

Development of hybrid catalysts for oxygen reduction reaction

Project Completion Report

SERB No: SB/FT/CS-048/2014



Submitted by

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PROJECT COMPLETION REPORT

- Notes:
1. The PCR should be in bound form.
 2. Cover page should include the title of the project, file number, names and addresses of the investigation

1. Title of the project: Development of Hybrid Catalysts for Oxygen Reduction Reaction

2. Principal Investigator(s) and Co-Investigator(s):

Dr. Pankaj Bharali

3. Implementing Institution(s) and other collaborating Institution(s):

Department of Chemical Sciences, Tezpur University,
Napaam, Assam, India

4. Date of commencement: 30/07/2015

5. Planned date of completion: 29/07/2018

6. Actual date of completion: 31/07/2018

7. Objectives as stated in the project proposal:

The main objective of this project is to develop newer shape, size and composition controlled non-platinum based hybrid catalysts for oxygen reduction reaction (ORR). Pd has been emerged as a good catalyst for the ORR and its abundance is at least fifty times more than that of Pt on earth. In this project, it is therefore aimed to tune the reactivity of Pd by modifying it with early transition metals, such as, Fe, Co, and Cu etc for ORR. To achieve this goal the following tasks will be undertaken:

- (i) To synthesize shape, size and composition controlled PdM (M = Fe, Co, and Cu) alloy nanostructures by modified surfactant assisted/surfactant-free hydrothermal and solvothermal techniques.
- (ii) To evaluate ORR activity of synthesized nanoalloys by voltammetry and other techniques.
- (iii) To investigate the impact of promoter, such as, nanostructured CeO₂ to form hybrid catalysts with PdM for ORR.
- (iv) To characterize PdM nanoalloys, promoter oxide, and corresponding hybrid PdM/CeO₂ nanocatalysts by various spectroscopic and microscopic techniques.
- (v) To investigate in depth quantitative evaluation of the electrocatalytic activities and kinetics of ORR with the help Rotating-disk electrode (RDE) measurements.
- (vi) To investigate the influence of carbon support on the performance of catalysts for ORR.

8. Deviation made from original objectives if any, while implementing the project and reasons thereof:

The project objectives were slightly deviated because of unavailability/ delay in installation of electrochemical workstation. However, the materials synthesized have been employed for related catalytic applications. The actual works which were proposed have been communicated to journals and we are expecting the articles to be accepted soon.

9. Experimental work giving full details of experimental set up, methods adopted, data collected supported by necessary table, charts, diagrams & photographs:

Methodology

Synthesis of novel catalysts

Different shape, size and composition controlled PdM/C (M = Fe, Co and Cu) alloy nanostructures and promoter oxide, CeO₂ as well as hybrid PdM/CeO₂ have been synthesized by modified solvothermal method. Effect of various solvent, metal precursor-base ratio, treatment temperatures, aging time, etc. are also investigated to design the metal/metal oxide nanostructures.

Characterization of the catalysts

A wide range of spectroscopic (FTIR, EPR, XPS/AES, ISS, CV and Raman), structural (XRD, SEM-EDX, TEM, TPR/TPO/TPD) methods are extensively utilized to characterize the synthesized catalysts systems for the study of active centres and intermediate species in the above referred catalytic reaction.

Evaluation of catalytic activity

Evaluation of various synthesized PdM alloy nanocatalysts for ORR is carried out with the help of cyclic voltammetry and other techniques. The working electrodes were fabricated by using the ink prepared by ultrasonically dispersing the catalyst powders in distilled water/Nafion solution. A known volume of the composite ink was then spread onto a mirror finished glassy carbon electrode using a micropipette tip. The solvent of the ink was evaporated at 40-50 °C, under vacuum, to obtain a well-dispersed catalyst layer on the glassy carbon electrode. For in depth quantitative evaluation of the electrocatalytic activities and kinetics w.r.t. ORR, rotation-rate dependent Rotating-disk electrode (RDE) experiments were carried out. The kinetic current density (j_k) can be calculated using the Koutecky-Levich equation.

Experimental Section

Hydrothermal synthesis of PdM (M = Co, Cu) nanoparticles:

PdM (M = Co, Cu) bimetallic nanoparticles having different composition such as Pd:M= 1:9, 2:8 and 3:7 were synthesized by modified hydrothermal method. In a typical procedure, to prepare PdCu with 1:9 ratio, 50 ml aqueous solution of 0.3834 g (100 mM) $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ and 0.04433g (100 mM) PdCl_2 was added to 50 ml NaOH solution (0.8 gm, 0.4 M) at room temperature under constant stirring. After stirring about 15 min, 3.5 ml of 85 % hydrazine hydrate was added drop wise to the mixture and stirred vigorously. After about 20 min the solution was transferred into a teflon cup (250 ml capacity) in a stainless steel autoclave. The autoclave was sealed and kept in an oven and maintained at 120 °C for 6 h. The autoclave was allowed to cool naturally (2.5 h); a black fluffy solid product was deposited on the bottom of the teflon cup which indicates the formation of PdCu bimetallic nanoparticles. The final product was washed several times with distilled water and then 50% $\text{C}_2\text{H}_5\text{OH}$ solution to remove the un-reacted NaOH and Cl^- , and finally collected by centrifugation. The product was dried in a vacuum oven. The same procedure was employed to prepare PdCo bimetallic nanoparticles.

Synthesis of CeO_2 support:

An oxalate route was employed for the synthesis of CeO_2 nanostructure. Typically, 0.48 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$ was dissolved in 50 mL water to form a clear solution which was quickly added to the 50 mL cerium salt solution with a concentration of 0.2 M under strong stirring at room temperature (about 25 °C). The precipitation occurred in several seconds. After stirring for 40 min, the precipitate obtained was aged for 24 h under static condition. The precipitate was filtered, washed with deionized water and absolute ethanol and dried at 80 °C. The samples were obtained by sintering the precursor in a muffle furnace at 450 °C for 5 h.

Synthesis of CeO_2 supported PdM (M = Co, Cu) catalyst:

CeO_2 supported PdM (M = Co, Cu) nanoparticles were also synthesized by modified hydrothermal method. In a typical procedure, to synthesize $\text{Pd}_1\text{Cu}_9/\text{CeO}_2$; 25 mL 0.01 M PdCl_2 with 0.09 M CuCl_2 were added to previously dispersed CeO_2 (0.33 g) in distilled water. The metal loading was kept as 3 wt%. 50 mL 0.8 M solution of NaOH was then added slowly with vigorous stirring at room temperature and pressure. After that 5 mL of $\text{NH}_2\text{NH}_2 \cdot \text{H}_2\text{O}$ solution was added dropwise and the whole solution was transferred to the autoclave and put in oven at 120 °C for 6 h. Then the precipitate was filtered, washed with distilled water and absolute ethanol several times to remove the unreacted NaOH and Cl^- and finally dried in a

vacuum oven.

Preparation of Carbon-supported Pd_{4-x}Cu_x Nanoparticles (Pd_{4-x}Cu_x/C NPs)

The Pd_{4-x}Cu_x/C NPs ($x = 1-3$) were prepared by the solvothermal method. In a typical procedure to prepare Pd₃Cu/C, CuCl₂·2H₂O (0.01113 g) and PdCl₂ (0.0347 g), which were used as the Cu and Pd precursor respectively, was dispersed in 50 mL of solution containing equal proportion of absolute grade ethanol and distilled water along with 0.1 g of carbon black (Vulcan XC-72R). The mixture was stirred for about 15 min followed by 5 min sonication. After sonication, 30 mL NaOH solution (0.24 g, 0.2 M) was added drop wise to the mixture followed by vigorous stirring. After 30 min, 2 mL 85% hydrazine hydrate was added dropwise to the mixture and stirred vigorously for another 25 min. After that, the solution was transferred into a teflon-lined stainless steel autoclave of 250 mL capacity. The autoclave was sealed and kept in an oven maintaining a temperature of 130 °C for 6 h. The autoclave was allowed to get cooled to room temperature. The final product obtained was washed several times with distilled water and then with 50% EtOH to remove the unreacted NaOH and Cl⁻ and finally collected by centrifugation and dried in a vacuum oven. The identical procedure was employed for the synthesis of various compositions of PdCu/C and Pd₃M/C NPs (M = Ni and Co) keeping other conditions unaltered and only varying the initial mole ratio of Pd and Cu/Ni/Co salt precursors to required compositions to achieve the final NPs.

Synthesis of Pd₃Fe_{0.5}Cu_{0.5}/C NCs

The 20wt% of Pd₃Fe_{0.5}Cu_{0.5}/C NCs was synthesized by the typical solvothermal method. To prepare the NCs, to a mixture solutions of 25 mL distilled water and 25 mL C₂H₅OH was mix, 0.197 mmol PdCl₂ (0.0350 g), 0.065 mmol FeCl₂·4H₂O (0.0132 g), 0.065 mmol CuCl₂·2H₂O (0.0112 g), and 0.1 g Vulcan XC-72 R were added sequentially at room temperature with constant stirring. The mixture solution was ultra-sonicated for 5 min and 30 mL 0.05 M (0.2 g) NaOH solution was added dropwise at room temperature under constant stirring. After ca. 20 min, 2 mL 85% hydrazine hydrate was added dropwise to the mixture and stirred vigorously for another 25 min. The solution was then transferred into a Teflon cup (250 mL capacity) in a stainless steel autoclave. The autoclave was sealed and kept in an oven at 120 °C for 6 h and allowed to cool naturally (~2.5 h); the final product was washed several times with distilled water and then 50% C₂H₅OH to remove the unreacted NaOH and Cl⁻, and finally collected by centrifugation. The product was dried in a vacuum oven at 55 °C.

Synthesis of Pd_{4-x}Fe_x/C NCs

The synthesis procedures for Pd_{4-x}Fe_x/C NCs (x = 1, 2 and 3) were similar to that of Pd₃Fe_{0.5}Cu_{0.5}/C NCs keeping other conditions unaltered and only varying the initial mole ratio of Pd and Fe salt precursors to 1:3, 1:1 and 3:1.

Synthesis of Pd/C NCs

The Pd/C NCs were also synthesized using the method similar to that for Pd₃Fe_{0.5}Cu_{0.5}/C. In this case, only PdCl₂ was employed as the metal precursors.

Physical Characterizations

The electrocatalysts were characterized by powder X-ray diffraction (pXRD) on a BRUKER AXS instrument using nickel-filtered CuK α ($\lambda = 0.15418$ nm) radiation source and a scintillation counter detector. The intensity data were collected over a 2θ range of 10-80° at a scan rate of 0.05° s⁻¹. The shape, size and distribution of the metal particles over the carbon support were obtained through transmission electron microscopy (TEM), using a Technai G² instrument equipped with a slow-scan CCD camera and at an accelerating voltage of 200 kV. The TEM forms an image by accelerating a beam of electrons that pass through the specimen. To identify the bulk composition and the relative concentrations Pd and M (M = Cu, Ni and Co) in the synthesized samples, ICP-OES measurements were performed in PerkinElmer, USA instrument (Model: Optima 2100 DV) with the software Winlab-32. Energy-dispersive X-ray spectroscopy (EDS) elemental mapping were performed on an Oxford instrument attached to the “JEOL, JSM Model 6390 LV” scanning electron microscope operated at 15 kV. In addition, we have also employed the XPS studies were performed using a Thermo K-Alpha XPS equipped with an Al K α radiation (1486.6 eV) X-ray source at a pressure < 10⁻⁷ Torr and an electron take-off angle (angle between electron emission direction and surface plane) of 90°. A survey scan was performed using pass energy of 200 eV to determine possible contaminants. The binding energies of the samples were charge-corrected with respect to the adventitious carbon (C 1s) peak at 284.6 eV.

Electrocatalytic measurements

The catalytic activities of the freshly prepared catalysts for the electrocatalytic oxygen reduction were measured following a typical procedure. In brief, glassy carbon (GC) electrode (3 mm diameter, Rotating disk electrode, RDE) was polished with 0.005- μ m Al₂O₃ powder. A typical suspension of the carbon-supported catalysts was prepared by adding 5 mg of fresh catalyst to 0.5 mL 2-propanol, deionized water and 0.5% Nafion solution,

respectively. The resultant mixture was sonicated for 30 min until it became a uniform dark ink. Then, 3 μL of the suspension was quantitatively deposited onto the surface of the polished GC electrode. The electrode coated with a thin film of the ink was dried at 35°C under N_2 atmosphere for 6 h under vacuum. The modified GC electrode was inserted into an electrochemical cell and used as the working electrode. The cell includes two additional electrodes: a KCl saturated Ag/AgCl (Metrohm) as the reference electrode and a platinum wire (Metrohm) as the counter electrode. Cyclic voltammetry (CV) was performed using a computer-controlled electrochemical analyzer (Metrohm Autolab PGSTAT204 workstation, The Netherlands) at room temperature. The CV was measured in high purity N_2 and O_2 saturated (0.1 M KOH and 0.5 M H_2SO_4) solution with scan rate 50 mV s^{-1} . RDE measurements were performed in O_2 -saturated 0.1 M KOH. Oxygen was purged for at least 30 min before measurement and continuously bubbled through the electrolyte, in order to ensure the saturation of the electrolyte with O_2 and then blanketing the solution with an O_2 atmosphere during the entire experiment and all experiments were performed at room temperature. The electrochemical active surface area (ECSA) of the catalysts was evaluated by CVs in a nitrogen-saturated 0.1 M KOH solution. For Pd-based catalysts, to evaluate the ECSA of the samples the coulombic charge for the reduction of Pd–O monolayer, formed on Pd catalysts at the forward scan, was applied. The ECSA were calculated using the following equation: $\text{ECSA} = Q/\text{SL}$, where L is the Pd loading, Q is the collected charge that calculated from the Pd–O stripping, and S is a constant of 210 $\mu\text{C cm}^{-2}$ that assumes a monolayer of Pd–O on the surface. The stability of the ORR electrode was tested using chronoamperometry. The chronoamperometric response for the ORR was obtained at -0.3 V (vs. Ag/AgCl) of in O_2 -saturated 0.1 M KOH solution at 1600 rpm. For comparison, a commercial 20 wt % Pd/C and Pt/C (Alfa Aesar) were measured under the identical experimental conditions.

Results and discussion

Characterization related to unsupported and CeO_2 supported PdM ($M = \text{Co}, \text{Cu}$) catalyst

XRD analysis: XRD profile of as-synthesized unsupported and CeO_2 supported nanoparticles are shown in Fig 1 A and B, respectively. Diffraction peak at angle of 40.1° and 43.2° corresponds to the Pd (111) (JCPDS 05-0681) and Cu (111) (JCPDS 04-0836), respectively. The diffraction peak of bimetallic CuPd nanoparticle is positioned between those of monometallic Pd and Cu nanoparticles. This confirms the formation of CuPd alloy structure

(Fig 1A). The peaks observed at diffraction angle $2\theta = 28.518, 38.37, 47.59, \text{ and } 56.20^\circ$ (Fig 1B) assigned to (111), (200), (220) and (311) reflection, respectively for fcc-CeO₂, (JCPDS 43-1002). In case of bimetallic Pd₁Cu₉/CeO₂ and Pd₂Co₈/CeO₂ where metal loading is ~3 wt% the XRD patterns predominantly shows the peaks of CeO₂ (i.e. the support) only. The intensity of the metals present is quite low or sometimes it is negligible due to their minute composition.

