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It is further certified that the student has fulfilled all the requirements of Comprehensive Examination, Candidacy and SOTA for the award of Ph.D. Degree.

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I, “**Murli Dhar Mitra**”, certify that the work embodied in this thesis is my own bonafide work and carried out by me under the supervision of “**Prof. P. C. Pandey**” from “**July-2017**” to “**June-2022,**” at the **Department of Chemistry, Indian Institute of Technology, (BHU), Varanasi**. The matter embodied in this thesis has not been submitted for the award of any other degree/diploma. I declare that I have faithfully acknowledged and given credits to the research workers wherever their works have been cited in my work in this thesis. I further declare that I have not willfully copied any other's work, paragraphs, text, data, results, etc., reported in journals, books, magazines, reports dissertations, theses, etc., or available at websites and have not included them in this thesis and have not cited as my own work.

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**DEDICATED TO MY LOVING
PARENTS...**

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List of Symbols/Abbreviations

3-APTMS	:3-Aminopropyltrimethoxysilane
3-GPTMS	:3-Glycidoxypropyltrimethoxysilane
EETMS	: 2-(3,4 Epoxycyclohexyl)ethyltrimethoxysilane
PEI	: Polyethyleneimine
K₂PdCl₄	: Potassium tetrachloropalladate
AgNO₃	: Silver nitrate
AuNPs	: Gold nanoparticles
AgNPs	: Silver nanoparticles
PdNPs	: Palladium nanoparticles
BMNPs	: Bimetallic nanoparticles
TMNPs	: Trimetallic nanoparticles
NMNPs	: Noble Metal nanoparticles
EDX	: Energy-dispersive X-ray spectroscopy
PVA	: Polyvinyl alcohol
CSF	: Cerebro spinal fluid
DA	: Dopamine
DQ	: Dopamine-ortho-quinone
PVP	: Polyvinylpyrrolidone
CR	: Congo Red
Rh B	: Rhodamine B
MB	: Methylene Blue
LMB	: Leuco Methylene Blue
NaBH₄	: Sodium Borohydride
MSNPs	: Mesoporous silica nanoparticles
MSPs	: Mesoporous silica Particles

TEM	: Transmission electron microscopy
HRSEM	: High Resolution Scanning Electron Microscope
XPS	:X-ray photoelectron spectroscopy
XRD	:X-ray Diffraction
DLS	: Dynamic light scattering
SAED	: Selected area electron diffraction pattern
UV-Vis	: Ultraviolet-Visible
FL Spectroscopy	: Fluorescence Spectroscopy
BET	: Brunauer-emmett-teller surface area analysis
SiO₂	: Silica
N₂H₄	: Hydrous hydrazine
λ	: Wavelength
θ	: Angle (degree)
μM	: micro molar
nM	:nanomolar
mM	: millimolar
s	: Time (second)
QY	: Quantum yield
QS	: Quinine sulfate
R²	: Correlation coefficient
LOD	: Limit of detection

Preface

Recent decades have seen much interest in nanoscaled metals in both phases, homogenous and heterogenous, due to their potential application in science and technology. It has been demonstrated that nanoscale particles, in particular noble metal nanoparticles, have unique properties influenced by size, structure, and composition. The design of homogeneous and heterogeneous nucleation as well as controlled morphology has been an immensely challenging aspect of making noble metal nanoparticles. Based on our current progress in synthetic techniques, a number of promising approaches have been developed for producing noble metal nanoparticles with various shapes and sizes. Different methods have been explored in order to obtain metal nanoparticles, including top-down methods (which involve mechanical scraping and stabilization with surfactants) and bottom-up approaches (which involve chemical vapour infiltration, plasma discharge, polyol reduction, thermal decomposition of organometallic compounds, inter matrix synthesis (IMS), electrochemical deposition, digestive ripening, and chemical reduction with NaBH_4). Although the procedures listed above have some limitations, such as not confirming the dispersibility of nanoparticles in a wide range of solvents, Moreover, most published methods restrict their ability to disperse in a variety of solvents, thus limiting their use as a practical material. Furthermore, nanomaterials are synthesized very slowly via the previous reported method. The use of functional trialkoxysilane mediated in the synthesis of noble metal nanoparticles have been very well documented previously in our laboratory. Although such a process yielded the formation of all NMNPs, however the time course of NPs synthesis varied from 1 h to 12 h. Thus, the purpose of this thesis is to synthesize NMNPs using microwave-assisted controlled processes within minutes and use them for several catalytic applications. The microwave

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provides the optimal temperature within seconds for the synthesis of noble metal nanoparticles, leading to evaporation and solidification of the solvent, which has a low boiling point. Since previous solvents, such as ethanol, methanol, or acetone, were volatile in nature, they do not work effectively because they enable the nanoparticles to solidify. For the microwave-assisted rapid synthesis of noble metal nanoparticles, a solvent with a higher boiling point as well as a dispersible functional trialkoxysilane are necessary to prevent nanoparticles from evaporating. A microwave-assisted rapid synthesis of noble metal nanoparticles and multimetallic analogues uses either an ethylene glycol solvent or a mixture of previously used solvent and ethylene glycol. Therefore, a study has been conducted to understand what role trialkoxysilanes (3-GPTMS, 3-APTMS) play in producing nanoparticles with exceptional properties. In this thesis, noble metal nanoparticles are used in a variety of ways relevant to the discussion.

The thesis is divided into five chapters that analyze the role of trialkoxysilane in a variety of ways from various perspectives.

A brief overview of noble metal nanoparticles is presented in the first chapter, along with a discussion of the conventional routes available for synthesis. Additionally, the analytical behaviour of these noble metal nanoparticles in terms of their physical and chemical parameters has been well characterized. The introductory part also discussed the importance of multimetallic analogues over monometallic noble metal nanoparticles. Throughout this chapter, trialkoxysilane is discussed as a nanomaterial stabilizer. As well as describing the research programme objectives and workplan, this chapter also provides a brief description of the methodology.

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Chapter 2 includes a detailed overview of the rapid synthesis of monometallic (AuNPs, AgNPs, PdNPs), bimetallic (Au-Ag, Au-Pd, Ag-Pd), and trimetallic (Au-Ag-Pd) NMNPs using 3-APTMS 3-GPTMS as a mild reducing agent and stabilizing agents. In addition, we have described systematic experiments on synchronous fluorescence spectroscopy-based glucose-sensing acceleration using various NMNPs.

Chapter 3 describes the successful synthesis of fluorescent gold nanoparticles (AuNPs), involving the effective role of 3-APTMS and 3-GPTMS as reducing and stabilizing agents. The dimension of AuNPs was found to be a function of trialkoxysilane concentration, which demonstrates size-dependent catalytic activity toward the fluorometric sensing of dopamine.

Chapter 4 work plan puts emphasis on the synthesis of different size gold nanoparticles and synthesized Pd-Ni bimetallic nanocatalyst for selective sensing and decomposition of hydrous hydrazine, respectively, as well as the study of sensing and decomposition employing synchronous fluorescence spectroscopy.

Chapter 5 involves the discussion of Pd and Pd-Ni bimetallic nanocrystallite supported mesoporous silica nanoparticles made at a controlled ratio of Pd/Ni via functional trialkoxysilane mediated reduction of palladium cations are reported. The functional trialkoxysilane serves as a template for the stabilization and formation of bimetallic Pd-Ni nanocrystallite within MSNPs and their application for degradation of both cationic and anionic dyes, Rh B and Congo red, respectively.

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