)

Signature of Principal

V. J. BEMBALKAR & CO. A-505, Soni City, Laxmi Peth, Solapur 413 001. CHARTERED ACCOUNTANT

V. J. Bembalkar & Co.
Chartered Accountants
Proprietor V. J. Bembalkar
M. No. 034426

Signature of C.A. with seal