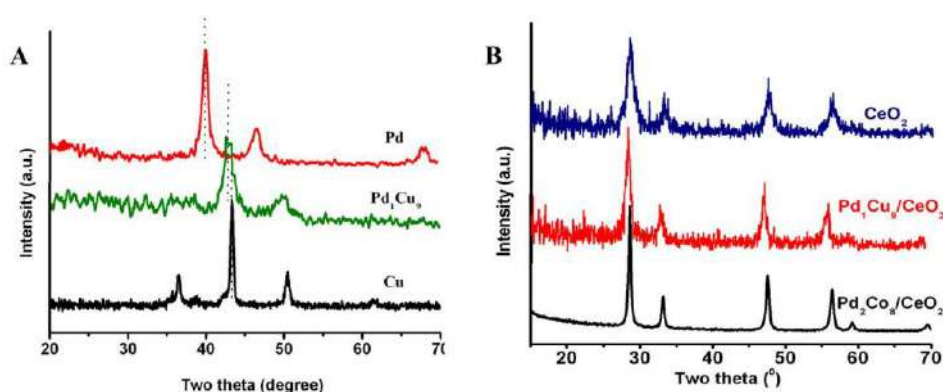


Fig 1: XRD profile of (A) unsupported Pd, Pd₁Cu₉, Cu nanoparticles and (B) CeO₂, Pd₁Cu₉/CeO₂, Pd₂Co₈/CeO₂ nanoparticles.

Raman, UV-DRS and TEM analysis of CeO₂: The Raman spectra for CeO₂ samples were obtained using Ar⁺ ion laser with a wavelength of 514 nm. The peak at 462 cm⁻¹ typically assigned to the F_{2g} symmetrical stretching mode of Ce-O bond in the fluorite type lattice. Complementary two more weak bands at 591 and 266 cm⁻¹ due to the second order transverse acoustic (2TA) mode and defect-induced (D) mode (Fig 2A). From the UV-Vis DRS analysis it is observed that CeO₂ exhibits two adsorption maxima at ~340 and 255 nm (Fig 2B) which are due to Ce³⁺←O²⁻ charge transfer (255 nm) and inter band transitions (340 nm). Representative TEM images for CeO₂ samples are shown in Fig 2C. The honeycomb like structures describes the existence of spherical aggregates of cerium oxide nanoparticles in the synthesized material.

TEM analysis of unsupported and CeO₂ supported PdCo nanoparticle:

TEM images of unsupported PdCo nanoparticles are shown in Fig 3 A. The shapes of the particles drop nearly in spherical nature and scattered in a homogenous manner. Moreover, TEM analysis of CeO₂ supported Pd₁Co₉ clearly shows the formation of PdCo bimetallic nanoparticles on the surface of face centered cubic lattice of CeO₂ (Fig 3B). The metal nanoparticles are distributed homogeneously over the surface of CeO₂ particles.

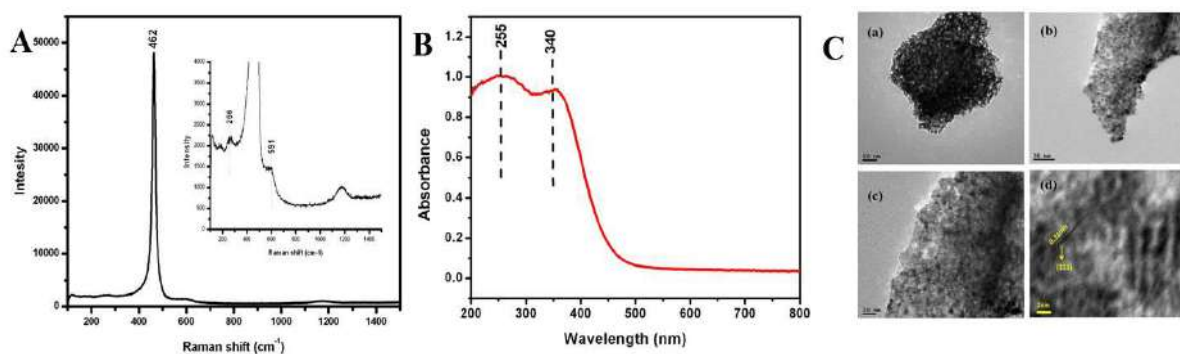


Fig 2: (A) Raman spectra, (B) UV-DRS analysis and (C) TEM images of CeO₂.

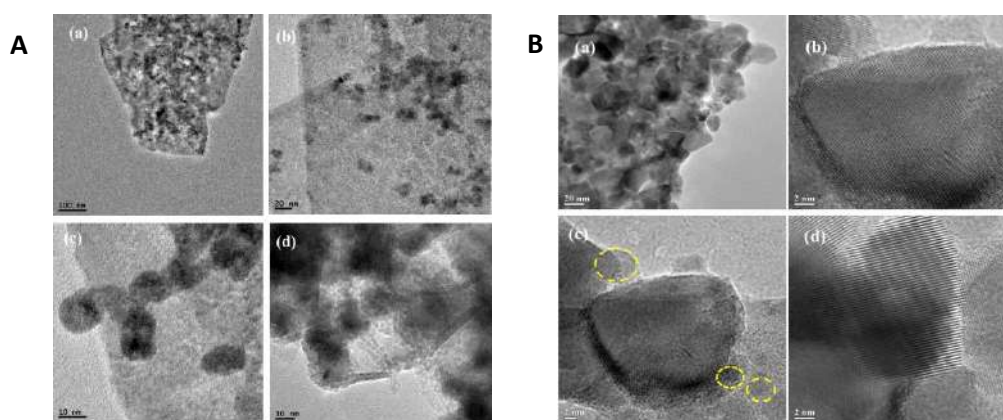


Fig 3: TEM analysis of (A) unsupported PdCo metal nanoparticles and (B) CeO₂ supported PdCo metal nanoparticles.

XPS analysis:

The catalyst surface composition of unsupported PdCu and PdCo bimetallic nanoparticles were studied by XPS. The XPS survey spectrum clearly shows appearance of peaks for C 1s, Pd 3d and Cu 2p of PdCu and C 1s, Pd 3d and Co 2p of PdCo nanoparticles as shown in Figure 4 (A) and (B), respectively. In case of Pd 3d, two major peaks formed at binding energy 335.2 and 340.3 eV are corresponding to Pd 3d_{5/2} and Pd 3d_{3/2}, respectively. Two major peaks located at the binding energy of 932.3 and 952.1 eV which are corresponding to Cu 2p_{3/2} and Cu 2p_{1/2}, respectively typically due to the zero-valent Cu (Fig 4A (c)). The representative XPS spectrum of Co 2p shows a peak with a binding energy of 780.2 eV which is corresponds to Co 2p_{3/2} as shown in Fig 4 B (c). Additionally one more peak at the binding energy of 798.2 eV is corresponds to Co 2p_{1/2} state.

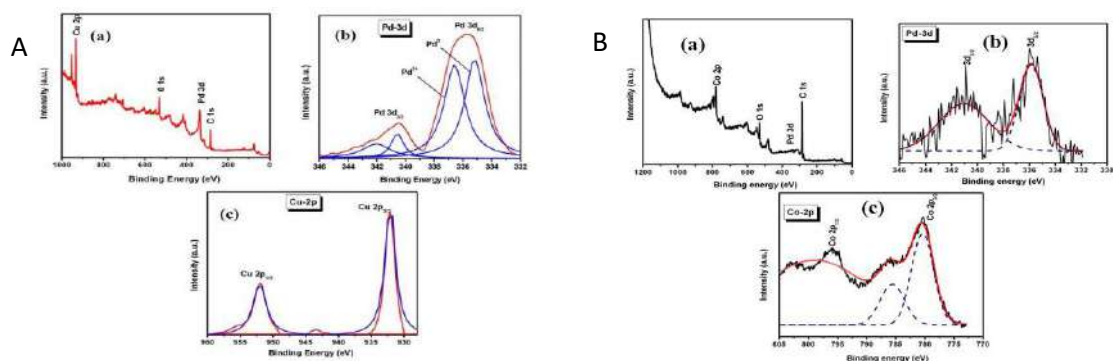


Fig 4: XPS analysis of (A) PdCu and (B) PdCo bimetallic nanoparticles.

Characterization related to Carbon-supported Pd_{4-x}Cu_x Nanoparticles (Pd_{4-x}Cu_x/C NPs)

XRD and BET surface area analysis: Figure 5(a) shows the powder XRD patterns of the as-synthesized PdCu/C NPs. The first broad characteristic diffraction peaks located at $2\theta = 24.8^\circ$ corresponds to (002) crystalline plane of carbon according to the JCPDS PDF 75-1621. The other three diffraction peaks at $2\theta = 40.2, 46.7,$ and 68.2° could be indexed to the (111), (200) and (220) crystal planes of fcc type of Pd nanoparticles (JCPDS PDF 88-2335). The diffraction peak at $2\theta = 40.1^\circ$ is slightly shifted to the right in case of Pd₁Cu₃/C i.e. with increasing the Cu content. Additionally, it is noted that the relative intensity of the peak at 40.2° is gradually increases. No other additional peaks were detected signifying the high purity and good crystallinity of the sample. Figure 5(b) represents the N₂ adsorption/desorption isotherms and BJH measurements (inset) for Pd₃Cu₁/C NPs. The isotherms represents type IV hysteresis loop of H1 type which signifies the mesoporous material. The BET surface area and the pore diameter are found to $30.1 \text{ m}^2 \text{ g}^{-1}$ and 3.22 nm respectively.

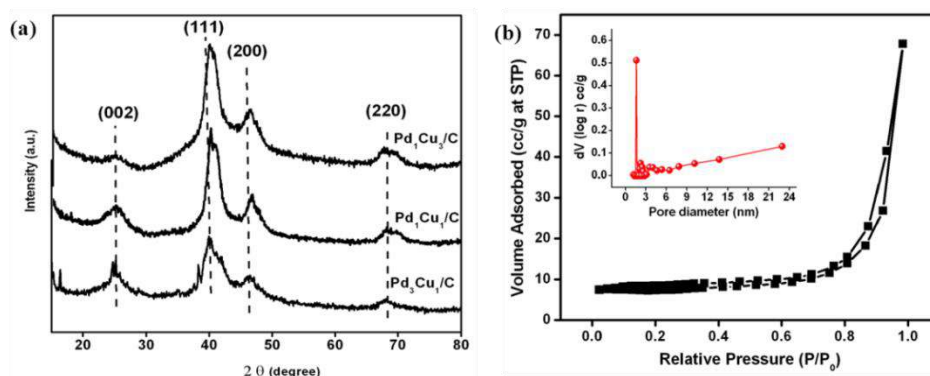


Fig 5: (a) Powder XRD pattern of as synthesized Pd₁Cu₃/C, PdCu/C and Pd₃Cu₁/C, respectively and (b) N₂ adsorption/desorption isotherms and pore size (inset) for Pd₃Cu₁/C NPs.

TEM analysis: Figure 6 (a)-(c) shows the TEM images of Pd₃Cu₁/C at different resolutions. Homogeneous dispersion of the particles on the carbon support can be demonstrated from the TEM images. The stability of the synthesized NPs is significant as they are quite uniformly distributed on the support evidenced from the respective TEM images. The size and the distribution of the particles most of the particles fall in the range of 3-5 nm (inset Fig 6a) with spherical morphology. Moreover the selected area diffraction pattern (inset Fig 6 b) represents well defined concentric rings which signify the polycrystallinity of the sample.

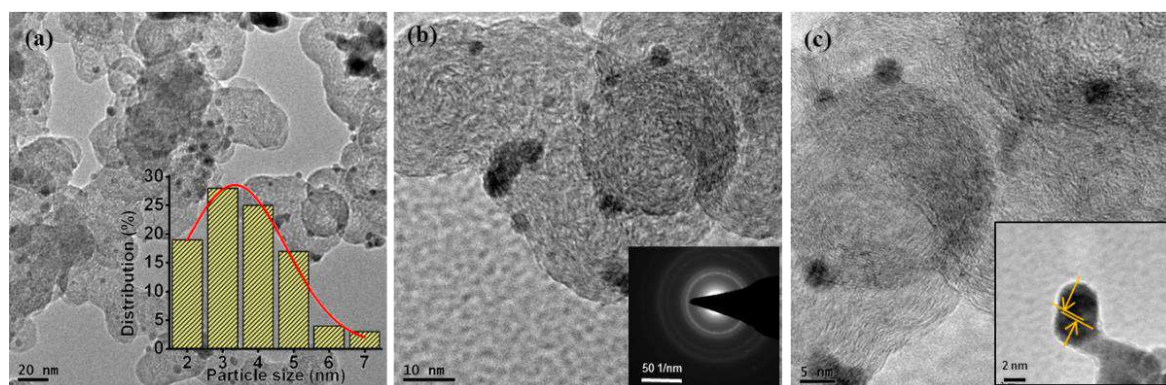


Fig 6: (a) -(c) TEM images of Pd₃Cu₁/C at different resolutions.

XPS analysis:

Figure 7 (a)-(d) represents survey spectrum, high resolution C1s, Pd 3d and Cu 2p XP spectrum respectively for Pd₃Cu₁/C. The four different components of C1s at the binding energies of 284.4, 285.4, 286.6 and 289.3 eV corresponds to C–C (sp³), C=C (sp²), C–O and O=C–H species respectively. The Pd 3d region shows predominantly two major peaks at the binding energy of 335.2 and 340.9 eV (Figure 7c) which corresponds to Pd 3d_{5/2} and Pd 3d_{3/2} species respectively. Figure 7d displays the high resolution Cu2p core-level XP spectrum. Two major peaks located at the binding energy of 932.3 and 952.1 eV correspond to Cu 2p_{3/2} and Cu 2p_{1/2}, respectively. These two peaks are typically due to the zero-valent Cu.

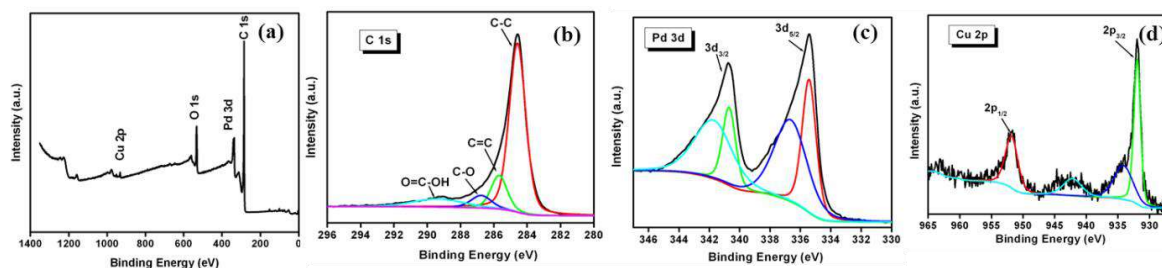


Fig 7: XPS analysis for Pd₃Cu₁/C NPs (a) the survey spectrum; and the high resolution core-level XP-spectrum of (b) C1s; (c) Pd 3d and (d) Cu 2p region, respectively.

Characterization related to Carbon-supported Pd₃Fe_{0.5}Cu_{0.5} and Pd₄₋₃Fe_x Nanoparticles

XRD and BET surface area analysis: Figure 8 (A) shows the XRD patterns of Pd₃Fe_{0.5}Cu_{0.5}/C, Pd₃Fe/C, PdFe/C, and PdFe₃/C. The diffraction peaks centred at ~40°, ~47°, and ~68° correspond to the (111), (200), and (220) reflections, respectively relative to the pure Pd (JCPDS no. 87-0641) and a broad peak centred at ~25° corresponds to the (002) reflection of a hexagonal structure in Vulcan XC-72R carbon. In the case of PdFe/C and PdFe₃/C with the increase of Fe content, minor peaks are observed in the XRD patterns at 30.1°, 35.5°, 57.2°, and 62.8° which can be assigned to Fe₃O₄ phases (JCPDS no. 19-0629). Figure 8(B) shows the N₂ adsorption-desorption isotherm of Pd₃Fe_{0.5}Cu_{0.5}/C, these carbon supported nanoalloy exhibits a high BET specific surface area of 69.8 m²/g and a large pore volume of 0.104 cc/g.

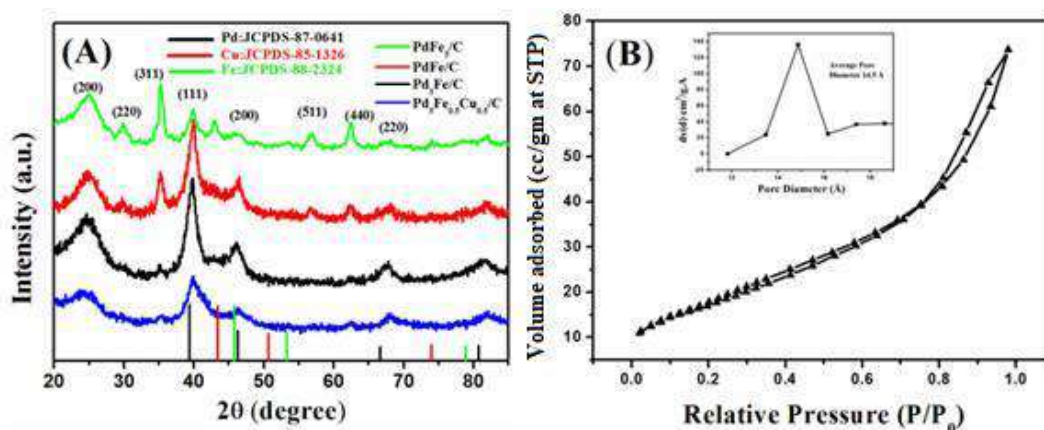


Fig 8: (A) XRD patterns of the Pd₃Fe_{0.5}Cu_{0.5}/C, Pd₃Fe/C, PdFe/C, and PdFe₃/C electrocatalysts; (B) N₂ adsorption-desorption isotherm of Pd₃Fe_{0.5}Cu_{0.5}/C. Inset the corresponding pore size distribution.

TEM analysis: From the TEM images (Figure 9A-C) it could be observed that many nearly-spherical Pd₃CuFe NPs of dark contrast are highly dispersed on the larger particles of Vulcan XC72R carbon of lighter contrast. The average particle-size of Pd₃Fe_{0.5}Cu_{0.5} NPs was estimated to be about ~ 6-7 nm (inset in Fig. 9A). The NPs exhibit various crystal defects on their surfaces, as manifested in HRTEM images (Figure 9 D,E). As shown in figure 9F, Pd₃CuFe NPs are half embedded in the carbon matrix. The concentric circles with well-defined crystalline facets in the SAED pattern (Figure 9G) infer high crystallinity of Pd₃CuFe/C NPs. The comprehensible d-spacings ~0.22 nm (Figure 9H) can be observed in the lattice fringes of a Pd₃CuFe/C NPs.

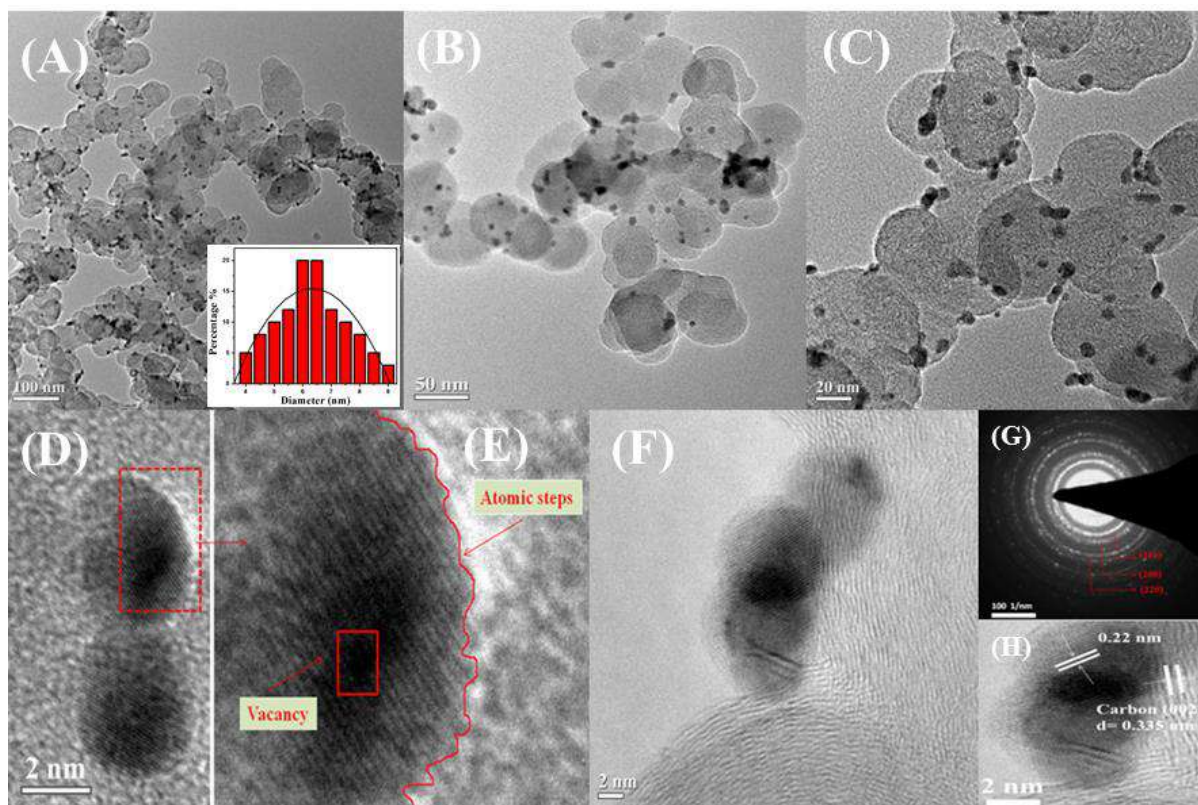


Fig 9: Typical (A) low, (B,C) medium and (D,E,F,H) high-resolution TEM images of $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$; (G) corresponding SAED pattern; (E) is magnified HRTEM image taken from (D), marked by the rectangle.

XPS analysis: Figure 10 (A-D) illustrates the XPS spectra of C 1s, Pd 3d, Fe 2p and Cu 2p regions. The C 1s core-level XPS spectrum (Fig. 10A) of $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$ is slightly asymmetric and can be deconvoluted into three different peaks viz one strong peak at 284.6 eV along with two small peaks at 285.94 and 289.82 eV, consistent to form $-\text{C}=\text{C}-$, $-\text{C}-\text{O}-$, and $\text{O}=\text{C}-\text{O}-$ bonds, respectively. The core-level Pd 3d spectra display a doublet signal with binding energies of 335.72 and 341.06 eV for Pd 3d_{5/2} and Pd 3d_{3/2}, respectively. The additional small doublets around 336.89 and 342.51 eV can be assigned to the Pd 3d_{5/2} and Pd 3d_{3/2} peaks of PdO (Fig. 10B). Fig. 10C displays the Fe core-level spectrum for the nanocatalyst. Two peaks at 711.13 and 724.96 eV are clearly visible which correspond to the Fe (2p_{3/2}) and Fe (2p_{1/2}) core-level energies for iron oxide.

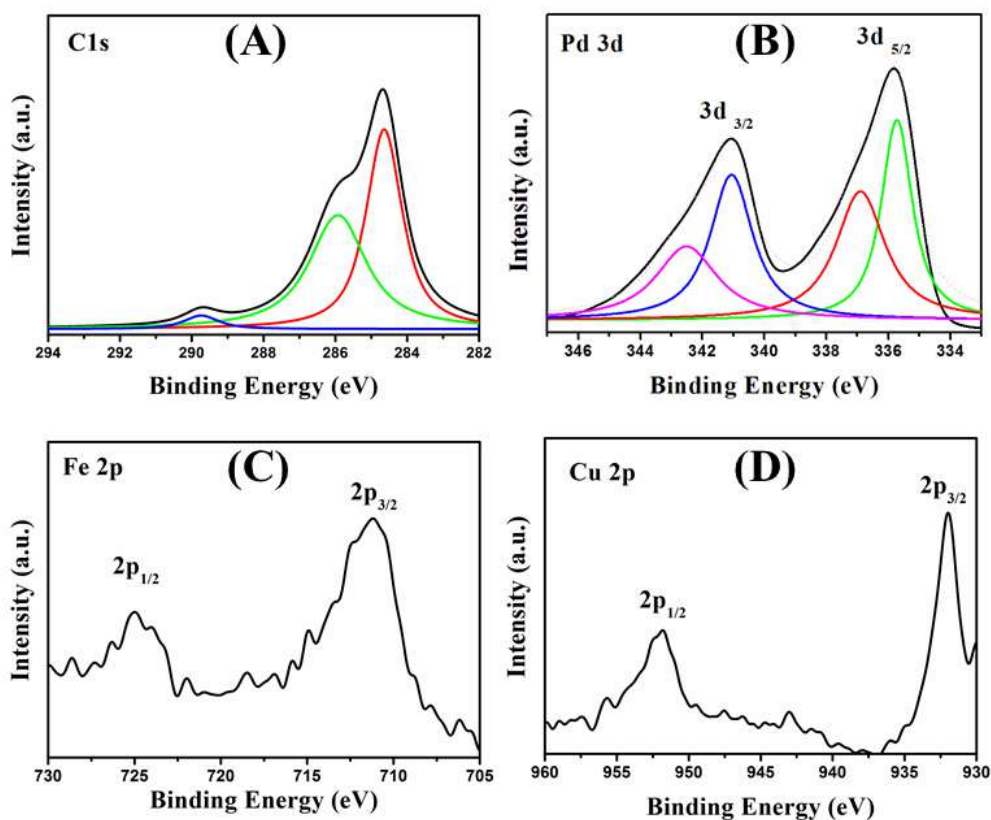


Fig 10: (A) C 1s, (B) Pd 3d, (C) Fe 2p, and (D) Cu 2p XPS spectra of Pd₃Fe_{0.5}Cu_{0.5}/C.

Electrocatalytic oxygen reduction activity related to Carbon-supported Pd_{4-x}Cu_x Nanoparticles (Pd_{4-x}Cu_x/C NPs)

The electrocatalytic activities of the as-prepared Pd_{4-x}Cu_x/C towards ORR were examined using cyclic voltammetry (CV) and rotating disc electrode (RDE) measurements. Figure 11a shows the CV plots of Pd₃Cu/C in 0.1 M KOH solution for both N₂ and O₂-saturated environment. An obvious and well-defined cathodic peak is observed in O₂ saturated KOH solution compared to that of N₂ which indicates good electrocatalytic activity of the catalyst towards ORR. As seen from the figure, Pd₃Cu/C shows ORR onset potential of ~ -0.37 V (vs. Ag/AgCl) in N₂-saturated solution which is much more positively shifted, i.e., ~ -0.26 V (vs. Ag/AgCl) in O₂-saturated 0.1 M KOH solution. The CV plots of PdCu/C and PdCu₃/C in 0.1 M KOH solution for both N₂ and O₂-saturated environment are presented in Figure 12. To get more insight on the catalytic activity for ORR, RDE measurements were performed at different rpm. Figure 11b,d,f shows representative linear sweep voltammograms (LSVs) at the rotating rate of 400, 900, 1600, 2500 and 3600 rpm for Pd₃Cu/C, PdCu/C and PdCu₃/C, respectively.

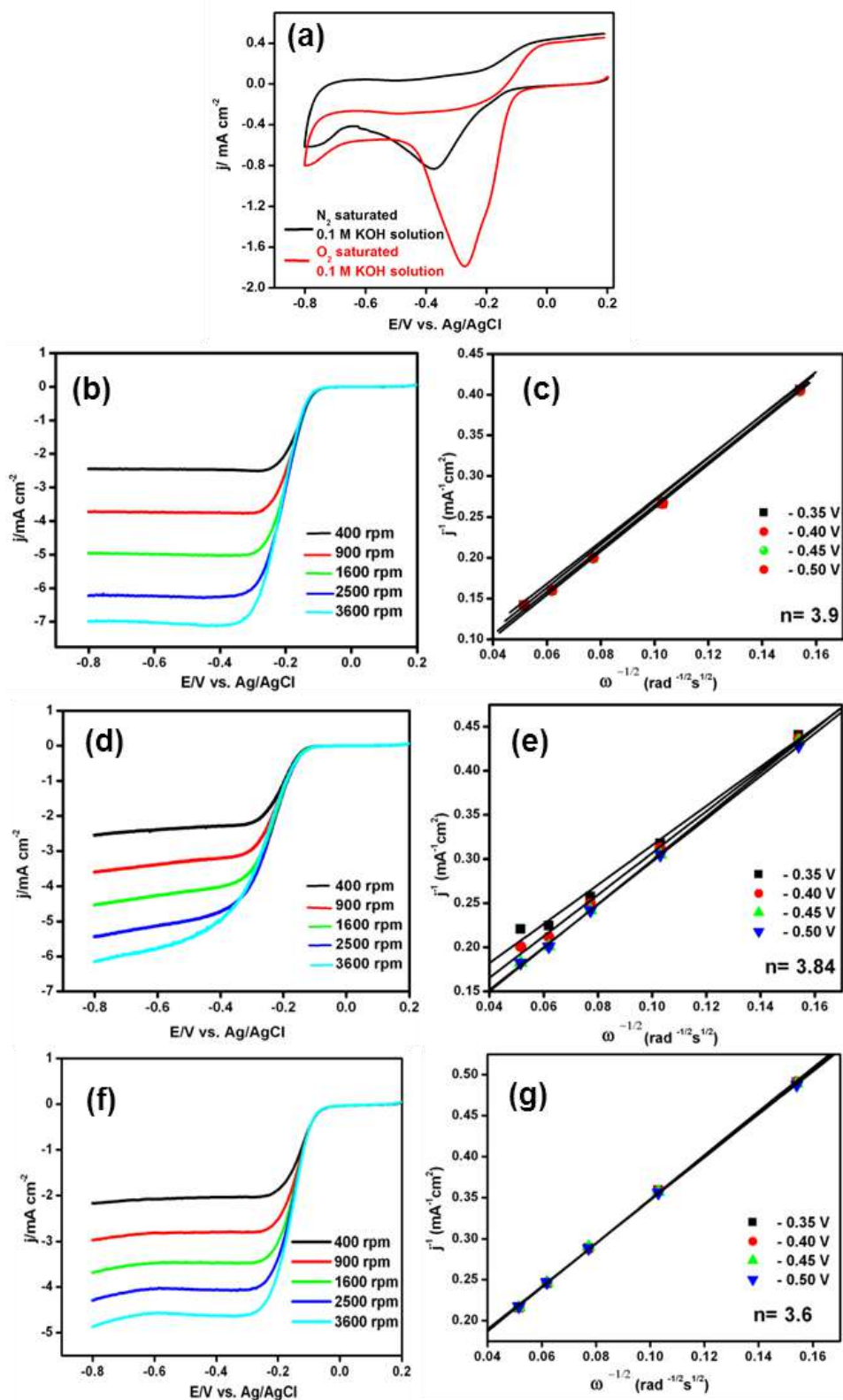


Fig 11: (a) Cyclic voltammetry plots of Pd₃Cu/C NPs in both N₂ and O₂-saturated 0.1 M KOH solution, rotating rate dependent linear sweep voltammogram (LSVs) at the scan rate of 10 mVs⁻¹ in O₂-saturated 0.1 M KOH and the Koutecky-Levich plots of j^{-1} vs. $\omega^{-1/2}$ at different potentials (b, c) for Pd₃Cu/C, (d, e) for PdCu/C and (f, g) for PdCu₃C NPs.

On the basis of these ORR polarization curves, the number of electron transferred (n) in ORR is calculated according to Koutecky-Levich (K-L) equation.

$$1/j = 1/j_k + 1/B \omega^{0.5}$$

$$B = 0.62 nF(D_{O_2})^{2/3} \nu^{-1/6} C_{O_2}$$

Where j is the current density, j_k is the kinetic current density, ω is the rotating rate of the electrode and B is the slope which could be obtained from the Koutecky-Levich plots. F is the Faraday constant (96500 C mol^{-1}), D_{O_2} is the diffusion co-efficient of O_2 in 0.1 M KOH ($2.0 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$), ν is the kinetic viscosity ($0.01 \text{ cm}^2 \text{ s}^{-1}$), C_{O_2} is the bulk concentration of O_2 ($1.2 \times 10^{-6} \text{ mol cm}^{-3}$) and the value of ' n ' represents the number of transferred electron in the ORR process. The K-L plots of j^{-1} vs. $\omega^{-1/2}$ at a potential of -0.35 V , -0.40 V , -0.45 V and -0.50 V displays good linearity (Figure 11c,e,g) which also indicates the first order kinetics of ORR. From the slope values the ' n ' value was calculated. At -0.40 V , ' n ' is found to be 3.9 for Pd_3Cu/C , 3.84 for $PdCu/C$, and 3.6 for $PdCu_3/C$ which are very close to 4.0. This implies that the ORR mechanism proceeds by " $4e^-$ " pathway ($O_2 + 2H_2O + 4e^- = 4OH^-$). Meanwhile the mass specific activities (j_m) are calculated at various potentials in the range of -0.35 V and -0.50 V for all the catalysts including the commercial Pd/C . Figure 13a shows the plot of mass specific activities of those NPs at different potentials.

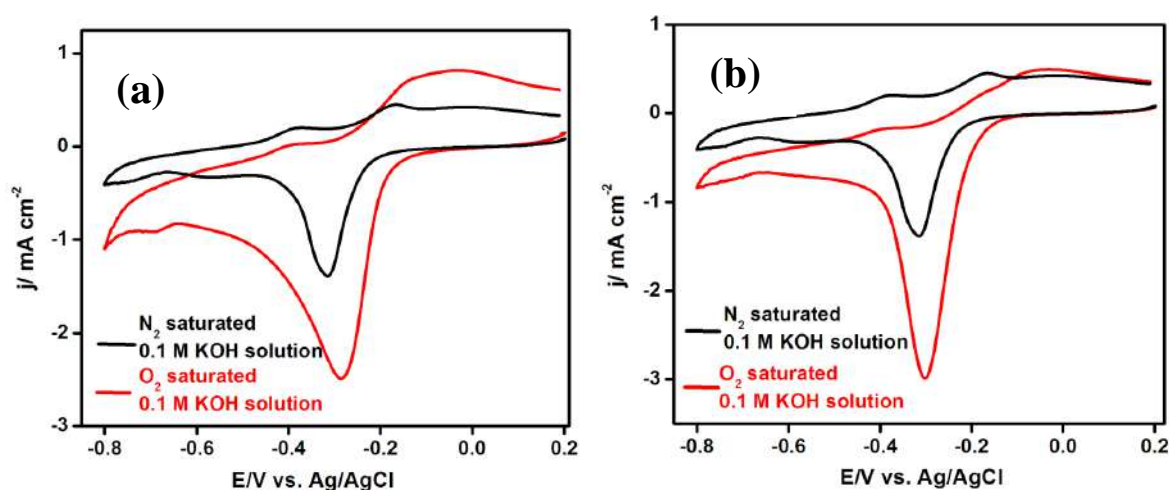


Fig 12: Cyclic voltammetry (CV) plots in both N_2 and O_2 -saturated 0.1 M KOH solution for (a) $PdCu/C$ and (b) $PdCu_3/C$ NPs, respectively.

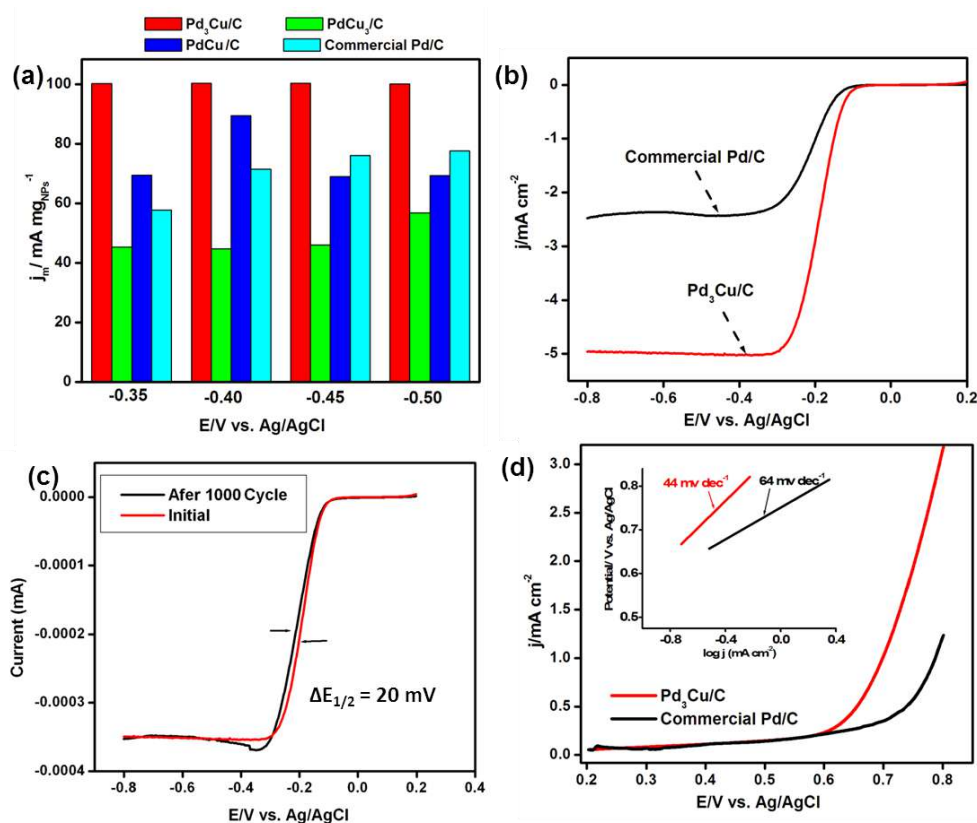


Fig 13: (a) Mass specific activities of $\text{Pd}_{4-x}\text{Cu}_x/\text{C}$ NPs at different potentials, (b) LSV curves at the rotating rate of 1600 rpm in O_2 -saturated 0.1 M KOH, (c) LSV curve after 1000 cycles to check the stability of $\text{Pd}_3\text{Cu}/\text{C}$ NPs and (d) LSVs and the tafel plots (inset) of OER measurements.

Based upon the calculated value they follow the order $\text{Pd}_3\text{Cu}/\text{C} > \text{PdCu}/\text{C} > \text{PdCu}_3/\text{C}$. It should be mentioned that $\text{Pd}_3\text{Cu}/\text{C}$ possess almost two fold enhanced mass activity compared to that of commercial Pd/C . Moreover, the LSV plots for both $\text{Pd}_3\text{Cu}/\text{C}$ and commercial Pd/C at 1600 rpm is compared in the Figure 13b from which it can be noticed that the synthesized catalyst exhibits lower ORR onset potential compared to that of the Pd/C . $\text{Pd}_3\text{Cu}/\text{C}$ shows ORR onset potential of -0.07 V which is much lower than that of Pd/C (-0.12 V). The above results revealed that $\text{Pd}_3\text{Cu}/\text{C}$ NPs exhibited much higher electrocatalytic activity toward ORR. This finding may be attributed to the structural behavior of that particular composition which enhances the electrocatalytic performance toward ORR. It follows an associative pathway/mechanism with the formation of $^*\text{OOH}$ intermediate which further dissociates to form O and OH. It can be believed that for $\text{Pd}_3\text{Cu}/\text{C}$ NPs the dissociation of $^*\text{OOH}$, i.e., O–O bond dissociation is more favorable which efficiently raise the d-band center and hence enhance the electrocatalytic performance. It is predicted that ORR would be enhanced by coupling Cu, with low occupancy of d-orbitals

with Pd possessing fully occupied d-orbitals. Catalyst stability and durability is one of the major challenges for practical application. To study the stability of the Pd₃Cu/C catalyst we have performed LSV at 1600 rpm after 1000 reaction cycles. As shown in the Figure 13c, the Pd₃Cu/C shows very high catalytic stability as the difference of $\Delta E_{1/2}$ (20 mV) with the initial is quite low. This indicates that the synthesized catalyst has good recycling stability for ORR. Considering the excellent electrocatalytic activity towards ORR, we also investigated activity of Pd₃Cu/C towards oxygen evolution reaction (OER) in 0.1 M KOH solution. Substantial efforts have been made to develop efficient electrocatalyst for OER in the last few years as it plays a vital role in several energy conversion and fuel cell applications. Figure 13d displays the LSVs for OER in the potential window of 0.2 V to 0.8 V vs. Ag/AgCl. Compared to the commercial Pd/C, Pd₃Cu/C exhibited high current nearly two fold greater and earliest onset potential which signifies the high electrocatalytic activity towards OER. The OER activity of the catalysts were further examined by the corresponding Tafel plots provided in the inset of Figure 13d. The calculated Tafel slope for Pd₃Cu/C NPs is 44 mV dec⁻¹ which is much lower than the commercially available one (64 mV dec⁻¹). As it is well known that smaller the Tafel slope and low onset potential indicates high electrocatalytic activity towards OER.

Electrocatalytic oxygen reduction activity related to Carbon-supported Pd₃Fe_{0.5}Cu_{0.5} and Pd_{4-x}Fe_x Nanoparticles

The electrocatalytic behavior of the binary PdFe₃/C, PdFe/C, and Pd₃Fe/C, and ternary Pd₃Fe_{0.5}Cu_{0.5}/C modified glassy carbon electrode (GCE) are measured in N₂ and O₂ saturated 0.1 M KOH, as shown in Fig. 14-17. Fig. 14A shows the CV plots of Pd₃Fe_{0.5}Cu_{0.5}/C NC in N₂ - and O₂-saturated 0.1 M KOH solution. A strong and well-defined reduction peak is observed in O₂-saturated 0.1 M KOH solution whereas no such characteristic reduction peak is observed in N₂-saturated electrolyte, signifying that the synthesized Pd₃Fe_{0.5}Cu_{0.5}/C NC has a good electrocatalytic activity towards ORR. Moreover, to determine the kinetics of ORR for Pd₃Fe_{0.5}Cu_{0.5}/C NC, rotating disk electrode (RDE) measurement is performed in O₂ saturated 0.1 M KOH solution at a rotating rate of 400, 900, 1600, 2500 and 3600 rpm as shown in Fig. 14B. Based on the ORR polarization curves (or linear sweep voltammograms), the number of electrons (n) transferred during ORR can be evaluated using the following Koutecky–Levich (K–L) equation.

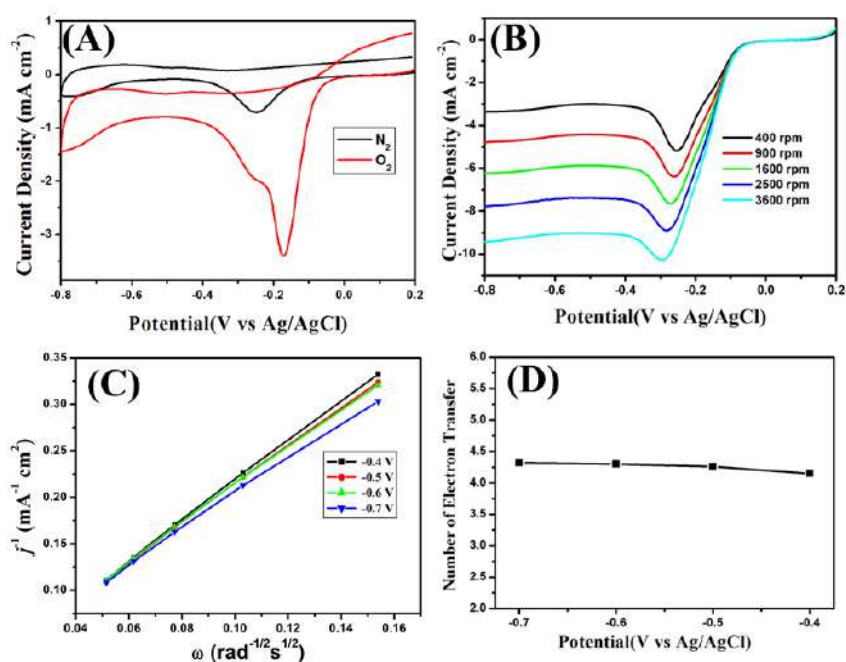


Fig. 14 (A) CV plots of Pd₃Fe_{0.5}Cu_{0.5}/C, N₂- and O₂-saturated 0.1 M KOH solution with a scan rate of 50 mV s⁻¹, (B) Rotating rate-dependent ORR polarization curves for respective NC with the scan rate of 10 mV s⁻¹, (C) K-L plots of j^{-1} vs. $\omega^{-1/2}$ for Pd₃Fe_{0.5}Cu_{0.5}/C NCs at different potential obtained from (B), and (D) The plot of the number of transferred electrons vs. potential for Pd₃Fe_{0.5}Cu_{0.5}/C NCs.

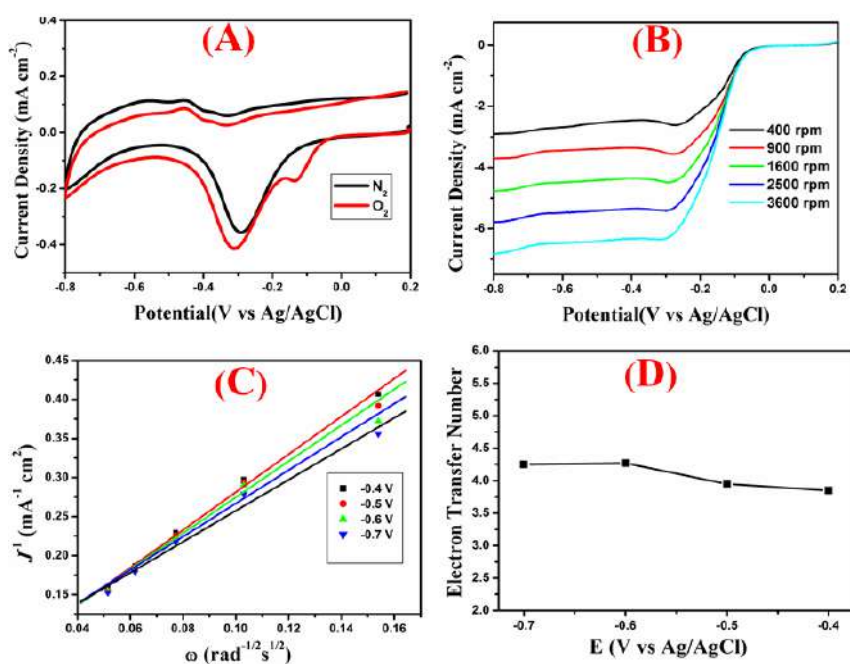


Fig. 15 Electrochemical data for ORR over Pd₃Fe/C nanocatalyst. (A) CV plots in the N₂ and O₂ saturated 0.1 M KOH solution with a scan rate of 50 mV s⁻¹, (B) Rotating rate-dependent ORR polarization curves with the scan rate of 10 mV s⁻¹, (C) K-L plots of j^{-1} vs. $\omega^{-1/2}$ at different potential obtained from ORR results, and (D) The plot of the number of transferred electrons vs. potential.

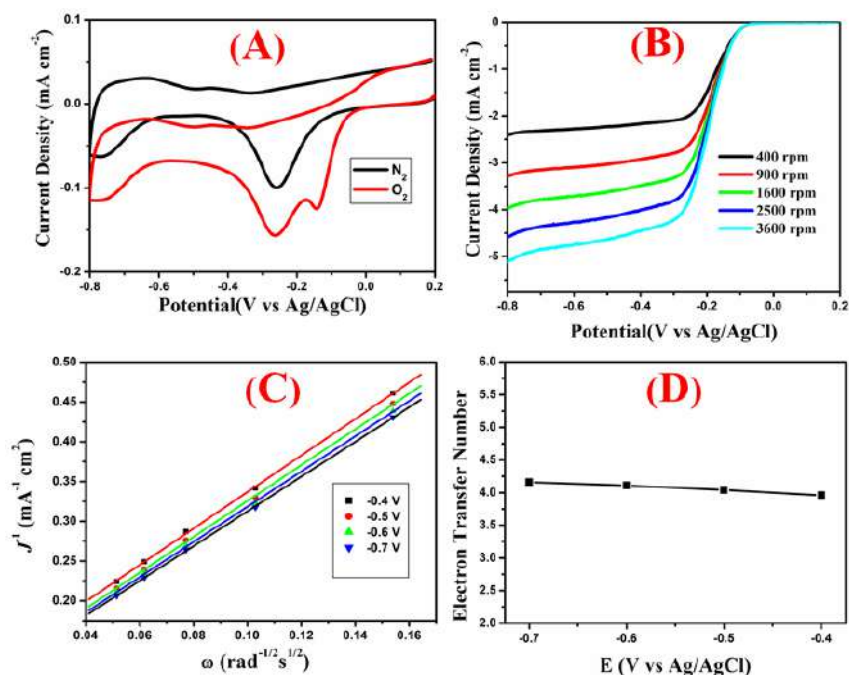


Fig. 16 Electrochemical data for ORR over PdFe/C nanocatalyst. (A) CV plots in the N₂ and O₂ saturated 0.1 M KOH solution with a scan rate of 50 mV s⁻¹, (B) Rotating rate-dependent ORR polarization curves with the scan rate of 10 mV s⁻¹, (C) K-L plots of j^{-1} vs. $\omega^{-1/2}$ at different potential obtained from ORR results, and (D) The plot of the number of transferred electrons vs. potential.

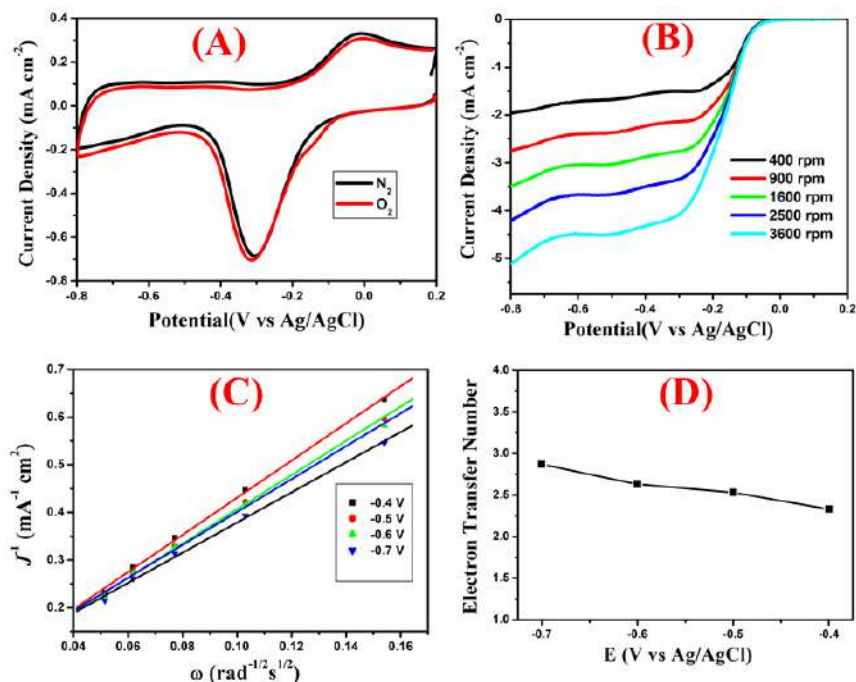


Fig. 17 Electrochemical data for ORR over PdFe₃/C nanocatalyst. (A) CV plots in the N₂ and O₂ saturated 0.1 M KOH solution with a scan rate of 50 mV s⁻¹, (B) Rotating rate-dependent ORR polarization curves with the scan rate of 10 mV s⁻¹, (C) K-L plots of j^{-1} vs. $\omega^{-1/2}$ at different potential obtained from ORR results, and (D) The plot of the number of transferred electrons vs. potential.

The K–L (j^{-1} vs. $\omega^{-1/2}$) plots obtained on the basis of the corresponding ORR polarization curves for Pd₃Fe_{0.5}Cu_{0.5}/C NC at a potential of –0.40, –0.50, –0.60, and –0.70 V show good linearity (Fig. 14C) thereby implying a first-order kinetics for ORR. From the slope (1/B) of K–L plots, the n values for Pd₃Fe_{0.5}Cu_{0.5}/C NC at –0.40, –0.50, –0.60, and –0.70 V can be calculated. Fig. 14D gives the corresponding plot of ‘n’ vs. potential (V). It can be seen that the ‘n’ values from –0.40 to –0.70 V are ~4.0, suggesting that the ORR mechanism on Pd₃Fe_{0.5}Cu_{0.5}/C NC follows the direct “4e[–]” pathway (O₂ + 2H₂O + 4e[–] → 4OH[–]). The electrocatalytic ORR mechanism of binary PdFe/C NCs also follow the direct “4e[–]” pathway in the case of Pd₃Fe/C and PdFe/C NCs whereas “2e[–]” pathway possibly in the case of PdFe₃/C NC (See Fig. 15-17).

To elucidate the electrocatalytic behavior of the NCs, the electrochemically active surface area (ECSA) are calculated for all the NCs using cyclic voltammetry (CV) curves in N₂ saturated 0.1 M KOH electrolyte at a scan rate 50 mV s^{–1} (Fig. 18A). The peaks in the potential range –0.5–0 V originate from the palladium oxide reduction. The ECSA of ternary Pd₃Fe_{0.5}Cu_{0.5}/C (63.22 m²g^{–1}) is found to be higher than binary Pd₃Fe/C (32.99 m²g^{–1}), PdFe/C (17.87 m²g^{–1}), PdFe₃/C (8.3 m²g^{–1}), standard Pt/C (8.7 m²g^{–1}), and standard Pd/C (1.42 m²g^{–1}). This improved electrocatalytic ORR activity can be co-related to the higher ECSA values. The higher ECSA values of the binary and ternary NCs can be attributed to the synergistic effect between the elements. To understand the superiority of the electrocatalytic performance, the ORR polarization curves for the standard Pd/C, standard Pt/C, binary PdFe₃/C, PdFe/C, Pd₃Fe/C and ternary Pd₃Fe_{0.5}Cu_{0.5}/C NCs are studied in O₂-saturated 0.1 M KOH electrolyte at a rotation of 1600 rpm and a scan rate of 10 mVs^{–1} (Fig. 18B). The onset potential (E_{onset} = 0.044 V) and half-wave potential (E_{1/2} = –0.145 V) of Pd₃Fe_{0.5}Cu_{0.5}/C NC are found to be more positive than those of standard Pd/C (E_{onset} = –0.103 V, E_{1/2} = –0.212 V), Pt/C (E_{onset} = –0.069 V, E_{1/2} = –0.154 V), binary PdFe₃/C (E_{onset} = –0.047 V, E_{1/2} = –0.168 V), PdFe/C (E_{onset} = –0.036 V, E_{1/2} = –0.194 V), and Pd₃Fe/C (E_{onset} = –0.030 V, E_{1/2} = –0.147 V) under identical conditions, suggesting superior ORR activity of the ternary Pd₃Fe_{0.5}Cu_{0.5}/C NC. The overall ORR behavior follows the order: Pd₃Fe_{0.5}Cu_{0.5}/C > Pd₃Fe/C > PdFe/C > PdFe₃/C > Pt/C > Pd/C. Moreover, the apparent current density values follow the same order.

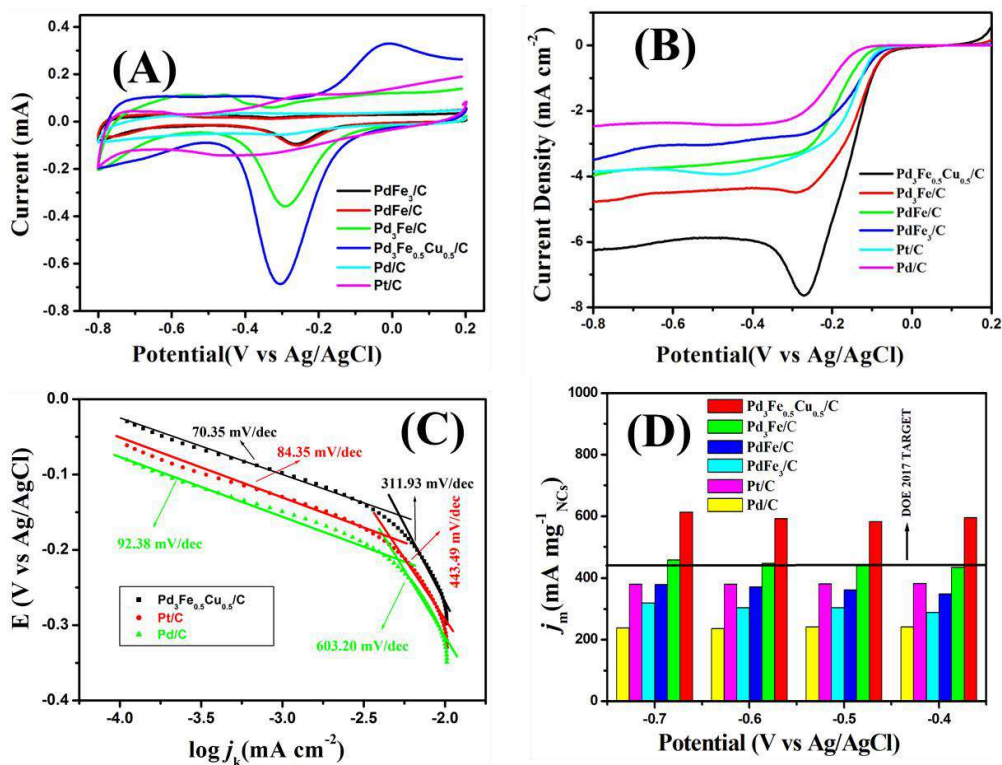


Fig. 18 (A) CV plots of all the electrocatalysts at N_2 -saturated 0.1 M KOH solution at a scan rate of 50 mV s^{-1} , (B) Comparison of rotating rate-dependent ORR polarization curves for different catalyst at 1600 rpm, (C) Tafel plots, and (D) Mass activities of NCs under different potential at 1600 rpm. (These values are calculated as per the loading mass of the NCs).

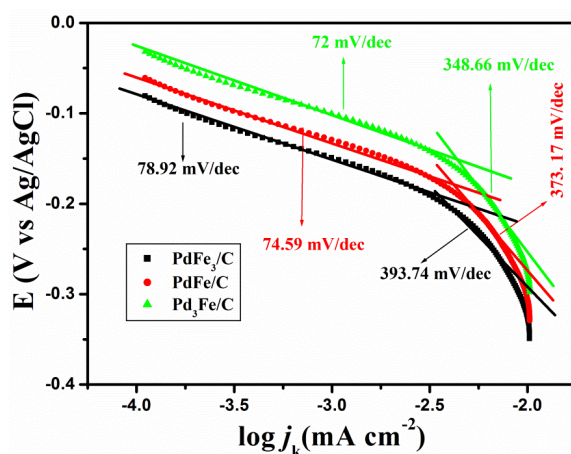


Fig. 19 Comparison of Tafel plots Pd₃Fe/C, PdFe/C and PdFe₃/C nanocatalysts.

To compare their electrocatalytic activities, the corresponding Tafel plots derived from the ORR polarization curves for all NCs are provided in Fig. 18C and 19. The Tafel slope calculated for Pd₃Fe_{0.5}Cu_{0.5}/C in the high potential range (*i.e.*, -3.9 V to -2.5 V) is $70.35 \text{ mV dec}^{-1}$, while the other in the low potential range is $311.93 \text{ mV dec}^{-1}$. The two Tafel

slopes indicates different ORR behaviors in different potential ranges. These two slopes are comparatively smaller than those of standard Pt/C (84 and 443.4 mV dec⁻¹), Pd/C (92.38 and 603.2 mV dec⁻¹) as well as for other catalysts compositions and follows the order: Pd₃Fe/C (72 and 348.66 mV dec⁻¹) < PdFe/C (74.59 and 373.17 mV dec⁻¹) < PdFe₃/C (78.92 and 393.74 mV dec⁻¹) < Pt/C (84 and 443.4 mV dec⁻¹) > Pd/C (92.38 and 603.2 mV dec⁻¹), in the high and low potential range, respectively. This indicates that ternary Pd₃Fe_{0.5}Cu_{0.5}/C has higher efficiency towards ORR as compared to that of the other catalyst systems studied and several reported Pd-based catalysts as detailed in Table S1 in the ESI. The mass activity (MA) and specific activity (SA) are the two important parameters for better assessment of the catalytic activity obtained by normalizing the kinetic current (j_k) w.r.t. the mass loading of the catalyst. Here, j_k has been calculated based on the K-L equation. The mass activities of various NCs under different potential at 1600 rpm are presented in Fig. 18. The MA and SA of Pd₃Fe_{0.5}Cu_{0.5}/C at -0.4 V (595.8 mA mg⁻¹ and 84 mA cm⁻², respectively) are much higher than the Pt/C (382.2 mA mg⁻¹ and 53.89 mA cm⁻²) and Pd/C (241.6 mA mg⁻¹ and 34.06 mA cm⁻²) under the identical experimental conditions. The MA and SA values of Pd₃Fe_{0.5}Cu_{0.5}/C are also larger than those of the PdFe/C NCs which follows the order: Pd₃Fe/C (434.9 mA mg⁻¹ and 61.32 mA cm⁻²) > PdFe/C (349 mA mg⁻¹ and 49.20 mA cm⁻²) > PdFe₃/C (288.5 mA mg⁻¹ and 40.67 mA cm⁻²). These experimental findings are evident of the enriched kinetic ability of the Pd₃Fe_{0.5}Cu_{0.5}/C, making them an auspicious ORR catalyst.

Apart from the activity, the high durability of the catalysts towards ORR is also an essential factor for energy conversion systems. Thus, to check the durability chronoamperometry (CA) test was performed in O₂-saturated 0.1 M KOH at -0.3V (V vs. Ag/AgCl) with a rotation speed of 1600 rpm for 7200 sec. It can be observed from Fig. 20A that the initial activity decay for Pt/C and Pd₃Fe/C NC are ~25.5% and ~3.8%, respectively after 7200 sec of testing, while the Pd₃Fe_{0.5}Cu_{0.5}/C NC unveils only a loss of ~1.4% of its initial activity after 7200 sec, under identical experimental conditions. This demonstrates the excellent stability of the Pd₃Fe_{0.5}Cu_{0.5}/C NC in basic media.

The structure and morphology of the Pd₃Fe_{0.5}Cu_{0.5}/C NC as measured by TEM analyses after the CA test are shown in Fig. 21. There is meagre change in the morphology of the Pd₃Fe_{0.5}Cu_{0.5}/C NC after the CA test. To support the structural stability, an accelerated durability test (ADT) is also performed for 1000 cycles between 0.2 V and -0.80 V vs. Ag/AgCl at a scan rate of 100 mV s⁻¹ in O₂-saturated 0.1 M KOH solution for Pt/C and Pd₃Fe_{0.5}Cu_{0.5}/C NC (Fig. 20B,C). Fig. 20B,C shows the associated ORR polarization curves

at 1600 rpm before and after 500 and 1000 CV cycles. The MA of the Pd₃Fe_{0.5}Cu_{0.5}/C NC has been deteriorated to a lower extent (5.17%) in comparison to Pt/C (11%) after 1000 potential CV cycles. There is a clear drop of onset potential in the case of Pt/C, whereas, the onset potential remains almost same after 500 and 1000 cycles for Pd₃Fe_{0.5}Cu_{0.5}/C catalyst. The E_{1/2} of Pt/C (26 mV) also shows a larger change compared to that of Pd₃Fe_{0.5}Cu_{0.5}/C (10 mV) NC. The above discussion indicates that Pd₃Fe_{0.5}Cu_{0.5}/C NC is the most efficient catalyst in basic media. To generalize the ORR behavior of the Pd₃Fe_{0.5}Cu_{0.5}/C NC in the acidic electrolyte, we conducted electrocatalytic studies in 0.5 M H₂SO₄ solution. As presented in Fig. 22, it is apparent that Pd₃Fe_{0.5}Cu_{0.5}/C NC is very active towards ORR in 0.5 M H₂SO₄ solution as well. It can be seen that the ‘n’ values from 0.1 to 0.05 V are ~4.0, suggesting that the ORR mechanism on Pd₃Fe_{0.5}Cu_{0.5}/C NC follows the direct “4e⁻” pathway (O₂ + 4H⁺ + 4e⁻ → 2 H₂O).

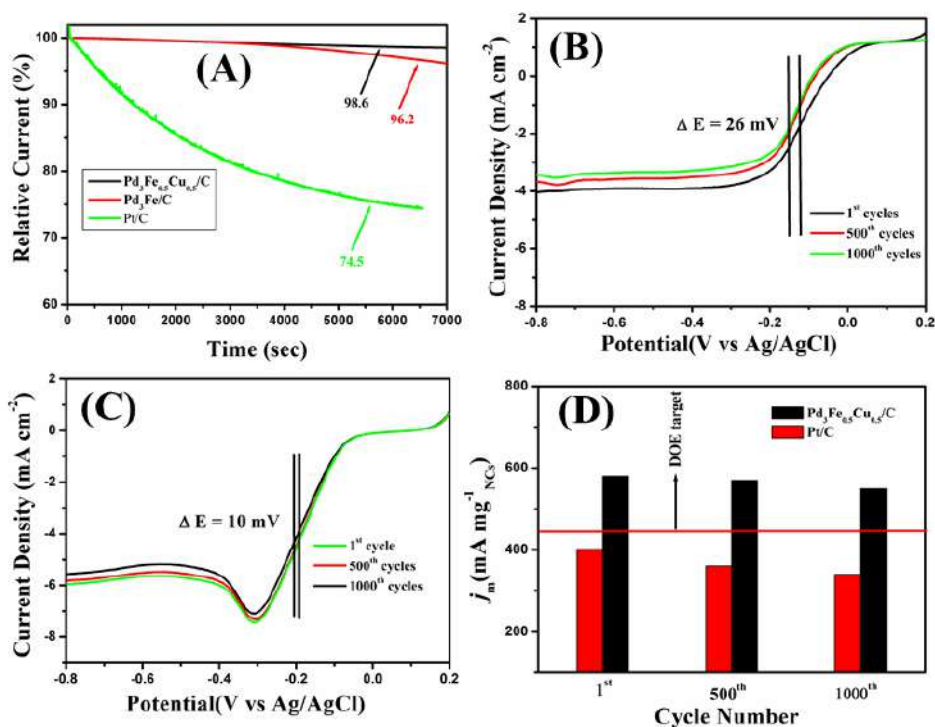


Fig. 20 (A) CA curves of different catalysts, recorded at -0.3 V in a O₂-saturated 0.1 M KOH solution with a rotation rate of 1600 rpm towards ORR, (B) ORR polarization curves of Pt/C and (C) ORR polarization curves of Pd₃Fe_{0.5}Cu_{0.5}/C at 1600 rpm before and after the stability test at different potential scans, (D) Comparison of mass activity after the stability test at different potential scans at 1600 rpm.

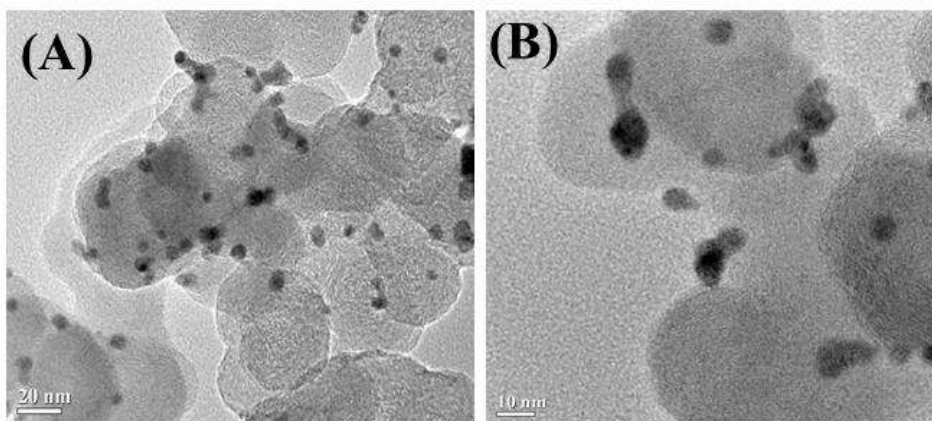


Fig. 21. TEM and HR-TEM images of Pd₃Fe_{0.5}Cu_{0.5}/C after chronoamperometry test.

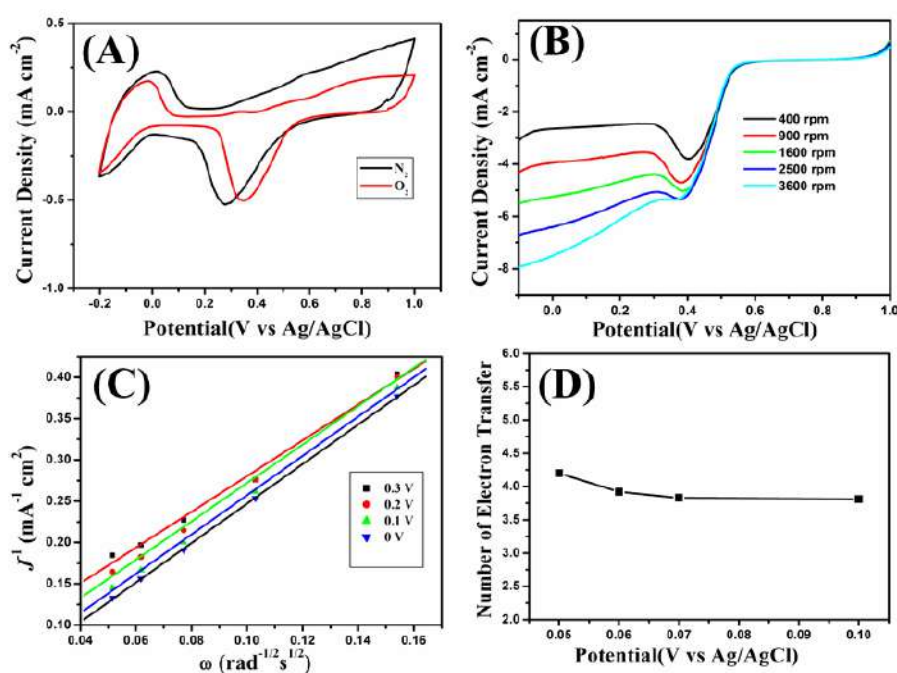


Fig. 22 Electrochemical data for ORR over Pd₃Fe_{0.5}Cu_{0.5}/C nanocatalyst. (A) CV plots of Pd₃Fe_{0.5}Cu_{0.5}/C, N₂- and O₂-saturated 0.5 M H₂SO₄ solution with a scan rate of 50 mV s⁻¹, (B) Rotating rate-dependent ORR polarization curves for respective NC with the scan rate of 10 mV s⁻¹, (C) K-L plots of j^{-1} vs. $\omega^{-1/2}$ for Pd₃Fe_{0.5}Cu_{0.5}/C NCs at different potential obtained from (B), and (D) The plot of the number of transferred electrons vs. potential for Pd₃Fe_{0.5}Cu_{0.5}/C NCs.

The enhanced ORR performance of Pd₃Fe_{0.5}Cu_{0.5}/C NC is due to their unique structural and compositional superiorities. From the HR-TEM images, the unique half-embedded and half expose interface structure was seen. i.e., one-half of NPs are embedded into the carbon matrix and another part exposed from the carbon matrix. Therefore, there is a

stable electronic coupling between the carbon matrix and Pd₃Fe_{0.5}Cu_{0.5} NPs. This means that the half embedded part possess very faster interfacial electron transfer ultimately enhancing the conductivity of the Pd₃Fe_{0.5}Cu_{0.5} NPs. Moreover the other exposed part is also highly active as the reactant molecule (O₂ diffusion layer) directly interacts with the catalyst layer. The carbon matrix also acts as superior support as they effectively prevent the NPs aggregation, improving the stability of the Pd₃Fe_{0.5}Cu_{0.5} in the electrochemical process. Furthermore, Pd₃Fe_{0.5}Cu_{0.5}/C nanoalloy exhibits many crystal defects on their surfaces, including vacancy, atomic steps and dislocation defects, that can work as abundant catalytically active sites accessible to the reactant, ultimately enhancing ORR activity. The ECSA due to unique structure of the catalyst, which can expose more electrochemically active sites available to reactant molecule. Alloying Pd (fully occupied d-orbital) with Fe and Cu (low occupancy d-orbital) can improve the electronic properties of Pd, which ultimately enhance the electro catalytic activity by decreasing the Gibbs free energy of the electronic transition involved in the ORR process. One of the salient parameters to understand the enhancement of ORR activity is the lattice strain effect in catalysts. The lattice strain changes the d-band center of the metal catalyst, which resolve the surface oxygen adsorption property of the catalyst surface. A down shift in the d-band center of Pd weakens the binding energy of absorption of intermediates such as OH⁻_{ads} and CO_{ads} thereby conserves more active sites on the catalyst surface. As a result, the catalyst shows better electrocatalytic activity. Incorporation of Cu and Fe (small lattice parameter) in Pd (0.38898 nm) with larger lattice parameter results in lattice strain in the Pd alloys. In this case, we have calculated lattice strain from XRD data that Pd₃Fe_{0.5}Cu_{0.5}/C has the smallest lattice parameter among all the PdFe/C NCs, Pd/C and Pt/C.

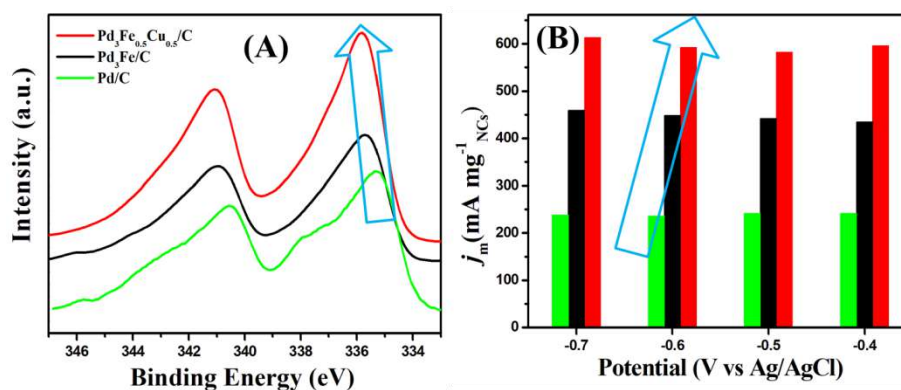


Fig. 23 Correlation of binding energy change of Pd 3d to ORR activity in terms of mass activity for Pd₃Fe_{0.5}Cu_{0.5}/C (red), Pd₃Fe/C (black), and Pd/C (green) NCs.

One of the most important parameters for high performance ORR activity is the linear relation between the metal d-band center (ϵ_d , with respect to Fermi level) and binding energy of adsorbed species on the metal surface (e.g., oxygen, hydrogen and methanol). For oxygen adsorption, an upper weight position in ϵ_d increases the interaction of the 2p states oxygen with the metal d states, i.e., forms a stronger metal-oxygen bond. On the other hand, a downshift of ϵ_d causes a weak interaction with oxygen. The positive shifts of binding energy represent a downshift of d-band center with respect to Fermi level. The downshift of Pd d-band center can further be closely correlated with the weaker Pd-O bond energy, by means of which the intermediate OH_{ad} species adsorption on the Pd surface can be decreased, thus sustaining the more active sites for ORR and at the end resulting in the enrichment in ORR activity. For that reason, the alteration of the d band center for Pt or Pd through the formation of alloys with second metals could increase the kinetics of the ORR. There are several theoretical reports on ORR where DFT calculation has been used over various catalyst alloys with compositions of Pt_3X or Pd_3X ($\text{X} = \text{Cu}, \text{Ni}, \text{Co}, \text{Fe}, \text{Ag}, \text{Au}, \text{Ir}, \text{etc.}$) by using d-band center model. The upshift of ~ 0.50 and ~ 0.40 eV in the binding energies of Pd $3d_{5/2}$ for $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$ and $\text{Pd}_3\text{Fe}/\text{C}$ NCs in comparison to Pd/C as shown in Fig. 23 implies the aforesaid down shift trends of Pd d-band center which leads to the enhancement of the catalytic performance and acceleration in terms of mass activity of the ORR activity. Thus, among all the catalytic systems, the $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$ shows the best electrocatalytic activity towards ORR in both acidic and basic media.

11. Conclusions summarizing the achievements and indication of scope for future work:

In this work, we have adopted a facile synthesis of various compositions of PdM/C ($\text{M} = \text{Co}, \text{Cu}, \text{Fe}$) and $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$ nanoalloys that exhibited remarkably enhanced catalytic activity towards ORR. The Cu incorporated $\text{Pd}_3\text{Fe}/\text{C}$ NCs modified the lattice constrain and d-band center of the $\text{Pd}_3\text{Fe}_{0.5}\text{Cu}_{0.5}/\text{C}$, which impressively displayed superior activity and long term stability when used as a cathode electrocatalyst. The nanoalloy with defects on their surfaces, for instance; vacancy, dislocation and low-coordinate atomic steps offer numerous energetic sites accessible to catalyze the fuel cell reaction. Therefore, the project work offers new possibilities for the development of sustainable energy conversion and related technologies with superior activity, favorable kinetics and long term durability in relatively low cost.

This project enabled us to develop various compositions of bimetallic and trimetallic Pd-based hybrid electrocatalysts with or without CeO₂ as promoter having tunable properties that have large impact on catalysts design for ORR, a fundamental reaction of fuel cell. The reaction mechanism and kinetic studies toward understanding the ORR have directed the avenues for possible commercial exploitation of non-platinum based electrocatalysts for cathodic ORR.

12. S&T benefits accrued

i. List of Research publications

S No	Authors	Title of the paper	Name of the Journal	Volume	Pages	Year
1	K.K. Hazarika, C. Goswami, H. Saikia, B.J. Borah, P. Bharali	Cubic Mn ₂ O ₃ nanoparticles on carbon as bifunctional electrocatalyst for oxygen reduction and oxygen evolution reactions	Molecular Catalysis	451	153-160	2018
2	C. Goswami, K.K. Hazarika, P. Bharali	Transition metal oxide nanocatalysts for oxygen reduction reaction	Material Science for Energy Technologies	1	117-128	2018
3	P. Deka, B.J. Borah, H. Saikia, P. Bharali	Cu-based nanoparticles as emerging environmental catalysts	The Chemical Record	18	1-13	2018
4	B.J. Borah, A. Mahanta, M. Mondal, H. Gogoi, Y. Yamada, P. Bharali	Cobalt-copper nanoparticles catalyzed selective oxidation reactions: Efficient catalysis at room temperature	ChemistrySelect	3	9826-9832	2018
5	H. Saikia, B.J. Borah, Y. Yamada, P. Bharali	Enhanced catalytic activity of CuPd alloy nanoparticles towards reduction of nitroaromatics	Journal of Colloid and Interface Science	486	46-57	2017

		and hexavalent chromium				
6	H. Saikia, B.J. Borah, P. Bharali	Room temperature reduction of nitroaromatics using Pd nanoparticles stabilized on nano-CeO ₂	ChemistrySelect	2	10524-10530	2017
7	H. Saikia, K.K. Hazarika, B. Chutia, B. Choudhury, P. Bharali	A simple chemical route toward high surface area CeO ₂ nanoparticles displaying remarkable radical scavenging activity	ChemistrySelect	2	3369-3375	2017
8	H. Saikia, R. Duarah, P. Sudarsanam, S.K. Bhargava, P. Bharali	PdCu nanoparticles stabilized on porous CeO ₂ for catalytic degradation of azo dyes: Structural characterization and kinetic studies	ChemistrySelect	2	2123-2130	2017
9	P. Deka, P. Sarmah, R.C. Deka, P. Bharali	Hetero-nanostructured Ni/ α -Mn ₂ O ₃ as highly active catalyst for aqueous phase reduction reactions	ChemistrySelect	1	4726-4735	2016
10	P. Deka, A. Hazarika, R.C. Deka, P. Bharali	Influence of CuO morphology on enhanced catalytic degradation of methylene blue and methyl orange	RSC Advances	6	95292 - 95305	2016
11	P. Deka, R. Choudhury, R.C. Deka, P. Bharali	Influence of Ni on enhanced catalytic activity of Cu/Co ₃ O ₄ towards reduction of nitroaromatic compounds: studies on the reduction kinetics	RSC Advances	6	71517 - 71528	2016

12	P. Deka, R.C. Deka, P. Bharali	Porous CuO nanostructure as reusable catalyst for oxidative degradation of organic water pollutants	New Journal of Chemistry	40	348-357	2016
13	C. Goswami, B. Chutia, P. Bharali	Metal and Metal Oxide-Based Nanomaterials for Electrochemical Applications ISBN: 978-3-030-04473-2	In Emerging Nanostructured Materials for Energy and Environmental Science (Springer)	Ch. 12		2018*
14	B.J. Borah, M. Mondal, P. Bharali	Palladium-based Hybrid Nanocatalysts: Application toward Reduction Reactions ISBN: 978-0-12-814134-2	In Noble Metal-Metal Oxide Hybrid Nanoparticles: Fundamentals and Applications (Elsevier)	Ch. 27	565-583	2018*

* Book chapters published.

ii. Manpower trained on the project

a) Research Scientists or Research Associates NIL

b) No. of Ph.D. produced One (Degree awarded) One (on going)

c) Other Technical Personnel trained NIL

iii. Patents taken, if any NIL

13. Financial Position:

No	Financial Position/ Budget Head	Funds Sanctioned	Expenditure	% of Total cost
I	Salaries/ Manpower costs	17,50,000 + (6,00,000 + 6,00,000 + 4,00,000 + 1,50,000)	17,32,340	99
II	Supplies & Materials			
III	Contingencies			
IV	Travel			
V	Overhead Expenses			
VI	Equipment	11,50,000	11,50,000	100
VII	Others, if any/ <u>Interest gained</u>	2,690	NA	NA
	Total	29,02,690	28,82,340	

14. Procurement/ Usage of Equipment

a)

S No	Name of Equipment	Make/ Model	Cost (FE/ Rs)	Date of Installation	Utilization Rate (%)	Remarks regarding maintenance/
1	Electrochemical work station with Rotating Disc electrode system	PGSTAT 204 & Autolab	9,85,496	25.05.2016	100	Using current project grant
2	Gas Cylinders (N ₂ -1No., O ₂ -1 No., H ₂ -1No.) with regulators	Aneer Engg.	77,332	12.01.2016	100	Refilling is necessary
3	Vacumm Oven + vacuum pump	NSW India + Millipore, Merck	1,23,000	02.03.2016	100	Using current project grant

b) Plans for utilizing the equipment facilities in future

The equipment would be actively employed for further studies related to electrochemical oxygen reduction activity for newly developed catalysts for possible commercialization in the near future. This instrument has been actively employed for carrying out MSc and PhD dissertation works in the last 2.5 years. It will further continue to train Master and PhD students in the field of electrocatalysis.

Name and Signature with Date:

a. Dr. Pankaj Bharali
(Principal Investigator)

b. Not applicable
(Co-Investigator)

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (2015- 2016)

U.C. Pertains to

✓ First Release	Second Release	Third Release	Fourth Release	Final Release
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✓ appropriate box

Is the UC provisional

YES / NO ✓

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : Amount NA
: Letter/Order No
: Date
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : Amount **11,50,000**
: Letter/Order No SB/FT/CS-048/2014
: Date 16.06.2015
- 6b. Interest earned, if any : 329
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 11,50,329
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : 2,00,332
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : 9,49,997
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: NA
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) 9,49,997


 31/7/16
Finance Officer
 Tezpur U



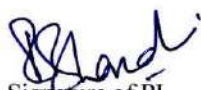
UTILISATION CERTIFICATE

Certified that out of Rs. 11,50,000/- of **Non-Recurring** grants-in-aid sanctioned during the year 2015-16 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 16.06.2015 and Rs. NIL on account of unspent balance of the previous year, a sum of Rs 2,00,332/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. 9,49,997/- [9,49,668/- + 329/- (Interest)] remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the grants-in-aid payable during the next year i.e. 2016-2017_____.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

Kinds of checks exercised:

- 1.
- 2.



Signature of PI

Date: 22.7.16



Signature of Registrar/ Accounts Officer

Date:

Finance Officer
Tezpur University



Signature of Head of the Institution

Date:

Registrar
Tezpur University,

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (2015- 2016)

U.C. Pertains to

✓ appropriate box

✓ First Release	Second Release	Third Release	Fourth Release	Final Release
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Is the UC provisional

YES / NO ✓

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : Amount NA
: Letter/Order No
: Date
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : Amount **6,00,000**
: Letter/Order No SB/FT/CS-048/2014
: Date 16.06.2015
- 6b. Interest earned, if any : NIL
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 6,00,000
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : 5,49,446
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : 50,554
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: NA
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) 50,554


 30/7/16
Finance Officer
Tezpur University



UTILISATION CERTIFICATE

Certified that out of Rs. 6,00,000/- of **Recurring** grants-in-aid sanctioned during the year 2015-16 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 16.06.2015 and Rs. NIL on account of unspent balance of the previous year, a sum of Rs 5,49,446/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. 50,554/- remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the **Recurring** grants-in-aid payable during the next year i.e. 2016-2017.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

Kinds of checks exercised:

- 1.
- 2.



Signature of PI

Date: 22.7.16


30/7/16

Signature of Registrar/ Accounts Officer

Date:

**Finance Officer
Tezpur University**



Signature of Head of the Institution

Date:

**Registrar
Tezpur University**

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

REQUEST FOR ANNUAL INSTALMENT WITH UP-TO-DATE STATEMENT OF EXPENDITURE

(Two copies)

1. SERB Sanction Order No and date : SB/FT/CS-048/2014 and 16.06.2015
 2. Name of the PI : Dr. Pankaj Bharali
 3. Total Project Cost : 29,49,600/-
 4. Revised Project Cost(if applicable) : NA
 5. Date of Commencement : 30.07.2015
 6. Statement of Expenditure : (month wise expenditure incurred during current financial year)

Month & Year	Expenditure incurred/ committed
September 2015	13,097
October 2015	14,000+62,500 = 76,500
November 2015	14,000
December 2015	14,000
January 2016	14,000 + 15,550 + 81,750 + 24,400 = 1,35,700
February 2016	14,000 + 2,36,051 + 77,332 + 11,028 = 338,411
March 2016	14,000 + 5,000 + 2,070+ 14,000 + 1,23,000 = 1,58,070
Total	7,49,778

7. Grant received in each year:
- a. 1st Year : 17,50,000/-
 b. 2nd Year : NA
 c. 3rd Year : NA
 d. Interest, if any : 329/-
 e. Total (a+b+c+d) : 17,50,329/-

P. Bharali
 7/6/2016

B. Kumar
 Finance Officer
 Tezpur University
 16.6.16

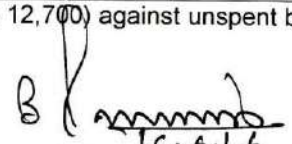
Statement of Expenditure
(For the financial Year 2015-2016, 30.06.2015 till 31.03.2016)

Sr No (I)	Sanctioned Heads (II)	Total Funds allocated (Indicate sanctioned or revised) (III)	Expenditure Incurred			Total Expenditure till (VII = IV + V + VI)	Balance as on 01.04.2016	Requirements of funds up to 31 st March Next year	Remarks (if any)
			1 st Year (30.06.2015 to 31.03.2016) (IV)	2 nd Year (01.04.2016 to 31.03.2017) (V)	3 rd Year & so on (01.04.2017 to 31.03.2018) (VI)				
1.	Manpower cost	6,00,000	1,11,097	NA	NA	1,11,097	50,554	6,00,000	
2.	Consumables		3,17,801	-	-	3,17,801			
3.	Travel		0	-	-	0			
4.	Contingencies		20,550	-	-	20,550			
5.	Others, if any		0	-	-	0			
6.	Overhead Expenses		99,998	-	-	99,998			
7.	Equipment	11,50,000	2,00,332	-	-	2,00,332	9,49,668		
8.	Total	17,50,000	7,49,778	NA	NA	7,49,778	10,00,222*	6,00,000	

* Committed expense of Rs. 9,85,496 for procurement of Electrochemical Instrument (Euro 12,700) against unspent balance of Rs. 10,00,222.

Name and Signature of Principal Investigator: 

Date: 7/6/2016

Signature of Competent financial authority: 
(with seal) Date: _____

* DOS - Date of Start of project

Note :

1. Expenditure under the sanctioned heads, at any point of time, should not exceed funds allocated under that head, without prior approval of SERB i.e. Figures in Column (VIII) should not exceed corresponding figures in Column (III)
2. Utilisation Certificate (Annexure III) for each financial year ending 31st March has to be enclosed along with request for carry-forward permission to the next financial year.

Finance Officer
Tezpur University

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (2016- 2017)

U.C. Pertains to	First Release	Second Release	Third Release	Fourth Release	Final Release
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√ *appropriate box*

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : *Amount* **9,49,997**
: Letter/Order No SB/FT/CS-048/2014
: *Date* 17.10.2016
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : *Amount* **NIL**
: Letter/Order No
: *Date*
- 6b. Interest earned, if any : NIL
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 9,49,997
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : 9,85,496
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : - 35,499
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: **NA**
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) - 35,499

P. Bharali
08/05/17

UTILISATION CERTIFICATE

Certified that out of Rs. NIL of **Non-Recurring** grants-in-aid sanctioned during the year 2016-17 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 17.10.2016 and Rs. 9,49,997/- on account of unspent balance of the previous year, a sum of Rs. 9,85,496/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. -35,499/- remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the grants-in-aid payable during the next year i.e. 2017-2018.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

1. Vouchers and Statement of Accounts
2. Expenditure Register

 Signature of PI Date: <u>08/09/17</u>	 Signature of Registrar/ Accounts Officer Date: _____ Finance Officer Tezpur University	 Signature of Head of the Institution Date: _____ Registrar Tezpur University
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Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31ST MARCH) (2016- 2017)

U.C. Pertains to	First Release	Second Release	Third Release	Fourth Release	Final Release
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√ *appropriate box*

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : *Amount* **50,554**
: Letter/Order No SB/FT/CS-048/2014
: *Date* 17.10.2016
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : *Amount* **6,00,000**
: Letter/Order No SB/FT/CS-048/2014
: *Date* 17.10.2016
- 6b. Interest earned, if any : 1,840
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 6,52,394
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : 5,62,489
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : 89,905
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: **NA**
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) : 89,905

P. Bharali
09/07/17


UTILISATION CERTIFICATE


Certified that out of Rs. 6,00,000/- of **Recurring** grants-in-aid sanctioned during the year 2016-17 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 17.10.2016 and Rs. 50,554/- on account of unspent balance of the previous year, a sum of Rs 5,62,489/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. 89,905/- [88,065/-+ 1840/- (Interest)] remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the **Recurring** grants-in-aid payable during the next year i.e. 2017-2018 _____.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

Kinds of checks exercised:

1. Vouchers and Statement of Accounts
2. Expenditure Register


Signature of PI
Date: 08/05/17


Signature of Registrar/ Accounts Officer
Date: 30/5/17
Finance Officer
Tezpur University


Signature of Head of the Institution
Date: _____
Registrar
Tezpur University

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

REQUEST FOR ANNUAL INSTALMENT WITH UP-TO-DATE STATEMENT OF EXPENDITURE

- (Two copies)
1. SERB Sanction Order No and date : SB/FT/CS-048/2014 and 16.06.2015
 2. Name of the PI : Dr. Pankaj Bharali
 3. Total Project Cost : 29,49,600/-
 4. Revised Project Cost(if applicable) : NA
 5. Date of Commencement : 30.07.2015
 6. Statement of Expenditure : (month wise expenditure incurred during current financial year)

Month & Year	Expenditure incurred/ committed
April 2016	14,000
May 2016	14,000
June 2016	14,000
July 2016	14,000 + 9,85,496 = 9,99,496
August 2016	14,000
September 2016	14,000
October 2016	14,000
November 2016	14,000 + 5,020 + 17,436 + + 62,500 = 98,956
December 2016	14,000
January 2017	14,000 + 18,210 = 32,210
February 2017	14,000 + 3,340 = 17,340
March 2017	14,000 + 97,373 + 13,375 + 1,14,622 + 60,990+ 1,623 = 3,01,983
Total	15,47,985

7. Grant received in each year:
 - a. 1st Year : 17,50,000/-
 - b. 2nd Year : 6,00,000/-
 - c. 3rd Year : NA
 - d. Interest, if any : 1840/-
 - e. Total (a+b+c+d) : 23,51,840/-

Pankaj Bharali
08/05/17

Statement of Expenditure
(For the financial Year 2016-2017, 01.04.2016 till 31.03.2017)

Sr No (I)	Sanctioned Heads (II)	Total Funds allocated (Indicate sanctioned or revised) (III)	Expenditure Incurred			Total Expenditure till (VII = IV + V + VI)	Balance as on 01.04.2016	Requirements of funds up to 31 st March Next year	Remarks (if any)
			1 st Year (30.06.2015 to 31.03.2016) (IV)	2 nd Year (01.04.2016 to 31.03.2017) (V)	3 rd Year & so on (01.04.2017 to 31.03.2018) (VI)				
1.	Manpower cost	6,00,000 + 6,00,000	1,11,097	1,68,000	NA	2,79,097	88,065	5,99,600	
2.	Consumables		3,17,801	2,43,580	-	5,61,381			
3.	Travel		0	8,360	-	8,360			
4.	Contingencies		20,550	80,049	-	1,00,599			
5.	Others, if any		0	0	-	0			
6.	Overhead Expenses		99,998	62,500	-	1,62,498			
7.	Equipment	11,50,000	2,00,332	9,85,496	-	11,85,828	-35,828		
8.	Total	23,50,000	7,49,778	15,47,985	NA	22,97,763	52,237	5,99,600	

Pankaj Bhandari (Pankaj Bhandari)

Name and Signature of Principal Investigator:

Date: 08/05/17

[Signature]
30/5/17

Signature of Competent financial authority: _____

(with seal) **Finance Officer** Date: _____

Tezpur University

* DOS - Date of Start of project

Note :

1. Expenditure under the sanctioned heads, at any point of time, should not exceed funds allocated under that head, without prior approval of SERB i.e. Figures in Column (VIII) should not exceed corresponding figures in Column (III)
2. Utilisation Certificate (Annexure III) for each financial year ending 31st March has to be enclosed along with request for carry-forward permission to the next financial year.

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (2017- 2018)

U.C. Pertains to	First Release	Second Release	Third Release	Fourth Release	Final Release
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√ *appropriate box*

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : *Amount 89,905/-*
: Letter/Order No SB/FT/CS-048/2014
: *Date 26.07.2017*
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : *Amount 4,00,000/-*
: Letter/Order No SB/FT/CS-048/2014
: *Date 26.07.2017*
- 6b. Interest earned, if any : 521/-
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 4,90,426/-
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : 4,37,023/-
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : 53,403/-
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: NA
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) : 53,403/-

P. Bharali
07/05/18


UTILISATION CERTIFICATE

Certified that out of Rs. 4,00,000/- of **Recurring** grants-in-aid sanctioned during the year 2017-18 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 26.07.2017 and Rs. 89,905/- on account of unspent balance of the previous year, a sum of Rs 4,37,023/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. 53,403/- [52,882/- + 521/- (Interest)] remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the **Recurring grants-in-aid** payable during the next year i.e. 2018-2019.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

Kinds of checks exercised:

1. Vouchers and Statement of Accounts
2. Expenditure Register


Signature of PI
Date: 09/05/18


Signature of Registrar/ Accounts Officer
Date: **Joint Registrar
Tezpur University**


Signature of Head of the Institution
Date: **Registrar
Tezpur University**

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

UTILISATION CERTIFICATE
FOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (2017- 2018)

U.C. Pertains to	First Release	Second Release	Third Release	Fourth Release	Final Release
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√ appropriate box

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : Amount – 35,499/-
: Letter/Order No SB/FT/CS-048/2014
: Date 26.07.2017
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : Amount NIL
: Letter/Order No
: Date
- 6b. Interest earned, if any : NIL
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : – 35,499/-
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : NIL
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : – 35,499/-
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: NA
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) – 35,499/-

P. Bharali
09/05/18

UTILISATION CERTIFICATE

Certified that out of Rs. NIL of **Non-Recurring** grants-in-aid sanctioned during the year 2017-18 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 26.07.2017 and Rs. -35,499/- on account of unspent balance of the previous year, a sum of Rs NIL has been utilised for the purpose for which it was sanctioned and that the balance of Rs. -35,499/- remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. _____ dated _____) /will be adjusted towards the grants-in-aid payable during the next year i.e. 2018-2019.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

1. Vouchers and Statement of Accounts
2. Expenditure Register


Signature of PI
Date: 09/05/18


Signature of Registrar/ Accounts Officer
Date: 6/5/18
Joint Registrar
Tezpur University


Signature of Head of the Institution
Date: Registrar
Tezpur University

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

REQUEST FOR ANNUAL INSTALMENT WITH UP-TO-DATE STATEMENT OF EXPENDITURE

- (Two copies)
1. SERB Sanction Order No and date : SB/FT/CS-048/2014 and 16.06.2015
 2. Name of the PI : Dr. Pankaj Bharali
 3. Total Project Cost : 29,49,600/-
 4. Revised Project Cost(if applicable) : NA
 5. Date of Commencement : 30.07.2015
 6. Statement of Expenditure : (month wise expenditure incurred during current financial year)

Month & Year	Expenditure incurred/ committed
April 2017	-
May 2017	14,000
June 2017	14,000
July 2017	14,000
August 2017	-
September 2017	28,000 + 15,150 + 62,500 = 1,05,650
October 2017	16,000 + 8,533 + 1,300 + 31,513 + 6,188 = 63,534
November 2017	16,000 + 2,333 = 18,333
December 2017	16,000 + 6,668 = 22,668
January 2018	16,000 + 1,22,720 = 1,38,720
February 2018	16,000
March 2018	30,118
Total	4,37,023

7. Grant received in each year:
 - a. 1st Year : 17,50,000/-
 - b. 2nd Year : 6,00,000/-
 - c. 3rd Year : 4,00,000/-
 - d. Interest, if any : 521/-
 - e. Total (a+b+c+d) : 27,50,521/-

P. Bharali
09/05/18

Statement of Expenditure
(For the financial Year 2017-2018, 01.04.2017 till 31.03.2018)

Sr No (I)	Sanctioned Heads (II)	Total Funds allocated (Indicate sanctioned or revised) (III)	Expenditure Incurred			Total Expenditure till (VII = IV + V + VI)	Balance as on 01.04.2018	Requirements of funds up to 29.07.2018, i.e. completion of project	Remarks (if any)
			1 st Year (30.06.2015 to 31.03.2016) (IV)	2 nd Year (01.04.2016 to 31.03.2017) (V)	3 rd Year (01.04.2017 to 31.03.2018) (VI)				
1.	Manpower cost	6,00,000 + 6,00,000 + 4,00,000	1,11,097	1,68,000	1,60,866	4,39,963	51,042	1,99,600	
2.	Consumables		3,17,801	2,43,580	1,22,720	6,84,101			
3.	Travel		0	8,360	12,856	21,216			
4.	Contingencies		20,550	80,049	46,568	1,47,167			
5.	Others, if any		0	0	0	0			
6.	Overhead Expenses		99,998	62,500	94,013	2,56,511			
7.	Equipment	11,50,000	2,00,332	9,85,496	-	11,85,828	-35,828		
8.	Total	27,50,000	7,49,778	15,47,985	4,37,023	27,34,786	15,214	1,99,600	

Pankaj Shrivastava
Name and Signature of Principal Investigator:

Date: 09/05/18

[Signature]
Signature of Competent financial authority:
(with seal) **Joint Registrar** Date: 06/05/18
Tezpur University

* DOS - Date of Start of project

Note :

1. Expenditure under the sanctioned heads, at any point of time, should not exceed funds allocated under that head, without prior approval of SERB i.e. Figures in Column (VIII) should not exceed corresponding figures in Column (III)
2. Utilisation Certificate (Annexure III) for each financial year ending 31st March has to be enclosed along with request for carry-forward permission to the next financial year.

UTILISATION CERTIFICATEFOR THE FINANCIAL YEAR – (ENDING 31ST MARCH) (1ST April, 2018- 31ST July, 2018)

U.C. Pertains to	First Release	Second Release	Third Release	Fourth Release	Final Release
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√ appropriate box

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : Amount **53,403/-**
: Letter/Order No SB/FT/CS-048/2014
: Date 09.07.2018
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : Amount **1,50,000/-**
: Letter/Order No SB/FT/CS-048/2014
: Date 09.07.2018
- 6b. Interest earned, if any : 329/-
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : 2,03,732/-
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31ST March) : 1,83,382/-
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : 20,350/-
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: **20,350/-**
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) NA


09.10.18


UTILISATION CERTIFICATE

Certified that out of Rs. 1,50,000/- of **Recurring** grants-in-aid sanctioned during the year 2018-19 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 09.07.2018 and Rs. 53,403/- on account of unspent balance of the previous year, a sum of Rs. 1,83,382/- has been utilised for the purpose for which it was sanctioned and that the balance of Rs. 20,350/- [20,021/- + 329/- (Interest)] remaining unutilised at the end of the year has been refunded/returned to SERB (vide DD/Cheque No. 532770 dated 11/12/18) ~~/will be adjusted towards the~~ **Recurring** grants-in-aid payable during the next year.

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

Kinds of checks exercised:

1. Vouchers and Statement of Accounts
2. Expenditure Register


Signature of PI
Date: 09.10.18


Signature of Registrar/Accounts Officer
Date: 26/11/18
Finance Officer
Tezpur University


Signature of Head of the Institution
Date: 26/11/18
Registrar
Tezpur University

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

UTILISATION CERTIFICATEFOR THE FINANCIAL YEAR – (ENDING 31st MARCH) (1st April, 2018- 31st July, 2018)

U.C. Pertains to

First Release	Second Release	Third Release	Fourth Release	Final Release
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√ appropriate box

Is the UC provisional

YES / NO

1. Title of the Project/ Scheme : Development of Hybrid Catalysts for Oxygen Reduction Reaction
2. Name of the Principal Investigator : Dr. Pankaj Bharali
3. Implementing institution : Tezpur university
4. SERB sanction order No & date : SB/FT/CS-048/2014
Dated: 16.06.2015
5. Amount brought forward from the previous financial year quoting SERB letter number and date in which the authority to carry forward the said amount was given : Amount – **35,499/-**
: Letter/Order No SB/FT/CS-048/2014
: Date 09.07.2018
- 6a. Amount received during the financial year (Please give SERB letter/Order No and date For the amount): : Amount **NIL**
: Letter/Order No
: Date
- 6b. Interest earned, if any : NIL
7. Total amount that was available for expenditure Rs.(excluding commitments) during the financial year (Sr. No. 5+6a+6b) : – 35,499/-
8. Actual expenditure Rs.(excluding commitments) incurred during the financial year (upto 31st March) : NIL
9. Balance amount available at the end of the financial year (8-9): OR / Negative balance (If expenditure incurred is more than the fund released) : NIL (Rs. – 35,499/- has been adjusted to the Institutional overhead)
10. Unspent balance, if any, refunded to SERB (Give details of cheque/DD No. etc.) : Amount: **NA**
: Cheque/DD No
: Date
11. Amount to be carried forward to next financial Year (if any) : NIL

P. Bharali
9.10.18

UTILISATION CERTIFICATE

Certified that out of Rs. NIL of **Non-Recurring** grants-in-aid sanctioned during the year 2018-19 in favour of Registrar, Tezpur University under SERB letter/order No. SB/FT/CS-048/2014 dated 09.07.2018 and Rs. -35,499/- on account of unspent balance of the previous year, a sum of Rs NIL has been utilised for the purpose for which it was sanctioned and that the balance of Rs. NIL remaining unutilised at the end of the year has been refunded/returned to SERB (vide ~~DD/Cheque~~ No. _____ dated _____) ~~/will be adjusted towards the grants-in-aid payable during the next year i.e.~~

Certified that we have satisfied ourselves that the conditions on which the grants-in-aid was sanctioned have been fulfilled/ are being fulfilled and that we have exercised the following checks to see that the money was actually utilised for the purpose for which it was sanctioned.

1. Vouchers and Statement of Accounts
2. Expenditure Register

Signature of PI
Date: 9.10.18

Signature of Registrar/Accounts Officer
Date: _____
Finance Officer
Tezpur University

Signature of Head of the Institution
Date: _____
Registrar
Tezpur University

Guidelines for preparation of UC

1. UC should be only for the grants released by the SERB. Please **do not account** for security deposits/ other matching grants/ account opening charges and miscellaneous items.
2. SERB Sanction No. and Dt. Should be accurately shown in the U.C.
3. Even if the grant is unutilised in the financial year in which the grant was released by SERB a NIL U.C. needs to be forwarded to SERB along with request for carrying forward the grant to the next financial year. Such grants which are carried forward must be shown in the subsequent U.C. as carried forward grant and not amount received in the subsequent year (ref SI No. 5 on pre-page)

Science and Engineering Research Board

UC accepted has been accepted by

Signature: _____

Name of the SERB Officer: _____

Designation: _____

REQUEST FOR ANNUAL INSTALMENT WITH UP-TO-DATE STATEMENT OF EXPENDITURE (FINAL)

- (Two copies)
1. SERB Sanction Order No and date : SB/FT/CS-048/2014 and 16.06.2015
 2. Name of the PI : Dr. Pankaj Bharali
 3. Total Project Cost : 29,49,600/-
 4. Revised Project Cost(if applicable) : NA
 5. Date of Commencement : 30.07.2015
 6. Statement of Expenditure : (month wise expenditure incurred during current financial year)

Month & Year	Expenditure incurred/ committed
April 2018	16000
May 2018	-
June 2018	-
July 2018	64,000 + 49071 + 10986 + 7497 + 35828
Total	1,83,382

7. Grant received in each year:
 - a. 1st Year : 17,50,000/-
 - b. 2nd Year : 6,00,000/-
 - c. 3rd Year : 4,00,000/-
 - d. 4th year : 1,50,000/-
 - e. Interest, if any : (329/-+1840/- + 521/-) = 2690/-
 - f. Total (a+b+c+d+e) : 29,02,690/-


 09-10-18

Statement of Expenditure
(For the financial Year 2018-2019, 01.04.2018 till 31.07.2018)

Sr No (I)	Sanctioned Heads (II)	Total Funds allocated (Indicate sanctioned or revised) (III)	Expenditure Incurred				Total Expenditure till (VIII = IV + V + VI+VII)	Balance as on 01.10.2018	Requirem ents of funds	Remark s (if any)
			1 st Year (30.06.2015 to 31.03.2016) (IV)	2 nd Year (01.04.2016 to 31.03.2017) (V)	3 rd Year (01.04.2017 to 31.03.2018) (VI)	4 th Year (01.04.2018 to 31.09.2018) (VII)				
1.	Manpower cost	6,00,000 + 6,00,000 + 4,00,000 + 1,50,000	1,11,097	1,68,000	1,60,866	80,000	5,19,963	17,660		
2.	Consumables		3,17,801	2,43,580	1,22,720	49,071	7,33,172			
3.	Travel		0	8,360	12,856	0	21,216			
4.	Contingencies		20,550	80,049	46,568	10,986	1,58,153			
5.	Others, if any		0	0	0	0	0			
6.	Overhead Expenses		99,998	62,500	94,013	43,325*	2,99,836			
7.	Equipment	11,50,000	2,00,332	9,85,496	-	(35,828)*	11,50,000*	NIL		
8.	Total	29,00,000	7,49,778	15,47,985	4,37,023	1,83,382	28,82,340	17,660		

* Excess expenditure of Rs. 35828/- in the equipment head has been adjusted to institutional overhead.

 (Pankaj Bhanal)

Name and Signature of Principal Investigator:

Date: 09.10.18


Signature of Competent financial authority: _____
(with seal) **Finance Officer** Date: _____
Teapar University

* DOS - Date of Start of project

Note :

1. Expenditure under the sanctioned heads, at any point of time, should not exceed funds allocated under that head, without prior approval of SERB i.e. Figures in Column (VIII) should not exceed corresponding figures in Column (III)
2. Utilisation Certificate (Annexure III) for each financial year ending 31st March has to be enclosed along with request for carry-forward permission to the next financial year.